EXHIBIT 152
PART 2

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM C177: Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded Hot-Plate Apparatus

CFR Section(s): 10 CFR 431.102

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: C 177 – 97

Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus[1]

This standard is issued under the fixed designation C 177; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method establishes the criteria for the laboratory measurement of the steady-state heat flux through flat, homogeneous specimen(s) when their surfaces are in contact with solid, parallel boundaries held at constant temperatures using the guarded-hot-plate apparatus.

1.2 The test apparatus designed for this purpose is known as a guarded-hot-plate apparatus and is a primary (or absolute) method. This test method is comparable, but not identical, to ISO 8302.

1.3 This test method sets forth the general design requirements necessary to construct and operate a satisfactory guarded-hot-plate apparatus. It covers a wide variety of apparatus constructions, test conditions, and operating conditions. Detailed designs conforming to this test method are not given but must be developed within the constraints of the general requirements. Examples of analysis tools, concepts and procedures used in the design, construction, calibration and operation of a guarded-hot-plate apparatus are given in Refs **(1–41)**.[2]

1.4 This test method encompasses both the single-sided and the double-sided modes of measurement. Both distributed and line source guarded heating plate designs are permitted. The user should consult the standard practices on the single-sided mode of operation, Practice C 1044, and on the line source apparatus, Practice C 1043, for further details on these heater designs.

1.5 The guarded-hot-plate apparatus can be operated with either vertical or horizontal heat flow. The user is cautioned however, since the test results from the two orientations may be different if convective heat flow occurs within the specimens.

1.6 Although no definitive upper limit can be given for the magnitude of specimen conductance that is measurable on a guarded-hot-plate, for practical reasons the specimen conductance should be less than 16 W/(m^{2}K).

1.7 This test method is applicable to the measurement of a wide variety of specimens, ranging from opaque solids to porous or transparent materials, and a wide range of environmental conditions including measurements conducted at extremes of temperature and with various gases and pressures.

1.8 Inhomogeneities normal to the heat flux direction, such as layered structures, can be successfully evaluated using this test method. However, testing specimens with inhomogeneities in the heat flux direction, such as an insulation system with thermal bridges, can yield results that are location specific and shall not be attempted with this type of apparatus. See Test Methods C 976 or C 236 for guidance in testing these systems.

1.9 Calculations of thermal transmission properties based upon measurements using this method shall be performed in conformance with Practice C 1045.

1.10 In order to ensure the level of precision and accuracy expected, persons applying this standard must possess a knowledge of the requirements of thermal measurements and testing practice and of the practical application of heat transfer theory relating to thermal insulation materials and systems. Detailed operating procedures, including design schematics and electrical drawings, should be available for each apparatus to ensure that tests are in accordance with this test method. In addition, automated data collecting and handling systems connected to the apparatus must be verified as to their accuracy. This can be done by calibration and inputting data sets, which have known results associated with them, into computer programs.

1.11 It is not practical for a test method of this type to establish details of design and construction and the procedures to cover all contingencies that might offer difficulties to a person without technical knowledge concerning theory of heat flow, temperature measurements and general testing practices. The user may also find it necessary, when repairing or modifying the apparatus, to become a designer or builder, or both, on whom the demands for fundamental understanding and careful experimental technique are even greater. Standardization of this test method is not intended to restrict in any way the future development of new or improved apparatus or procedures.

1.12 This test method does not specify all details necessary for the operation of the apparatus. Decisions on sampling, specimen selection, preconditioning, specimen mounting and positioning, the choice of test conditions, and the evaluation of test data shall follow applicable ASTM Test Methods, Guides, Practices or Product Specifications or governmental regulations. If no applicable standard exists, sound engineering judgment that reflects accepted heat transfer principles must be used and documented.

1.13 This test method allows a wide range of apparatus

---

[1] This test method is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurement.

Current edition approved Feb. 10, 1997. Published June 1997. Originally published as C 177 – 42. Last previous edition C 177 – 85 (1993).

[2] The boldface numbers given in parentheses refer to the list of references at the end of this standard.

design and design accuracy to be used in order to satisfy the requirements of specific measurement problems. Compliance with this test method requires a statement of the uncertainty of each reported variable in the report. A discussion of the significant error factors involved is included.

1.14 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only. Either SI or Imperial units may be used in the report, unless otherwise specified.

1.15 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Note 22.

1.16 Major sections within this test method are arranged as follows:

2. Referenced Documents

2.1 *ASTM Standards:*

C 167 Test Methods for Thickness and Density of Blanket or Batt Thermal Insulations[3]

C 168 Terminology Relating to Thermal Insulating Materials[3]

C 236 Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box[3]

C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[3]

C 687 Practice for Determination of Thermal Resistance of Loose-Fill Building Insulation[3]

C 976 Test Method for Thermal Performance of Building Assemblies by Means of a Calibrated Hot Box[3]

C 1043 Practice for Guarded-Hot-Plate Design Using Circular Line-Heat Sources[3]

C 1044 Practice for Using the Guarded-Hot-Plate Apparatus in the One-Sided Mode to Measure Steady-State Heat Flux and Thermal Transmission Properties[3]

C 1045 Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements[3]

C 1058 Practice for Selecting Temperatures for Evaluating and Reporting Thermal Properties of Thermal Insulation[3]

E 230 Specification for Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[4]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

2.2 *ISO Standard:*

ISO 8302 Thermal Insulation—Determination of Steady-State Areal Thermal Resistance and Related Properties—Guarded-Hot-Plate Apparatus[6]

2.3 *ASTM Adjuncts:*

Table of Theoretical Maximum Thickness of Specimens and Associated Errors[7]

Descriptions of Three Guarded-Hot-Plate Designs[7]

Line-Heat-Source Guarded Hot-Plate Apparatus[8]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms and symbols used in this test method, refer to Terminology C 168 and the following subsections.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *auxiliary cold surface assembly, n*—the plate that provides an isothermal boundary at the outside surface of the auxiliary insulation.

3.2.2 *auxiliary insulation, n*—insulation placed on the back side of the hot-surface assembly, in place of a second test specimen, when the single sided mode of operation is used. (*Synonym*—backflow specimen.)

3.2.3 *cold surface assembly, n*—the plates that provide an isothermal boundary at the cold surfaces of the test specimen.

3.2.4 *controlled environment, n*—the environment in which an apparatus operates.

3.2.5 *guard, n*—promotes one-dimensional heat flow. Primary guards are planar, additional coplanar guards can be used and secondary or edge guards are axial.

3.2.6 *guarded-hot-plate apparatus, n*—an assembly, consisting of a hot surface assembly and two isothermal cold surface assemblies.

3.2.7 *guarded-hot-plate, n*—the inner (rectangular or circular) plate of the hot surface assembly, that provides the heat input to the metered section of the specimen(s).

3.2.8 *hot surface/assembly, n*—the complete center assembly providing heat to the specimen(s) and guarding for the meter section.

---

[3] *Annual Book of ASTM Standards*, Vol 04.06.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[7] Available from ASTM Headquarters, Request PCN No. 12-301770-00.

[8] Available from ASTM Headquarters, Request PCN No. 12-310430-61.

3.2.9 *metered section, n*—the portion of the test specimen (or auxiliary insulation) through which the heat input to the guarded-hot-plate flows under ideal guarding conditions.

3.2.10 *mode, double-sided, n*—operation of the guarded-hot-plate apparatus for testing two specimens, each specimen placed on either side of the hot surface assembly.

3.2.11 *mode, single-sided, n*—operation of the guarded-hot-plate apparatus for testing one specimen, placed on one side of the hot-surface assembly.

3.2.12 *thermal transmission properties, n*— those properties of a material or system that define the ability of a material or system to transfer heat such as thermal resistance, thermal conductance, thermal conductivity and thermal resistivity, as defined by Terminology C 168.

3.3 *Symbols*—The symbols used in this test method have the following significance:

3.3.1 ρ_m—specimen metered section density, kg/m^3.

3.3.2 ρ_s—specimen density, kg/m^3.

3.3.3 λ—thermal conductivity, W/(m K).

3.3.4 σ—Stefan-Boltzmann constant, W/m^2 K^4.

3.3.5 A—metered section area normal to heat flow, m^2.

3.3.6 A_g—area of the gap between the metered section and the primary guard, m^2.

3.3.7 A_m—area of the actual metered section, m^2.

3.3.8 A_s—area of the total specimen, m^2.

3.3.9 C—thermal conductance, W/(m^2 K).

3.3.10 C_i—the specific heat of the ith component of the metered section, J/(kg K).

3.3.11 dT/dt—potential or actual drift rate of the metered section, K/s.

3.3.12 λ_g—thermal conductivity of the material in the primary guard region, W/(m K).

3.3.13 L—in-situ specimen thickness, m.

3.3.14 m—mass of the specimen in the metered section, kg.

3.3.15 m_i—the mass of the ith component, kg.

3.3.16 m_s—mass of the specimen, kg.

3.3.17 Q—heat flow rate in the metered section, W.

3.3.18 q—heat flux (heat flow rate per unit area), Q, through area, A, W/m^2.

3.3.19 Q_{ge}—lateral edge heat flow rate between primary Guard and Controlled Environment, W.

3.3.20 Q_{gp}—lateral heat flow rate across the gap, W.

3.3.21 Q_{grd}—guard heat flow through Specimen, W.

3.3.22 Q_{se}—edge heat flow between Specimen and Controlled Environment, W.

3.3.23 R—thermal resistance, m^2 K/W.

3.3.24 ΔT—temperature difference across the specimen, $T_h - T_c$.

3.3.25 T_c—cold surface temperature, K.

3.3.26 T_h—hot surface temperature, K.

3.3.27 T_m—mean temperature, K, $(T_h + T_c)/2$.

4. Summary of Test Method

4.1 Figure 1 illustrates the main components of the idealized system: two isothermal cold surface assemblies and a guarded-hot-plate. The guarded-hot-plate is composed of a metered section thermally isolated from a concentric primary guard by a definite separation or gap. Some apparatus may have more than one guard. The test specimen is sandwiched between these three units as shown in Fig. 1. In the double-sided mode of measurement, the specimen is actually composed of two pieces. The measurement in this case produces a result that is the average of the two pieces and therefore it is important that the two pieces be closely identical. For guidance in the use of the one-sided mode of measurement, the user is directed to Practice C 1044. For guidance in the use of a guarded-hot-plate incorporating the use of a line source heater, refer to Practice C 1043.

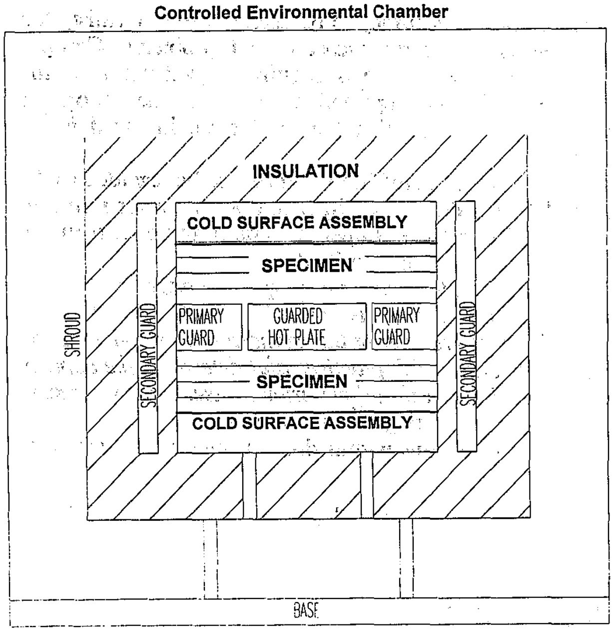


FIG. 1 General Arrangement of the Mechanical Components of the Guarded-Hot-Plate Apparatus

4.1.1 The guarded-hot-plate provides the power (heat flow per unit time) for the measurement and defines the actual test volume, that is, that portion of the specimen that is actually being measured. The function of the primary guard, and additional coplanar guard where applicable, of the guarded-hot-plate apparatus is to provide the proper thermal conditions within the test volume to reduce lateral heat flow within the apparatus. The proper (idealized) conditions are illustrated in Fig. 1 by the configuration of the isothermal surfaces and lines of constant heat flux within the specimen.

4.1.2 Deviations from the idealized configuration are caused by: specimen inhomogeneities, temperature differences between the metered section and the guard (gap imbalance), and temperature differences between the outer edge of the assembly and the surrounding controlled environment (edge imbalance). These experimental realities lead to heat flow measurements that are too small or too large because the power supplied to the metered section is not exactly equal to that which flows through the specimen in the metered section. The resulting qualitative heat flows are depicted in Fig. 2.

4.2 The three heating/cooling assemblies are designed to create isothermal surfaces on the faces of the specimens within the metered section. The two surfaces designated as the cold surface assemblies are adjusted to the same temperature for the double-sided mode of operation. In practice,

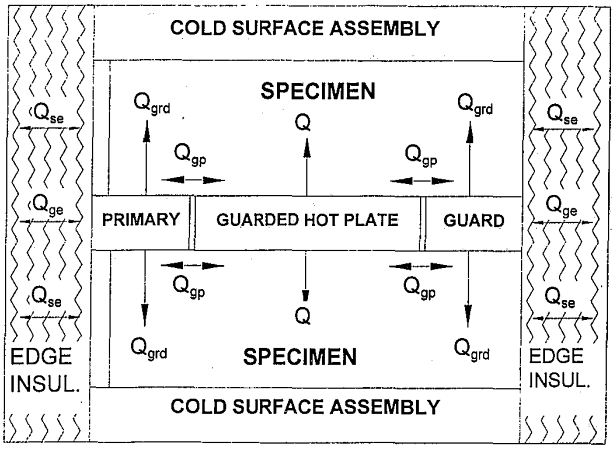


FIG. 2 Illustration of Idealized Heat Flow in a Guarded-Hot-Plate Apparatus

because the plates and specimens are of finite dimensions, and because the external controlled environment is often at a temperature different from the edge of the metered section, some lateral heat flow occurs. The primary guard for the guarded hot plate limits the magnitude of the lateral heat flow in the metered section. The effectiveness of the primary guard is determined, in part, by the ratio of its lateral dimension to that of the metered section and to the specimen thickness **(6,7,8,20,31)**.

4.3 Compliance with this test method requires: the establishment of steady-state conditions, and the measurement of the unidirectional heat flow Q in the metered section, the metered section area A, the temperature gradient across the specimen, in terms of the temperature T_h of the hot surface and the temperature T_c of the cold surface, (or equivalently, the temperature T between the two surfaces), the thickness’ L_1 and L_2 of each specimen, and guard balance between the metered section and primary guard.

5. Significance and Use

5.1 This test method covers the measurement of heat flux and associated test conditions for flat specimens. The guarded-hot-plate apparatus is generally used to measure steady-state heat flux through materials having a "low" thermal conductivity and commonly denoted as "thermal insulators." Acceptable measurement accuracy requires a specimen geometry with a large ratio of area to thickness.

5.2 Two specimens are selected with their thickness, areas, and densities as identical as possible, and one specimen is placed on each side of the guarded-hot-plate. The faces of the specimens opposite the guarded-hot-plate and primary guard are placed in contact with the surfaces of the cold surface assemblies.

5.3 Steady-state heat transmission through thermal insulators is not easily measured, even at room temperature. This is because heat may be transmitted through a specimen by any or all of three separate modes of heat transfer (radiation, conduction, and convection); any inhomogeneity or anisotropy in the specimen may require special experimental precautions to measure that flow of heat; hours or even days may be required to achieve the thermal steady-state; no guarding system can be constructed to force the metered heat to pass only through the test area of insulation specimen being measured; moisture content within the material may cause transient behaviour; and physical or chemical change in the material with time or environmental condition may permanently alter the specimen.

5.4 Application of this test method on different test insulations requires that the designer make choices in the design selection of materials of construction and measurement and control systems. Thus there may be different designs for the guarded-hot-plate apparatus when used at ambient versus cryogenic or high temperatures. Test thickness, temperature range, temperature difference range, ambient conditions and other system parameters must also be selected during the design phase. Annex A1 is referenced to the user, which addresses such issues as limitations of the apparatus, thickness measurement considerations and measurement uncertainties, all of which must be considered in the design and operation of the apparatus.

5.5 Apparatus constructed and operated in accordance with this test method should be capable of accurate measurements for its design range of application. Since this test method is applicable to a wide range of specimen characteristics, test conditions, and apparatus design, it is impractical to give an all-inclusive statement of precision and bias for the test method. Analysis of the specific apparatus used is required to specify a precision and bias for the reported results. For this reason, conformance with the test method requires that the user must estimate and report the uncertainty of the results under the reported test conditions.

5.6 Qualification of a new apparatus. When a new or modified design is developed, tests shall be conducted on at least two materials of known thermal stability and having verified or calibrated properties traceable to a national standards laboratory. Tests shall be conducted for at least two sets of temperature conditions that cover the operating range for the apparatus. If the differences between the test results and the national standards laboratory characterization are determined to be significant, then the source of the error shall, if possible, be identified. Only after successful comparison with the certified samples, can the apparatus claim conformance with this test method. It is recommended that checks be continued on a periodic basis to confirm continued conformance of the apparatus.

5.7 The thermal transmission properties of a specimen of material: may vary due to the composition of the material; may be affected by moisture or other environmental conditions; may change with time or temperature exposure; may change with thickness; may change with temperature difference across the specimen; or may change with mean temperature. It must be recognized, therefore, that the selection of a representative value of thermal transmission properties for a material must be based upon a consideration of these factors and an adequate amount of test information.

5.8 Since both heat flux and its uncertainty may be dependent upon environmental and apparatus test conditions, as well as intrinsic characteristics of the specimen, the report for this test method shall include a thorough description of the specimen and of the test conditions.

5.9 The results of comparative test methods such as Test Method C 518 depend on the quality of the heat flux reference standards. The apparatus in this test method is one of the absolute methods used for generation of the reference standards. The accuracy of any comparative method can be no better than that of the referenced procedure. While the precision of a comparative method such as Test Method C 518 may be comparable with that of this test method, Test Method C 518 cannot be more accurate. In cases of dispute, this test method is the recommended procedure.

6. Apparatus

6.1 A general arrangement of the mechanical components of such a guarded-hot-plate apparatus is illustrated in Fig. 1. This consists of a hot surface assembly comprised of a metered section and a primary guard, two cold surface assemblies, and secondary guarding in the form of edge insulation, a temperature-controlled secondary guard(s), and often an environmental chamber. Some of the components illustrated in Fig. 1 are omitted in systems designed for ambient conditions, although a controlled laboratory environment is still required; edge insulation and the secondary guard are typically used only at temperatures that are more than ±10°C (20°F) from ambient. At ambient conditions, the environmental chamber is recommended to help eliminate the effects of air movement within the laboratory and to help ensure that a dry environment is maintained.

6.1.1 The purpose of the hot surface assembly is to produce a steady-state, one-dimensional heat flux through the specimens. The purpose of the edge insulation, secondary guard, and environmental chamber is to restrict heat losses from the outer edge of the primary guard. The cold surface assemblies are isothermal heat sinks for removing the energy generated by the heating units; the cold surface assemblies are adjusted so they are at the same temperature.

6.2 *Design Criteria*—Establish specifications for the following specifications prior to the design. Various parameters influence the design of the apparatus and shall be considered throughout the design process, maximum specimen thickness; range of specimen thermal conductances; range of hot surface and cold surface temperatures; characteristics of the specimens (that is, rigidity, density, hardness); orientation of the apparatus (vertical or horizontal heat flow); and required accuracy.

6.3 *Hot Surface Assembly*—The hot surface assembly consists of a central metered section and a primary guard. The metered section consists of a metered section heater sandwiched between metered section surface plates. The primary guard is comprised of one or more guard heaters sandwiched between primary guard surface plates. The metered section and primary guard shall be thermally isolated from each other by means of a physical space or gap located between the sections. The hot surface assembly using a line-heat-source is covered in Practice C 1043.

NOTE 1—The primary guard, in some cases, is further divided into two concentric sections (double guard) with a gap separator to improve the guard effectiveness.

6.3.1 *Requirements*—The hot surface assembly shall be designed and constructed to satisfy the following minimum requirements during operation.

6.3.1.1 The maximum departure from a plane for any surface plate shall not exceed 0.025 % of the linear dimension of the metered section during operation.

NOTE 2—Planeness of the surface can be checked with a metal straightedge held against the surface and viewed at grazing incidence with a light source behind the straightedge. Departures as small as 2.5 μm are readily visible, and large departures can be measured using shim-stock, thickness gages or thin paper.

6.3.1.2 The average temperature difference between the metered section surface plate and the primary guard surface plate shall not exceed 0.2 K. In addition, the temperature difference across any surface plate in the lateral direction shall be less than 2 % of the temperature difference imposed across the specimen.

NOTE 3—When qualifying the apparatus, additional temperature sensors shall be applied to the surface plates of the metered section and primary guards that verify that the requirements of 6.3.1.2 are satisfied.

6.3.1.3 The surfaces of the metered and primary guard surface plates that are in contact with the test specimen shall be treated to maintain a total hemispherical emittance greater than 0.8 over the entire range of operating conditions.

NOTE 4—At high temperatures the importance of high emittance of the surfaces adjacent to the specimens cannot be stressed too strongly since radiative heat transfer predominates in many materials as the temperature increases.

6.3.1.4 The metered section and primary guard surface plates shall remain planar during the operation of the apparatus. See 6.3.1.1.

6.3.2 *Materials*—The materials used in the construction of the hot surface assembly shall be carefully chosen after considering the following material property criteria.

6.3.2.1 *Temperature Stability*—Materials are selected for the heaters and surface plates that are dimensionally and chemically stable and suitably strong to withstand warpage and distortion when a clamping force is applied. For modest temperatures, electric resistance heaters embedded in silicone have been successfully employed; at higher temperatures, heating elements sandwiched between mica sheets or inserted into a ceramic core have been used. Surface plates for hot surface assemblies used at modest temperatures have been fabricated from copper and aluminum. High purity nickel alloys have been used for higher temperature applications.

6.3.2.2 *Thermal Conductivity*—To reduce the lateral temperature differences across the metered and primary guard surface plates, fabricate these plates from materials that possess a high thermal conductivity for the temperature and environmental conditions of operation. Copper and aluminum are excellent choices for modest temperature applications; at higher temperatures consider using nickel, high purity alumina or aluminum nitride. These are examples of materials used and the operator must fully understand the thermal conductivity versus temperature dependency of the materials selected.

6.3.2.3 *Emittance*—To obtain a uniform and durable high surface emittance in the desired range, select a surface plate material or suitable surface treatment, or both. For modest temperature applications, high emittance paints may be employed. Aluminum can be anodized to provide the necessary high emittance. For high temperature applications, most ceramics will inherently satisfy this requirement while nickel surface plates can be treated with an oxide coating.

6.3.2.4 *Temperature Uniformity*—Select a heating element design that will supply the necessary heat flux density for the range of specimen thermal conductances to be investigated. The design of the heating element shall also consider the heat flux distribution of the surface of the heating element. Most apparatus incorporate the use of a distributed electric resistance heating element dispersed uniformly across the metered section and the primary guard. The surface plates and heating elements shall be clamped or bolted together in a uniform manner such that the temperature difference requirements specified in 6.3.1.2 are satisfied. Bolting the composite constructions together has been found satisfactory.

6.3.2.5 The insertion of insulating sheets between the heating elements and surface plates (that is, to mount a gap temperature imbalance detector) is allowed. To satisfy the requirements of 6.3.1.2, similar sheets shall be mounted between the heating element and the opposing surface plate.

6.3.2.6 *Hot Surface Assembly Size*—Design criteria established in 6.2 will determine the size of the apparatus. The size of the metered section shall be large enough so that the amount of specimen material in contact with the metered section (and therefore being measured) can be considered representative of the material being tested.

6.3.2.7 After determining the maximum specimen thickness that will be tested by this design, refer to Adjunct, Table of Theoretical Maximum Thickness of Specimens and Associated Errors, regarding associated errors attributable to combinations of metered section size, primary guard width, and specimen thickness.

NOTE 5— Typically the width of the primary guard equal to approximately one-half of the linear dimension of the metered section has been found to reduce edge heat loss to acceptable levels.

6.3.2.8 *Heat Capacitance*—The heat capacity of the hot surface assembly will impact the time required to achieve thermal equilibrium. Selecting materials with a low specific heat will increase the responsiveness of the apparatus. The thickness of the surface plates needs to be carefully considered; thick plates assist in reducing lateral temperature distributions but reduce responsiveness. A balance between these requirements is needed.

6.4 *The Gap*—The metered section and the primary guard shall be physically separated by a gap. The gap provides a lateral thermal resistance between these sections of the hot surface assembly. The area of the gap in the plane of the surface plates shall not be more than 5 % of the metered section area.

6.4.1 The heater windings from the metered section and primary guard heating elements shall be designed to create a uniform temperature along the gap perimeter.

6.4.2 The metered section area shall be determined by measurements to the center of the gap that surrounds this area, unless detailed calculations or tests are used to define this area more precisely.

6.4.3 Any connections between the metered section and the primary guard shall be designed to minimize heat flow across the gap. If a mechanical means is used to satisfy the requirements of 6.3.1.4, these connections shall be fabricated with materials having a high thermal resistance. Instrumentation or heater leads that cross the gap should be fabricated with fine-gage wire and traverse the gap at an oblique angle.

6.4.4 The gap may be filled with a fibrous insulation. Packing the gap with this insulation has been found to maintain the metered section and primary guard surface plates planar. An additional benefit of this practice for high temperature applications is that the densely packed insulation reduces the amount of heat conducted across the gap spacing.

6.5 *Cold Surface Assembly*—The cold surface assembly consists of a single temperature controlled section and is comprised of a cold surface heater sandwiched between cold surface plates and a heat sink. It is recommended that the size of the cold surface assembly be identical to the hot surface assembly, including the primary guard. Cold surface assemblies may be constructed with a gap where operation of the apparatus is susceptible to edge loss effects. This design is the ideal design, however, this assembly has traditionally been constructed without a gap with great success.

NOTE 7—The temperature of the cold surface assembly may be maintained through the use of a temperature-controlled bath; in this instance, there is no need to install a cold surface heater. Care must be taken in this instance; the flow rate of the bath must be sufficient to satisfy the temperature uniformity requirements specified in 6.3.1.2 and 6.5.1.

6.5.1 *Requirements*—The cold surface assemblies shall be designed and constructed to satisfy all of the requirements of 6.3.1 except that, since only one surface plate of each cold surface assembly is in contact with the test specimens, the requirement that specifies the temperature difference between the surface plates shall not apply.

6.5.2 *Materials*—The criteria to select materials that will be used in the construction of the cold surface assemblies are identical to the hot surface assembly and are listed in 6.3.2.

6.5.3 *High Temperature Operation*—When the cold surface assemblies will be operated at high temperatures, several thin sheets of insulation may be inserted between the heat sink and cold surface heater. The addition of these insulation sheets will reduce the energy requirements to the cold surface heater and extend service life.

6.6 *Additional Edge Loss Protection*—Deviation from one-dimensional heat flow in the test specimen is due to non-adiabatic conditions at the edges of the hot surface assembly and the specimens. This deviation is greatly increased when the apparatus is used at temperatures other than ambient. When the guarded-hot-plate apparatus is operated at temperatures that deviate from ambient by more than 10°C (20°F), the apparatus shall be outfitted with additional components to reduce edge losses. These components are described in the following sections and shall be used if edge losses cannot be minimized.

NOTE 8—Another means of assessing whether edge insulation is required is to attach a temperature sensor to the mid-height of the exterior edge of the specimen. Sufficient edge insulation is present if the edge temperature, T_e, satisfies the following requirement.

$$(T_e - T_m)/\Delta T < 0.05 \qquad (1)$$

6.6.1 *Secondary Guard*—To reduce heat exchange between the edges of the guarded-hot-plate and the environment, the guarded-hot-plate shall be outfitted with a co-axial temperature-controlled container referred to as the secondary guard. The secondary guard will be employed to adjust the ambient temperature to approximate the mean temperature of the test specimen.

6.6.1.1 *Size*—The secondary guard should have an inner dimension that is at least twice the dimension of the hot surface heater and the height should be equal to the thickness of the hot surface heater plus twice the thickness of the thickest specimen that will be tested.

6.6.1.2 *Materials*—The materials used in the construction of the secondary guard are not as critical as those selected for the hot and cold surface assemblies. However, the materials used in the design of the secondary guard shall be selected so that they are thermally stable over the intended temperature range, the heating element shall be capable of producing the necessary heat flux density to adjust the ambient temperature, and a means of cooling the secondary guard is required if the apparatus is intended for use at temperatures below the laboratory ambient. The use of high thermal conductivity metals is recommended for the construction since the secondary guard should be isothermal.

NOTE 9—Successful secondary guard designs consist of a sheathed heater wire or cable wrapped around an adequately-sized metal tube and pressed against the metal tube with another sheet of metal. For low-temperature operation, a cooling coil has been wrapped around the exterior surface of the secondary guard.

6.6.1.3 *Location*—The secondary guard shall be positioned around the hot surface assembly such that a uniform spacing is created between the components. The height of the secondary guard shall be adjusted such that the mid-height of the secondary guard is aligned with the center of the hot surface assembly thickness.

6.6.2 *Edge Insulation*—The interspace between the hot and cold surface assemblies, specimens and the secondary guard shall be filled with an insulating material. Due to the complex shapes of this interspace, a powder or fibrous insulation is recommended.

6.6.2.1 The selection of an edge insulation material will depend on the test conditions. Vermiculite is easy to use but should not be employed at temperatures above 540°C (1000°F) because it's thermal conductivity increases dramatically with temperature.

NOTE 10—Avoid the use of vermiculite when the guarded-hot-plate is used to evaluate specimens in different gaseous environments; vermiculite is extremely hygroscopic and the system is difficult to evacuate when it is used.

NOTE 11—Care shall be taken to ensure that there are no voids, pockets, or other extraneous sources of radiative heat transfer occurring at or near the guarded-hot-plate.

6.6.3 *Enclosure*—The guarded-hot-plate shall be placed inside an enclosure when the apparatus is used in to maintain a gaseous environment that is different than the laboratory ambient.

6.6.3.1 For low-temperature operation, a dry gas environment shall be used to prevent condensation from occurring on the cold surface assemblies and specimens.

6.6.3.2 For high temperature operation, it may be desirable to protect the apparatus from severe degradation by using a non-oxidizing gas.

6.6.3.3 The enclosure can also be used for substituting different gaseous environments and control of the ambient pressure.

6.7 *Clamping Force*—A means shall be provided for imposing a reproducible constant clamping force on the guarded-hot-plate to promote good thermal contact between the hot and cold surface assemblies and the specimens and to maintain accurate spacing between the hot and cold surface assemblies. It is unlikely that a force greater than 2.5 kPa (50 lb/ft^2) will be required for the majority of insulating materials. In the case of compressible materials, a constant pressure arrangement is not needed and spacers between the plates may be necessary to maintain constant thickness.

6.7.1 A steady force, that will thrust the cold surface assemblies toward each other can be imposed by using constant-force springs or an equivalent method.

6.7.2 For compressible specimens, spacers are required if the test thickness can not be measured by other means. The spacers shall be small in cross-section and located near the exterior perimeter of the primary guard. Avoid placing spacers on surfaces where underlying sensors are being used to measure plate conditions.

NOTE 12—Because of the changes of specimen thickness possible as a result of temperature exposure, or compression by the plates, it is recommended that, when possible, specimen thickness be measured in the apparatus at the existing test temperature and compression conditions. Gaging points, or measuring studs along the outer perimeter of the cold surface assemblies, will serve for these measurements. The effective combined specimen thickness is determined by the average difference in the distance between the gaging points when the specimen is in place in the apparatus and when it is not in place.

6.8 *Temperature Measurements:*

6.8.1 *Imbalance Detectors*—A suitable means shall be provided to detect the average temperature imbalance between surface plates of the metering section and the primary guard.

6.8.1.1 *Sensors*—The gap region shall be instrumented with temperature sensors to monitor and control the average temperature imbalance across the gap. Fine-gage thermocouples connected as thermopiles are often used for this purpose, although other temperature control sensors, such as thermistors, have been used. Highly alloyed thermocouples, rather than pure metals, should be used to maximize the thermal resistance across the gap. Because of nonuniform heat flux within the surface plates, temperature imbalance is not always constant along the gap perimeter. It has been found that with proper design the thermal conductance of the wires crossing the gap can be made relatively small and, therefore, a large number of thermocouples can be used to increase the gap imbalance sensitivity. It is not uncommon to use ten or more sensing elements.

6.8.1.2 *Sensitivity*—The detection system shall be sufficiently sensitive to ensure that variation in measured properties due to gap temperature imbalance shall be restricted to not more than 0.5 % of the metered section power, as determined experimentally or analytically.

NOTE 13—The sensitivity of many temperature sensors is reduced drastically at temperatures below the laboratory ambient. Particular care must be used in designing thermopile measurement systems to operate under these conditions.

6.8.1.3 *Location*—When using only a minimum number of sensing elements along the gap, the most representative positions to detect the average balance for a square plate are those at a distance from the corners equal to one-fourth of the side of the metering area. The corners and the axes should be avoided. For a round plate, the sensors should be spaced equally around the gap.

6.8.1.4 Electrically isolated gap imbalance sensors should be placed on both surface plates of the guarded heating unit to average the imbalance on both faces of the heating unit.

6.8.1.5 Thermal junctions or other sensitive elements should each be located in similar areas of the hot surface assembly. It is suggested that all junctions should be located at points directly adjacent to the centers of the areas between heater windings. Any leads crossing the gap should be thermally anchored to the primary guard to provide a heat sink from external thermal variations. In some instances it may be desirable to provide a heat sink for these leads outside the primary guard to minimize any radial heat flow.

6.8.2 *Temperature Sensors*—Methods possessing adequate accuracy, such as thermistors, thermocouples, diodes and precision resistance thermometers may be used for the measurement of temperatures in the apparatus. Thermocouples are the most widely used detector due to their wide range of applicability and accuracy. The goal is to measure the temperature gradient within the specimen, and the method chosen (sensors mounted on the specimen surface, in grooves, or between interior layers) should be that which yields the highest accuracy in the measurement of the temperature gradient. A discussion of these alternatives is provided in 6.8.2.3 and 6.8.2.4.

6.8.2.1 *Use of Thermocouples*—Precautions should be used to minimize spurious voltages in temperature control and measuring circuits. Spurious voltages, due to wire inhomogeneities, generally increase as the temperature gradients within the measuring leads increase. For the same reason, junctions between dissimilar metal leads should not be made in the regions of appreciable temperature gradients. Low thermal emf switches should be used in the temperature measurement circuits. An insulated, isothermal box of heavy sheet metal can be used when joining leads of dissimilar metals in the thermocouple circuit. It is recommended that all connections of thermocouple wire to copper wire be accomplished within the isothermal box in order that the junctions are at the same temperature; then the copper, not the thermocouple, leads are connected to the needed switching devices and/or voltmeters.

6.8.2.2 *Accuracy*—Thermocouples whose outputs are used to calculate thermal transmission properties shall be fabricated from either calibrated thermocouple wire or wire that has been certified by the supplier, and shall have a standard limit of error equal to or less than the specifications of Tables E 230. The resulting error in temperature differences due to distortion of the heat flow around the sensor, to sensor drift, and other sensor characteristics shall be less than 1 %.

6.8.2.3 *Methods of Attachment*—The surface temperatures of the specimens are most often measured by means of permanently mounted thermocouples placed in grooves cut into the surface plates. Precautions shall be taken to ensure that the thermocouple is thermally anchored to the surface being measured. This method of instrumentation is employed when the contact resistance between the specimen and the surface plates is a small fraction of the specimen thermal resistance. The hot- and cold-surface assembly plate sensors on each side are sometimes connected differentially. Thermocouples mounted in this manner shall be made of wire not larger than 0.6 mm in diameter for large apparatus and preferably not larger than 0.2 mm for small apparatus.

NOTE 14—This method of deploying thermocouples is traditionally used for compressible specimens and for rigid specimens possessing flat surfaces that have a thermal resistance of greater than 0.2 m^2 K/W (1 h ft^2 F/Btu) at ambient conditions.

NOTE 15—For rigid specimens not satisfying the requirements of 6.8.2.2, two techniques for attaching temperature sensors are recommended. Small grooves may be cut into the surfaces of the specimens and thermocouples can be affixed into these grooves. As an alternative, thermocouples may be installed onto the surfaces of the specimen and thin sheets of a compressible homogeneous material interposed between the specimen and surface plates. In this latter case, an applied force should be used as indicated in 6.7 to ensure sufficient surface contact. For either of these applications, thermocouples shall be made of wire not larger than 0.2 mm in diameter.

6.8.2.4 *Electrical Isolation*—Temperature sensors can be either completely insulated electrically from the surface plates or grounded to the surface plate at one location. Consequently, thermocouples connected differentially can only have a single junction ground. Computations or experimental verifications, or both, shall be performed to confirm that other circuits do not affect the accuracy of the temperature measurements.

6.8.2.5 *Number of Sensors*—The number of temperature sensors on each side of the specimen in the metering area shall not be less than $10 \times \sqrt{A}$, or 2, whichever is greater.

NOTE 16—It is recommended that one temperature sensor be placed in the center of the metered section and that additional sensor be uniformly distributed radially.

6.9 *Thickness Measurements*—A means shall be provided for measuring the thickness of the specimen, preferably in the apparatus, to within 0.5 %.

6.10 *Metered Section Power Measurement*—Dc power is highly recommended for the metered section. Ac power may be used but the user should note that ac power determinations are more prone to error than dc measurements. The power to the metered section is determined with a wattmeter or from voltage and current measurements across the heater in the metered section. The voltage taps for this measurement should be placed to measure the voltage from the mid-point of the gap. The current can be determined from the voltage drop across a precision resistor placed in series with the metered section heater.

6.11 *Electrical Measurement System*—A measuring system having a sensitivity and accuracy of at least ±0.1 K shall be used for measurement of the output of all temperature and temperature difference detectors. The system shall have sufficient sensitivity to measure the gap imbalance to a level equal to 1 % of the imbalance detector output that satisfies the requirement of 6.8.1.2. Measurement of the power to the metered section shall be made to within 0.2 % over the entire operating range.

6.12 *Performance Checks*—When a new apparatus is commissioned or an apparatus has undergone significant refurbishment, a series of careful checks shall be performed before initiating routine testing.

6.12.1 *Planeness*—The planeness of each surface plate shall be measured. See 6.3.1.1.

6.12.2 *Temperature Measurements*—With specimens installed in the apparatus, the coolant supply to the cold surface assembly shut off, and no electrical power being supplied to any of the heaters, mount the apparatus inside the enclosure. Allow the system sufficient time to come to thermal equilibrium. With no energy being supplied to the apparatus, note the output of all of the temperature sensors. The temperature sensors shall have an output that agrees to within the uncertainty prescribed in 6.8.2.2. The output of the imbalance detection circuit shall be within the noise level of the electrical measurement system.

6.12.3 *Imbalance Detection*—Determine the maximum imbalance that can be allowed that satisfies the requirements in 6.8.2.2. With the apparatus energized and operating normally, note the thermal resistance of a specimen and the imbalance detector output at equilibrium. Repeat the test at various levels of imbalance. Linearly fit the thermal resistance data as a function of bias. The slope of this relationship will define the maximum imbalance detector output that can be allowed during routine operation.

NOTE 17—The number of bias levels that need to be analyzed will depend on the quality of the curve fit; the scatter within the data set, as defined by twice the standard deviation, shall be less than the noise level of the electrical measurement system as defined in 6.11.

6.12.4 *Edge Heat Losses*—Edge heat losses give rise to the greatest measurement errors when the specimens approach the maximum specified thickness and thermal resistance. This series of experiments will determine which edge loss strategies must be employed to maintain edge losses to levels prescribed by this method.

6.12.4.1 Install specimens in the apparatus that approach the apparatus limits described above and instrument these specimens with the edge temperature sensors described in 6.6. Do not install any components described in 6.6 to reduce edge heat loss. While performing a test, verify that the difference between the specimen mean temperature and edge temperature satisfy the requirements of 6.6. Add additional edge loss apparatus components (edge insulation, secondary guard, enclosure) until the requirements of 6.6 are satisfied. These experiments will define the required levels of edge loss that shall be incorporated into the routine testing. In extreme cases, the secondary guard may have to be biased to satisfy these requirements; include these biases as part of the routine test procedure.

6.12.5 *Emittance of Surface Plates*—The emittance of the surfaces can be experimentally verified by testing an air gap, where the thickness of the air gap is limited to prevent the onset of convection. The heat flow rate per unit temperature difference is the sum of the thermal conductance of air and $4\sigma T_m^3 (2/\epsilon-1)$. A best fit of the plot of the heat flow rate per unit temperature difference and the inverse of the air space thickness supplies both the thermal conductivity of the air and $4_n T_m^3 (2/\epsilon-1)$. From this plot, the plate emittance can be verified **(42)**.

6.12.6 *Overall Design Verification*—When all of the other checks have been successfully completed, tests shall be performed on specimens that are traceable to a national standards organization. These tests shall cover the range of temperatures for which the apparatus has been designed. Verification of the apparatus may be limited by the temperature range of available standards. See 5.7.

7. Specimen Preparation and Conditioning

7.1 *Specimen Selection*—Only those specimen selection factors important to the performance of the apparatus are considered here. Factors related to the specimens' thermal properties are typically described in material specifications. When two specimens are required, the specimens should be selected to be as similar in thickness and thermal characteristics as possible. The use of Test Method C 518 can be used to check the similarity of the specimens' thermal characteristics.

7.1.1 *Thickness*—The maximum specimen thickness that can be measured to a given accuracy depends on several parameters, including the size of the apparatus, thermal resistance of the specimen, and the accuracy desired. To maintain edge heat losses below approximately 0.5 %, for a guard width that is about one-half the linear dimension of the metered section, the recommended maximum thickness of the specimen is one-third the maximum linear dimension of the metered section. For more specific quantitative information on this limitation see Refs **(1,5,7,8)** and adjunct material given in this test method.

7.1.2 *Size*—The specimen shall be sized to cover the entire metered section and guard area when possible. It is desirable to cover the gap between the guarded-hot-plate and the primary guard when sample size is limited. The guard portion of the volume between the heating and cooling plates should be filled with material having similar thermal conductance characteristics as the specimen. When the specimen has a high lateral conductance such as a dense solid, a gap between the metered section and the primary guard shall be provided within the specimen. Refer to 7.2.3 for special precautions.

7.1.3 *Homogeneity*—Specimens exhibiting appreciable inhomogeneities in the heat flux direction shall not be tested with this method. There are two potential problems in attempting to determine the heat flux through highly inhomogeneous specimens. One is related to the interpretation and application of the resulting data, see Practice C 1045. The other is the degradation in the performance of the apparatus. If the specimen is highly inhomogeneous, that is, the heat flux varies appreciably over the metered section, several errors can be significantly increased. The plate temperature distribution can deviate appreciably from isothermal conditions which, in turn, can cause large uncertainties in the average temperature difference across the specimen. The increased plate temperature variations can also lead to increased gap and edge heat losses. The importance of measuring the plate or specimen surface temperatures at numerous points is greatly increased under such conditions.

7.2 *Specimen Preparation*—Prepare and condition the specimens in accordance with the appropriate material specification. Use the following guidelines when the material specification is unavailable. In general, the surfaces of the specimen should be prepared to ensure that they are parallel with and have uniform thermal contact with the heating and cooling plates.

7.2.1 *Compressible Specimens*—The surfaces of the uncompressed specimens may be comparatively uneven so long as surface undulations are removed under test compression. It may be necessary to smooth the specimen surfaces to achieve better plate-to-specimen contact. If the apparent thermal conductivity of the contact void is greater than that of the specimen, compressible or otherwise, the measured heat flux will be greater than the heat flux that would be obtained if the voids were absent. This may often be the case at higher temperatures where radiant heat transfer predominates in the void. For the measurement of compressible specimens, the temperature sensors are often mounted directly in the plate surfaces. Also, plate spacers may be required for the measurement of compressible specimens.

7.2.2 *Rigid and High Conductance Specimens*—The measurement of rigid specimens or high conductance specimens requires careful surface preparation. First, the surfaces should be made flat and parallel to the same degree as the guarded-hot-plate. If the specimen has a thermal resistance that is sufficiently high compared to the specimen-to-plate interface resistance, temperature sensors mounted in the plates may be adequate. However, for materials such as plastics or ceramics, when the thermal conductivity of the material exceeds 0.1 W/m·K, the following techniques shall be used to ensure accurate surface temperature measurement.

7.2.2.1 In some cases it is necessary to mount the temperature sensors directly on the specimen surfaces or in grooves in the specimens. Under vacuum conditions, the slightest space between plate and specimen is essentially an infinite thermal resistance (except for radiative heat transfer). Under these conditions extreme heat flux nonuniformities will occur. In any event the user should always try to minimize the ratio of contact resistance to specimen resistance and to strive for a constant ratio over the entire surface.

7.2.2.2 Another potential solution (that must be used with caution) is to mount a compressible thin sheet (for example, a soft rubber or thin fibrous pad) between the plates and specimen to improve the uniformity of the thermal contact. When this procedure is used, temperature sensors shall be instrumented in or on the surface of the specimens to ensure accurate temperature measurement of the specimen surface. An applied force should be used as in 6.7 to ensure sufficient surface contact.

7.2.3 *Anisotropic Specimens*—Specimens that have a high lateral to axial conductance ratio require that a low conductance gap be created in the specimen directly in line with the gap between the metered section and the primary guard.

7.2.4 *Loose-Fill Specimens*—The measurement of loose-fill specimens requires special handling, conditioning, and measurement techniques. The user is directed to Practice C 687 for details.

7.3 *Specimen Conditioning*—Condition the specimens either as stated in the material specification or where no guideline is given, at 22 ± 5°C (72 ± 3°F) and 50 ± 10 % relative humidity for a period of time until less than a 1 % mass change in 24 h is observed.

NOTE 18—Specimens can be conditioned at different conditions in order to determine the effect on the thermal properties of the specimens. Conditioning environments shall be reported with the test results.

8. Procedure

8.1 For a double sided test, select a pair of test specimens as outlined in Section 7.

8.2 Measure and record the specimen mass and dimensions. Also see 8.12.

8.3 Install the specimen into the apparatus at the desired test thickness.

8.4 Install the appropriate secondary guarding and an environmental chamber (as required).

8.5 If the test is to be conducted with gases other than air in the specimen-plate assembly, purge the environmental chamber and backfill with the desired gas. Care should be taken to limit the pressure of the fill-gas to below its condensation point at the lowest temperature expected within the chamber. Since the measured heat flux is dependent on both the type of fill gas and pressure, record both of these parameters.

8.6 Adjust the heating and cooling systems to establish the desired test conditions. For guidance in establishing test temperatures, refer to Practice C 1058. The ambient temperature should be the same as or slightly above the mean temperature of the test. This may require the use of a temperature controlled surrounding. This can be accomplished utilizing a controlled perimeter heater and insulation materials to aid in the control of the surrounding temperature.

8.7 Record the start time and date of the test. Begin data acquisition. The recorded data shall include: the date and time of data acquisition; power to the guarded-hot-plate; hot side guarded-hot-plate surface temperature; hot side guard temperatures; cold surface assembly temperatures; controlled environment ambient temperature and relative humidity; temperature difference or thermopile output across the gap between the guard and metered section; and calculated heat flux and estimated thermal property of interest.

NOTE 19—Thermal steady-state is the time required for the test apparatus to stabilize. This varies considerably with the apparatus design, specimen to be measured, and test conditions. Generally, however, the stabilization time is on the order of hours. Stabilization times generally increase with thick specimens, specimens with low thermal diffusivity and is dependent on the mass of the metered section area. Measurements in a vacuum and on microporous materials create small monotonic changes over a long period of time and may take longer to stabilize.

8.8 Thermal steady state must be achieved for this test method to be valid. To determine if steady state is achieved, the operator must document steady state by time averaging the data, computing the variation and performing the following tests on the data taken in Section 8.

8.8.1 Thermal steady state for the purpose of this test method is defined analytically as:

8.8.1.1 The temperatures of the hot and cold surfaces are stable within the capability of the equipment at the test conditions. Ideally an error analysis will determine the magnitude of the allowable differences, however the difference is usually less than 0.1 % of the temperature difference.

8.8.1.2 The power to the metering area is stable within the capability of the equipment. Ideally an error analysis will determine the magnitude of the allowable differences, however the difference is usually less than 0.2 % of the average result expected.

8.8.1.3 The required conditions above exist during at least four intervals 30 min in duration or four system time constants, whichever is longer.

NOTE 20—The thermal time constant of the system is the time required to come to within 1/*e* (37 %) of the fixed value after a step thermal disturbance of the system. The thermal time constant in the constant power mode is the time required to come to within 37 % of the final temperature. The thermal time constant in the constant temperature mode is the time required to come to within 37 % of the final power. The thermal time constant of a system can be approximated from the thermal diffusivities of the system components, but is generally determined experimentally.

8.9 After achievement of the desired steady-state as defined in 8.8.1, three successive repeat data acquisition runs shall be completed. These runs shall be conducted at intervals of at least 30 min and should not be less than the thermal time constant of the system (see Note 20). This combination of three runs shall be considered a valid test if each datum obtained for each measured variable meets the following criteria.

8.9.1 The data do not differ from the mean by no more than the uncertainty of that variable, see A1.5.

8.9.2 The data obtained does not change monotonically with time. This is determined by comparing the average result of the final three test periods to the averages of the previous four periods. Graphing of the test parameters versus time or monitoring the slope of the data are techniques for determining monotonic conditions.

8.9.3 If the data continues to drift, the test shall be considered incomplete and further data acquisition sets shall be conducted until thermal steady state is achieved. Drift, even at low levels, may indicate that either the specimen characteristics are changing or the system is not at steady-state. For further details see Refs **(3,12,13)**.

8.10 Prior to terminating the test, measure and record the pressure of the chamber.

8.11 Upon completion of the thermal test outlined above, remove the specimen and examine the system components, such as temperature sensor mounting, for proper placement and operation.

8.12 Determine the specimen thickness and weight after the test to ensure that they have not changed from the initial condition. Record any changes in the physical characteristics of the specimen.

9. Calculation

9.1 The primary data required for this test method include electrical power, surface temperatures, area, and thickness. Of these, only thickness is generally a directly measured quantity. The others are either calculated from other more fundamental measurements or are converted by an electrical device. The manner in which these variables can be obtained is discussed in 8.9 and below.

9.2 *Heat Flow*—The heat flow to be reported is that which passes through each specimen. This is equal to the power generated by the metered section heater. For the double-sided mode of operation, only one-half the power generated by the heater flows through each specimen. Determine the power, Q, from emf, E, and current, I, and calculate as follows:

$$Q = E \times I \tag{2}$$

9.3 *Metered Section Area*—Determine the metered section area, A, from the area, A_m, of the guarded-hot-plate and the gap area, A_g. If there is no discontinuity in specimen characteristics in the gap region, the metered area is calculated as follows:

$$A = A_m + \frac{A_g}{2} \tag{3}$$

For high precision measurements, this assumption that the gap contributes half of its area to the effective metered section area, A, may need to be verified for the particular apparatus used. If there is a discontinuity between the specimen in the metered section and the guard region, this equation is modified slightly, as in ISO 8302, to include the effect of heat flux distortion in the gap region:

$$A = A_m + \frac{A_g \lambda_g}{2\lambda} \tag{4}$$

Where significant expansion, or contraction, of the guarded-hot-plate is known during a test, appropriate corrections to the area shall be made.

9.4 *Heat Flux*—The heat flux is obtained from the ratio of the heat flow, Q, and the total metered section area, A, and is calculated as follows:

$$q = \frac{Q}{A} \tag{5}$$

9.5 *Temperature*—Electrical readings from the temperature sensors are normally converted to temperature using a mathematical equation based on either the sensor's calibration curve or an appropriate reference such as a thermocouple voltage table.

9.6 *Density*— The metered section area specimen density, ρ_m, or the sample density, ρ_s where metered section area density cannot be obtained, are to be reported as the average of the two pieces. The equation for density, is the following:

$$\rho_m = \frac{m}{A \times L} \tag{6}$$

or:

$$\rho_s = \frac{m_s}{A_s \times L}$$

9.7 *Thermal Transmission Properties*—These properties shall be reported only in accordance with the requirements and restrictions of Practice C 1045.

10. Report

10.1 To be in conformance with this test method, report the following:

10.1.1 The report shall be identified with a unique numbering system to allow traceability to the individual measurements taken during each test performed,

10.1.2 The average values as obtained from the test. Standard deviation about that average. The results may be reported in a form similar to that shown in Fig. 3,

10.1.2.1 Identification of the test organization, responsible person in charge, test operator (optional) and the test sponsor,

10.1.2.2 The generic name, or other identification required to provide a complete and detailed description of the tested material. For hygroscopic materials, such as concrete and wood, the moisture content should also be given,

NOTE 21—A generic description in addition to the brand name should be reported where possible.

10.1.2.3 Information regarding the specimen preconditioning,

10.1.2.4 Variables that effect thermal transmission prop-

Test Report

Date:

Test Report Number:

Operator:

Duration of Test:

Specimen Identification:	Product, name, manufacturers description.
Specimen Characteristics:	Unique characteristics such as degree of homogeneity or anisotropy, density (optional).
Specimen Conditioning:	Temperature, time, humidity.
Specimen Dimensions and Mass:	Before and after conditioning and after measurement.

Apparatus Description:	Size, shape and orientation of plates. Single or double-sided operation, description of secondary guarding, unique procedures.

Experimental Results

		Uncertainty	
Variable	Measured Value	Systematic	Random
Q, W			
Th, K			
Tc, K			
Tm, K			
$\Delta T, K$			
A, m^2			
L, m			
Fill gas pressure, Pa			
Other			

Derived thermal transmission properties including the applicable range of conditions shall be in conformance with Practice C 1045.

FIG. 3 Example Test Report Form

erties, such as fill-gas and pressure, shall be specified when applicable,

10.1.2.5 The dimensions of the metered section and guard(s) and their relationship to the overall specimen dimensions (m). The plate emittance,

10.1.2.6 Specimen orientation and the direction of heat transfer during the test,

10.1.2.7 The total area of the specimen (m^2),

10.1.2.8 The specimen density of the metered section area or sample density where metered section area density cannot be obtained (kg/m^3),

10.1.2.9 The thickness of the specimen(s) within the metered section (m),

10.1.2.10 The area averaged temperatures of both hot and cold specimen surfaces (K),

10.1.2.11 Net steady-state average heat flux through the specimen (W/m^2),

10.1.2.12 Any thermal transmission properties calculated and reported and their estimated error, and

10.1.2.13 The test date and time, the time required for steady temperature conditions, the time to reach steady-state, the data acquisition time period, frequency of data collection and the end date and time.

10.2 The following is optional information that may be included in the report:

10.2.1 Values for guard loss, back side energy loss and other losses included in the net energy calculation (W/m^2), and

10.2.2 A full description (or references) of test procedures and data analysis techniques used.

10.3 When certification of the test results is required, include the date of the latest apparatus verification and a description of the procedures used. References for the verification report(s) shall also be included. Where applicable, include a statement of laboratory accreditation of the test facility used, including date of latest inspection.

10.3.1 Where agreed upon between the customer and the test laboratory, less maybe reported but the remainder of the results shall be made available.

NOTE 22: **Caution**—Where this test method might be specifically referenced in published test reports and published data claims, and where deviations from the specifics of the test method existed in the tests used to obtain said data, the following statement shall be required to accompany such published information: **"This test did not fully comply with following the provisions of Test Method C 177."** This statement shall be followed by a listing of specific deviations from this test method and any special test conditions that were applied.

11. Precision and Bias

11.1 This section on precision and bias for the guarded hot plate apparatus includes a discussion of; general statistical terms; statistical control; factors affecting test results; ruggedness tests; interlaboratory comparisons conducted by ASTM Committee C-16; proficiency testing conducted under the auspices of the National Voluntary Laboratory Accreditation Program (NVLAP); and error propagation formulae.

11.2 *General Statistical Terms*—The accuracy of a test result refers to the closeness of agreement between the observed value and an accepted reference value. When applied to a set of observed values, the accuracy includes a random component (imprecision) and a systematic component (bias). The variability associated with the set of observed values is an indication of the uncertainty of the test result. Additional information on statistical terminology is available in Terminology E 456.

11.3 *Statistical Control*—The user of the guarded-hot-plate apparatus shall demonstrate that the apparatus is capable of performing in a consistent manner over time **(35)**. The use of control charts (see Manual 7 **(34)**) to monitor the operation of the guarded hot plate is one recommended way to monitor the control stability of the apparatus. When possible, it is recommended that a reference material traceable to a national standards laboratory be used as the control specimen. Ideally, the long-term variation should be no greater than the short-term variability.

11.4 *Factors Affecting Test Results*—Experiments and theoretical analyses have identified two principal (systematic) errors that affect the operation of an idealized guarded hot plate apparatus. These errors are edge heat flows at the periphery of the specimens; and, heat flow across the gap due to a thermal imbalance. Other errors studied include the effect of gap width on the heat flow; and, the proper determination of the metered section area. These errors and others are discussed in detail in A1.3.

11.4.1 *Edge Loss Errors*—These have been found to depend on the size (and type) of the guard, the specimen thermal conductivity and thickness, and ambient temperature **(7,18,20,21,31,33)**. By using a sufficiently wide guard (see Section 6), appropriate levels of edge insulation, and proper selection of the ambient temperature (see Section 8), the edge loss error can be reduced to a negligible value relative to the specimen heat flow (see A4.2). There is only limited experience (at room temperature) with measurement of apparent conductivity at large thickness' (above 30 cm), but errors may be expected to be above 2 %, especially if the user does not reduce the problems associated with long time constants and large lateral heat flows **(31)**.

11.4.2 *Gap Imbalance Error*—These have been found to depend on several parameters including the temperature difference, the gap geometry, the structural support system, the wires crossing the gap (number, size, and type), the gap fill material (gas or insulation), the emittance of the gap surfaces, and the specimen material in the vicinity of the gap **(5,6,8,18,22,36)**. The resulting heat flow due to a temperature imbalance can be obtained either by calculation based on the above parameters or empirical data. An empirical relationship for the gap heat flow can be determined by purposely introducing a temperature imbalance across the gap and measuring the resulting change in the specimen heat flow (see A1.4.3).

11.5 *Ruggedness Tests*—The results of one ruggedness study for a 200 mm^2 guarded hot plate and two materials having different thermal conductivity's have been reported **(37)**. Matched pairs, 85 mm thick, of polyurethane foam and silicone rubber were measured at a mean temperature of 297 K and a temperature difference of 23 K. For each specimen, the width of edge insulation was set at one of five levels (0, 12.7, 25.4, 50.8, and 76.2 mm) while the ambient temperature was varied at one of three levels. The results indicate that the edge losses are reduced with edge insulation but only become zero when the ambient temperature is at one specific value. The optimum ambient temperature appears to be a function of specimen thickness and thermal conductivity, and edge insulation thickness.

NOTE 23—As noted in Section 8, the value of the ambient temperature is set to either the same value as the mean temperature of the test or a value slightly above the mean temperature. The user should determine the optimum value for their apparatus and test conditions by using the sensitivity analysis described in A4.2. This dependence may change appreciably for different specimens or apparatus conditions and, therefore, should be done under typical test conditions.

11.6 *Interlaboratory Tests*—The results of three published interlaboratory tests for guarded-hot-plate apparatus are discussed below. The results, where appropriate, state an index of precision (between laboratory) of two-standard deviation limits (2s). Certain aspects of the interlaboratory tests were not conducted completely in accordance with the requirements of Practice E 691, for example, the number of test laboratories was less than six in one study and none of the studies required replicates. Furthermore, a study involving a variety of materials is needed. Consequently, a general statement for the index of precision and bias that covers all conditions and materials is unavailable. In the interim, the user is directed to the interlaboratory tests if information on precision and bias is needed (see Practice C 687 for loose-fill materials).

11.6.1 In 1951, results of an interlaboratory comparison were reported **(38)** for 20 guarded-hot-plate apparatus from 17 laboratories. The plates ranged in size from 200 to 600 mm square. Different (numbered) pairs of corkboard (25 mm thick) were measured by each laboratory at a mean temperature from 266 to 322 K. The data from 15 of the 20 apparatus (75 %) were within ±3 % of the mean value as determined by the National Bureau of Standards (now the National Institute of Standards and Technology). The maximum deviations were +13 and −16 %.

11.6.2 In 1985, results of a third round of interlaboratory comparisons were reported **(41)** for five large guarded-

TABLE 1 NVLAP Proficiency Tests for Guarded-Hot-Plate Apparatus Ref (39)

Material	Nominal Thickness, mm	Thermal Conductivity Group Mean, W/(m K)	Number of Labs	Coefficient of Variation, %	Round
Expanded polystyrene board	25	0.037	6	1.80	10
Foam Board, rigid	25	0.040	9	2.52	4
Glass-fiber batt	25	0.040	10	2.15	5
Glass-fiber batt	25	0.040[A]	6[A]	2.26[A]	7[A]
Glass-fiber batt	25	0.039[A]	7[A]	2.82[A]	3B[A]
Glass-fiber batt	25	0.040	9	3.28	3A
Glass-fiber batt	25	0.040	7	3.43	7
Glass-fiber batt	25	0.040	9	4.66	3B
Glass-fiber batt, foil faced	25	0.032	9	0.98	6
Glass-fiber batt, foil-faced (stacked)	50	0.033	7	1.45	9
Glass-fiber batt, foil faced	25	0.032	8	1.95	8

[A] Recalculation with one or more laboratories excluded from the group statistics because their test results deviated from the pre-characterized value by more than 6 %.

hot-plate apparatus ranging from 610 to 1219 mm^2 and 1016 mm diameter (the last apparatus mentioned being a circular line-heat-source guarded-hot-plate). The same specimens of fibrous-glass blanket (16 kg/m^3) were circulated to each laboratory. Matched pairs were tested at 297 K and thicknesses of 25.4, 50.8, 76.2, and 101.6 mm. Imprecision of the data versus a semi-empirical model for a density range of 11 to 20 kg/m^3 were 1.9, 2.3, 2.6, 2.9 % (2s level) at thicknesses of 25.4, 50.8, 76.2, 101.6 mm, respectively.

11.6.3 In 1988, results of a interlaboratory comparison were reported **(30)** for seven high-temperature guarded-hot-plate apparatus. The plates ranged in size from 203 to 406 mm in diameter and 300 to 610 mm^2. Different matched pairs of fibrous alumina-silica and calcium silicate were measured by each laboratory over a mean temperature range from 330 to 701 K. Reference equations based on NIST-Boulder corrections were fit to the data. Imprecision in the deviations from the model were 15 and 16 % (2s level) for fibrous alumina-silica and calcium silicate, respectively. It was established that a significant percentage of the standard deviation in this comparison was due to material variability and not apparatus error.

11.7 *Proficiency Tests*—In 1985, the results of a series of proficiency tests conducted for NVLAP over a four-year period were reported **(39)** for guarded-hot-plate apparatus (plate size not reported). Different specimens of four thermal insulation materials were distributed to each laboratory for testing. The materials were expanded polystyrene; foam board; low-density glass-fiber batt (8 to 16 kg/m^3); and, high-density glass-fiber batt, foil-faced (64 kg/m^3). Each laboratory reported a single test result, that is, no replicates were conducted. Results of the proficiency tests are summarized in Table 1. The index of precision (between laboratory) is expressed as a percentage for the one-standard deviation limit(s) divided by the mean of the test result, or one-coefficient of variation (CV %).

11.8 *Error Propagation*—Several formulae are available **(40)** for determining the apparatus uncertainty by error propagation. For guidelines on using a standard procedure, the user is referred to ISO Guide to the Expression of Uncertainty in Measurement **(32)**. Strictly speaking, determining a statement of uncertainty for a test result requires treating random and systematic errors separately. A description of random and systematic errors and possible sources of error are discussed below.

11.8.1 *Random Error,* δ_r—In a measurement, random errors (imprecision) are considered to be the sum total of all the small (negligible) independent errors that are uncontrolled, for example small fluctuations in environmental conditions or plate temperatures. Random errors are assumed normally distributed, uncorrelated, and preferably small. In general, random errors are a function of the capabilities of the control system and, to a lesser extent, the measurement system.

11.8.2 *Systematic Error,* δ_s—A systematic error (bias) is a fixed deviation that is inherent in each and every measurement. If the magnitude and direction of the systematic error are known, the user can make appropriate correction(s) to the measured value. Under such circumstances a justification for the correction should be provided. In general, the magnitude of the error, $|\delta_s|$, is estimated by experience or judgment.

11.8.3 *Statement of Uncertainty*—The statement of uncertainty requires an expression having credible limits for its inaccuracy. Different traditions and usage have resulted in different expressions of uncertainty that may be summarized as follows: both imprecision and bias negligible; imprecision negligible, bias not negligible; neither imprecision nor bias negligible; and, imprecision not negligible, bias negligible.

11.8.4 *Sources of Errors*—The uncertainty of the apparatus as determined by propagation of errors should consider the error in each of the separate measurements used to determine the test result. For a guarded-hot-plate apparatus, these errors in measurements are the uncertainty in: heat flow δQ; temperature difference, $\delta \Delta T$; metered section area, δA; and specimen thickness, δL. These errors and an example are discussed in A1.3.

12. Keywords

12.1 error analysis; guarded-hot-plate; heat flow; heat flux; steady-state; thermal conductivity; thermal resistance; thermal transmission; thermal conductance; thermal testing

ANNEX

(Mandatory Information)

A1. THICKNESS MEASUREMENT, LIMITATIONS AND MEASUREMENT UNCERTAINTY

A1.1 *Importance of the Thickness of the Insulation Specimens in Guarded-Hot-Plate Measurements*—The thickness of the specimen as installed in the apparatus determines both the density of the material and the temperature gradient applied to it during the measurement of the thermal property. If the thickness of a specimen is changed from its room-temperature value by thermal effects (thermally reversible expansion or contraction, or thermally induced irreversible shrinkage or expansion of the specimen), or by compression, then use of the room-temperature thickness outside the apparatus will lead to error in the determination of the apparent conductivity (or resistivity) of the specimen. A given relative (percentage) error in the thickness leads to an equal relative error in the determination of the conductivity. For measurements of thermal properties at mean specimen temperatures near room temperature the error in neglecting any changes in thickness may be negligible, but this can be ascertained only by observation in the specific case at hand.

A1.2 *Suggested Ways to Measure Thickness of Incompressible Specimens*—In determining the thickness of a specimen, one assumes that it is properly shaped, so that the measured thickness is valid. However, two different situations may sometimes occur to affect the thickness measurement. The shape of the specimen may be distorted by warping or bowing at the time it is first installed in the apparatus. In this case, either the (flexible) specimen should be compressed enough to remove the distortion when installed, (or, preferably, a specimen of better quality should be selected). Independent of, the specimen may undergo a change of shape as it is subjected either to high mean temperatures or to large temperature gradients, due to chemical changes occurring in the specimen at high temperatures. In this case it is difficult to define what the thickness of the specimen actually is during the measurement. The thickness of the specimen should be measured both before and after the thermal transmission property is measured, to show whether such dimensional changes are occurring. Any warping or bowing of the specimen, before or during measurement of thermal properties, adds to the uncertainty in the value of thickness. Some materials such as polymers have large coefficients of expansion and the material tends to bow unless a small thickness and temperature difference across the specimen is used.

A1.2.1 The recommended procedure for measuring specimen thickness is to measure the thickness while installed in the apparatus. This is necessary if the correct temperature gradient actually applied to the specimen during the measurement of the thermal property is to be obtained. Rigid rods may be securely installed extending laterally from the outer edges of the metered area/primary guard assembly, at two or three equally spaced locations along the circumference of the plate. The portion of the rod extending from the plate should be smooth and parallel to the plane of the plate surface. Alternatively, the plates may be machined with flat, horizontal plates extending from the circumference. Similar rods (or plates) are likewise located on each auxiliary heater plate, at the same circumferential positions, vertically (within 5° of arc) above or below the rods on the metered area/primary guard assembly.

A1.2.1.1 With no specimens installed, with the heater plates contacting each other in their usual order, and taking care not to change the plate separation, measure the separation between each vertical pair of rods on two adjacent plates with a vernier calliper. Compute the arithmetic mean of the plate separation for each pair of adjacent plates. Then, with specimens installed between the plates in the apparatus, and with the usual mechanical loading applied, measure the separation between the pairs of rods on adjacent plates, taking care not to change the plate separation. Compute the arithmetic mean. Subtract the mean separation obtained with no specimen from the mean separation with the specimen present, for corresponding pairs of plates, to obtain the as-installed thickness of each specimen. The standard deviation about the average of values from repeated measurements of the plate separation, starting from total disassembly, gives a statistical measure of the reproducibility. If contact cannot be made between the plates, standard spacers can be inserted between the plates. Bringing the plates in contact with the spacers can determine the adjustment in specimen measured thickness required.

A1.2.1.2 The accuracy of this procedure is equal to the imprecision with which the vernier can be read. The accuracy of this test method depends on the precision with which the rods are mounted in a true horizontal orientation, and on not changing the plate separation during the measurement. The standard deviation about the average of values from repeated measurements of the plate separation, starting from total disassembly, gives a statistical measure of the reproducibility.

A1.2.2 An alternative is to place the specimen on a flat surface and measure the thickness at various points across the specimen with a thickness gage mounted above the specimen. The zero is first established by resting the foot of the gage on the flat surface. The specimen is then measured. This procedure has the advantage that specimen flatness and warp can be measured. Thickness is measured typically in at least five different locations across the full specimen and within the metered section to establish the metered thickness within the apparatus. The thickness, when applicable, should be measured after the test to monitor any significant changes that may have effected the results.

A1.2.2.1 The accuracy of this test method is equal to the imprecision with which the gage can be read. The accuracy and reproducibility of this test method depends on the ability of the operator to reproduce the amount of force exerted on the specimen especially in the case of compressible specimens.

A1.2.3 Another alternative is to use a micrometer or vernier calliper. This assumes that the specimen is not bowed or warped, that should of course be ascertained. During a measurement of thickness with a calliper, prevent the narrow jaws of the measuring tool from penetrating into the surface of the specimen. Cut two small pieces of flat, rigid rectangular metal sheet, about 20 by 40 mm and 0.5 to 1.0 mm thick. Measure the combined thickness of the two metal rectangles; then measure the thickness of the specimen while holding one metal piece under each jaw, between the surface of the specimen and the jaws of the micrometer or calliper. Be sure to subtract the combined thickness of the two metal plates from the total thickness of specimen plus metal pieces, to obtain the net specimen thickness. By this method measure the thickness at eight different, equally spaced locations around the outer margin of the specimen.

A1.2.3.1 The accuracy of this procedure is equal to the precision with which the vernier (or micrometer) can be read. The accuracy and reproducibility of this test method is lower than that described above in A1.2.1 and A1.2.2, due to the variable pressure used by different people in measuring the specimen between the jaws of the micrometer or calliper.

A1.3 *Limitations Due to Apparatus:*

A1.3.1 *Limitations Due to Contact Resistances*—When testing a rigid specimen of high thermal conductance (that is, specimens of a material too hard and unyielding to be appreciably altered in shape by the pressure of the heating and cooling units), even small, non-uniformities of the surface of both the specimen and the apparatus (surfaces not perfectly flat) will allow contact resistances not uniformly distributed between the specimens and the plates of the heating and cooling units.

A1.3.1.1 These will cause nonuniform heat flow-rate distribution and thermal field distortions within the specimens; moreover, accurate surface temperature measurements will be difficult. For specimens having thermal resistances less than 0.1 m^2 K/W, special techniques for measuring surface temperatures will be required. Metal surfaces should be machined or cut flat and parallel and stress-relieved.

A1.3.2 *Upper Limits for the Thermal Resistance:*

A1.3.2.1 The upper limit of thermal resistance that can be measured is limited by the stability of the power supplied to the metered section, the ability of the instrumentation to measure power level and the extent of the heat losses or gains due to temperature imbalance errors between the central and guard sections of the specimens and of the metered section.

A1.4 *Limits to Temperature Difference:*

A1.4.1 Providing uniformity and stability of the temperature of the hot and cold surfaces of the plates, the noise, resolution and temperature measurements can be maintained within the limits outlined in Section 6, temperature differences as low as 5 K, when measured differentially, can be used. Lower temperature differences shall be reported as not complying with this standard. See Practice C 1058.

A1.4.2 If temperature measurements of each plate are made by means of thermocouples with independent reference junctions, the accuracy of the calibration of each thermocouple may be the limiting factor in the accuracy of measured temperature differences. In this case, it is recommended that temperature differences of at least 10 K to 20 K are used in order to minimize temperature-difference measurement errors.

A1.4.3 Higher temperature differences are limited only by the capability of the apparatus to deliver enough power while maintaining required temperature uniformity.

A1.4.4 *Maximum Specimen Thickness:*

A1.4.4.1 The boundary conditions at the edges of the specimens due to the effects of edge insulation, of auxiliary guard heaters and of the surrounding ambient temperature will limit the maximum thickness of specimen for any one configuration, as described in Section 6. For composite or layered specimens, the mean measurable thermal conductivity of each layer should be less than twice that of any other layer.

A1.4.4.2 This is an approximation and the results do not necessarily imply the measurement of conductivity of each layer. The accuracy will remain close to that predictable for tests on homogeneous specimens. No guidelines can be supplied to assess measurement accuracy when the requirement of A1.4.4.1 is not met.

A1.4.5 *Minimum Specimen Thickness:*

A1.4.5.1 The minimum specimen thickness is limited by contact resistances given in A1.3.1. Where thermal conductivity or thermal resistivity is required, the minimum thickness is also limited by the accuracy of the instrumentation for measuring the specimen thickness.

A1.4.5.2 The metered area, that is, the area of the specimen traversed by the heat flow-rate fed by the metered section, is related to the specimen thickness and to the gap width. As the thickness tends to zero, the metered area tends to the area of the metered section, while for thick specimens the metered area is bounded by the line defining the centre of the primary guard gap. To avoid complex corrections, this definition can be retained, provided the thickness of the specimen is at least ten times the width of the gap.

A1.4.6 *Maximum Operating Temperature:*

A1.4.6.1 The maximum operating temperature of the heating and cooling units may be limited by oxidation, thermal stress or other factors that degrade the flatness and uniformity of the surface plate and by changes of electrical resistivity of electrical insulations which may affect accuracy of all electrical measurements.

A1.4.7 *Vacuum Conditions:*

A1.4.7.1 Care must be taken if a guarded hot plate is used for measurements under vacuum conditions. If a high vacuum is desired, the materials used in the design of the apparatus must be carefully selected to avoid excessive outgassing under such conditions. Under vacuum conditions, especially at lower temperatures, serious errors can arise if care is not taken when installing heater and temperature sensor leads so as to minimize extraneous heat flow-rates and temperature measurement errors.

A1.4.8 *Apparatus Size:*

A1.4.8.1 The overall size of a guarded hot plate will be governed by the specimen dimensions that typically range from 0.2 to 1 m diameter or square. Samples smaller than 0.3 m may not be representative of the bulk material, while specimens larger than 0.5 m may create considerable problems in maintaining the flatness of the specimens and plates, temperature uniformity, equilibrium time and total cost within acceptable limits.

A1.5 *Limitations Due to Specimen:*

A1.5.1 *Thermal Resistance or Thermal Conductance:*

A1.5.1.1 *Specimen Homogeneity*—In inhomogeneous specimens, the thermal flux density both within the specimen and over the faces of the metered section area may be neither unidirectional nor uniform. Thermal field distortions will be present within the specimen and can give rise to serious errors. The region in the specimen contiguous to the metered section area and especially near the edges of this area is most critical. It is hard to give reliable guidelines on the applicability of the method in such cases. The major risk is that the imbalance errors, edge heat loss errors, etc., now unpredictable, can vary in an unpredictable way when inhomogeneities take different relative positions within the specimen.

A1.5.1.2 One way to try to estimate the error is to compare the results for two specimens from the same sample, selected so that they have as widely different a structure near the edges or the metered section area. If the two extremes cannot be identified, a number of specimens may have to be tested.

A1.5.1.3 In some samples, the variation in structure may occur over small distances. This is true for many thermal insulations. In such cases, it may be possible to use a single specimen cut larger than the apparatus. This over-size specimen is tested twice, in each case with the specimen carefully positioned so that the edges of the test area are exposed to the two extremes in structure. The two results are then compared and the difference credited to distortion. The portion of the specimen(s) protruding from the apparatus should be well insulated in the two tests to reduce the possibility of the exposed section increasing edge losses. The size and thickness of the specimen affects the size of the variations in structure that can be accommodated. The larger the test area, the smaller the effect on the results. The effect of distortion may either increase or decrease with specimen thickness.

A1.5.1.4 Direct thermal short circuits may exist between the surfaces of the specimens in contact with the plates of the heating and cooling units. The largest effect occurs when sections of material which conduct heat readily, with extended surface area on each side of the specimen, are connected by a path of low thermal resistance relative to other paths. The effect can best be identified by breaking the thermal paths, especially when the collecting surfaces can be disconnected from the rest of the path. Sheets of thermally insulating materials can be used at the critical surfaces to provide the break. Sheets made of finely ground cork, or a similar material 2 mm or more thick, work well. The surfaces must be ground to the same degree of flatness as the heating unit. The thermal resistance of these sheets can be determined in separate measurements. The net change in thermal resistance of the specimen, due to thermal shorting, can thus be determined. If greater than 1 %, another measurement should be made with thicker sheets imposed.

A1.5.2 *Temperature-Difference Correlation:*

A1.5.2.1 Thermal resistance or thermal conductance are often a function of temperature differences across the specimen. In the report, the range of temperature differences that apply to the reported values of the two properties must be defined, or it must be clearly stated that the reported value was determined at a single temperature difference.

A1.5.3 *Mean Measurable Thermal Conductivity of a Specimen:*

A1.5.3.1 In order to determine the mean measurable thermal conductivity (or thermal resistivity) of a specimen, the criteria of A1.3.1 shall be fulfilled. The specimen shall be homogeneous. Homogeneous porous specimens shall be such that any inhomogeneity has dimensions smaller than one-tenth of the specimen thickness. In addition, at any one mean temperature, the thermal resistance shall also be independent of the temperature difference established across the specimen.

A1.5.3.2 The thermal resistance of a material is known to depend on the relative magnitude of the heat transfer process involved. Heat conduction, radiation and convection are the primary mechanisms. However, the mechanisms can combine or couple to produce non-linear effects that are difficult to analyze or measure even though the basic mechanisms are well researched and understood.

A1.5.4 The magnitude of all heat transfer processes depends upon the temperature difference established across the specimen. For many materials, products and systems, a complex dependence may occur at temperature differences which are typical of use. In these cases, it is preferable to use a temperature difference typical of use and then to determine an approximate relationship for a range of temperature differences. The dependence can be linear for a wide range of temperature differences.

A1.5.5 Some specimens, while being homogeneous, are anisotropic in that the thermal conductivity measured in a direction parallel to the surfaces is different to that measured in a direction normal to the surfaces. For such specimens, this can result in larger imbalance and edge loss errors. If the ratio between these two measurable values is lower than two, reporting according to this method is still possible if imbalance and edge heat loss errors are determined separately with anisotropic specimens mounted in the apparatus.

A1.5.6 *Thermal Conductivity or Thermal Resistivity of a Material:*

A1.5.6.1 In order to determine the thermal conductivity or thermal resistivity of a material, the criteria of A1.3.2 shall be fulfilled. In addition, adequate sampling must be performed to ensure that the material is homogeneous or homogeneous porous, and that the measurements are representative of the whole material product or system. The thickness of the specimens must be greater than that for which the thermal conductivity of the material product or system does not change by more than 2 % with further increase in thickness.

NOTE A1.1—Results obtained on specimens where thermal conductivity is still changing with specimen thickness are only applicable at that specific test thickness.

A1.5.7 *Dependence on Specimen Thickness:*

A1.5.7.1 Of the processes involved, only conduction produces a heat flow-rate that is directly proportional to the thickness of a specimen. The others result in a more complex relationship. The thinner and less dense the material, the more likely that the resistance depends on processes other than conduction. The result is a condition that does not satisfy the requirements of the definitions for thermal conductivity and thermal resistivity, both of which are intrinsic properties, since the transfer factor shows a dependence on the specimen thickness. For such materials, it may be desirable to determine the thermal resistance at conditions applicable to their use. There is believed to be a lower limiting thickness for all materials below which such a dependence occurs. Below this thickness, the specimen may have unique thermal transmission properties, but do not relate to the material. It remains, therefore, to establish this minimum thickness by measurements.

A1.5.7.2 Determination of minimum thickness above which thermal properties of the material may be defined.

A1.5.7.3 If the minimum thickness for which the thermal conductivity and resistivity can be defined is not known, it is necessary to estimate this thickness.

A1.5.7.4 In the absence of an established method, the procedure outlined below may be used to approximate the thickness and whether it occurs in the range of thickness in which a material is likely to be used.

A1.5.7.5 It is important to differentiate between added thermal resistance in measurements caused by the placement of the temperature sensors below the surfaces of the plates, added resistance caused by poor specimen surfaces, and added resistance caused by the coupling of the conduction and radiation modes of heat transfer in the specimens. All three can affect the measurements in the same way, and often the three may be additive.

A1.5.7.6 Select a sample uniform in density distribution, with the thickness L_5, equal to the greatest thickness of the material to be characterized or equal to the maximum allowable thickness for the test apparatus.

A1.5.7.7 Cut five sets of specimens in approximately equal increments from the sample ranging in thickness from the smallest likely to be used in practice. The set of specimens shall be designated s_1 to s_5 according to their respective thickness L_1 to L_5.

A1.5.7.8 For low density materials where heat is transferred by radiation and conduction mechanisms and where the absence of convection has been verified, the slope of a plot of thermal resistance versus thickness will very frequently diminish up to 1 to 2 cm and then will remain constant as the thickness increases. The reciprocal of this constant slope is the thermal conductivity to be assigned to high thickness specimens.

A1.5.7.9 Measure the thickness and thermal resistance of s_1, s_3, and s_5 at the same mean temperature and with the same temperature difference across the specimen. Plot the thermal resistance versus thickness. If these three values differ from a straight line relationship by less than ±1 %, the slope of the straight line shall be computed. If the three values differ by more than 1 %, then similar measurements shall be made on s_2 and s_4 to check if there is a thickness above which the thermal resistance does not differ from a straight line by more than 1 %.

A1.5.7.10 If this thickness exists, the slope of the straight line shall be determined to compute a thermal conductivity $\lambda_m = \Delta L/\Delta R$ defined as the ratio between the increments of thickness, ΔL, and increments of the thermal resistance, ΔR.

A1.5.7.11 The thickness at which this occurs will vary according to the densities, types and forms of different materials, products and systems for different mean temperatures.

A1.5.7.12 Thermal conductivity and thermal resistivity then characterizes the material, product or system for thicknesses above which the transfer factor differs by less than 2 % from λ_m.

A1.5.7.13 Allowance for experimental errors must be made in the interpretation of results. Least-square curve fitting of R versus L may also help. A larger number of specimens may be used where greater definition is required.

A1.5.7.14 Thickness dependence may be a function of temperature difference across the specimens. For the purposes of this test method, the above checks, if performed at typical operating temperature differences, shall be adequate to indicate the degree of thickness dependence.

A1.5.8 *Method of Determining Dependence on Temperature Difference*—If the temperature-difference dependence of the thermal properties is not known for a material, a minimum of three measurements shall be made. These are made with widely differing temperature differences. A second-order dependence can be revealed by these measurements. When a simple linear relationship is known to occur, only two measurements, that is, one extra, need be made. This establishes the linear dependence for that particular sample.

A1.5.9 *Warping*—Special care should be exercised with specimens with large coefficients of thermal expansion that warp excessively when subjected to a temperature gradient. The warping may damage the apparatus or may cause additional contact resistance that may lead to serious errors in the measurement. Specially designed apparatus may be necessary to measure such materials.

A1.6 *Measurement Uncertainty*—The uncertainty of the apparatus is based upon consideration of the random and systematic components of the following measurement uncertainties (**32**): uncertainty in heat flow, Q; uncertainty in temperature difference, $T = (T_H - T_C)$; uncertainty in metered area, A; and, uncertainty in specimen thickness, L.

A1.6.1 Other specimen characterization and test condition data may need to be reported. The precision and bias of these data are to be reported to the extent they have a direct bearing on the accuracy of the results. Prescribed precision and bias of the primary data are not mandated by this test method. However, it is required that the user assess and report the precision and bias of the data. The discussion below provides guidelines to assist the user in performing this uncertainty assessment. A variety of helpful performance checks are included in this discussion. In the following discussion both random and systematic errors are considered. The subscript $_s$ is used to denote systematic, and the subscript $_r$ is used for the random components.

A1.6.1.1 *Systematic Error,* $_s$—Systematic error, s, is any component of error that remains fixed during the runs that constitute a successful test. To simplify the discussion, this does not include any components of error that are known both in magnitude and sign. Under such circumstances, the user should make appropriate corrections to the conductivity measurements and supply the justification for them. The user may check for the presence of unexpected errors by using a reference specimen or transfer standard available from appropriate sources. If errors are discovered, their source should be identified and removed. A guarded hot plate cannot be calibrated. The task of estimating the remaining systematic errors is based on judgment and experience, including an awareness of the results of interlaboratory comparisons. The implications of such estimates is often that they are the maximum possible systematic errors. In this event the total maximum systematic error is the sum of the errors. It is, however, more likely that these estimates are probabilistic in nature and do not, in fact, represent the worst possible case. The total probable systematic errors are summed in the same manner as random errors, that is, the square root of the sum of squares. In the following discussion the latter approach is taken. However, the user must decide if the bias estimates are worst cases or probabilistic in nature, and sum them accordingly.

A1.6.1.2 *Random Error, $_r$*—Random error, *r*, is that component of error that may vary both in sign or magnitude during the runs that constitute a successful test. For simplicity, it is assumed that the variations are normally distributed and conventional statistical techniques are applicable. An estimate of random error components can be obtained by repeat measurements of each variable.

A1.6.1.3 It is important to distinguish between random and systematic errors for the following reason. The results reported in the test method are mean values derived from more than a single run. The uncertainties reported generally apply to these mean values. The uncertainty of a mean value due to the random error component decreases approximately as $1/n$ where n is the number of repeat runs. In contrast to this, the uncertainty of the mean value due to the systematic error component does not decrease with repeat runs. Thus, it is recommended that the error components be treated separately. The total uncertainty is expressed by reporting both components separately.

A1.7 *Error Components*—In the following sections, the error components of each reported variable are discussed. The total random or systematic uncertainty for each variable is taken to be the square root of the sum of squares.

A1.7.1 *Heat Flow, Q*—The objective of the test method is to establish and measure uniaxial heat flow through the metered area of the specimen. Any deviation from this objective represents error in the reported heat flow. The following sources of error should be considered:

A1.7.2 *Edge Heat Loss, $_sQ_{se}$*—Edge heat loss, $_sQ_{se}$ is a systematic error as the conditions surrounding the plate-specimen stack remain constant throughout the test procedure. Although tests have been reported that shed some light on the magnitude of this error, the results generally are not proven to the point where corrections based on these results are universally accepted **(1,4,6,7,18-22)**. However, the results are considered sufficiently valid for the basis of defining the maximum specimen thickness. The optimum environmental temperature to minimize this error is a small fraction of T above the mean test temperature. To determine the sensitivity of this error to test conditions, the user should determine the heat flux as a function of secondary guard temperature. This dependence may change appreciably with specimen and apparatus characteristics and, therefore, should be done under typical test conditions.

A1.7.3 *Gap Heat Loss*—Gap heat loss is considered to be composed of both systematic, $_sQ_{gp}$, and random, $_rQ_{gp}$, components. The systematic component can be, in part, due to the fact that there may be a finite number of locations along the gap at which the imbalance is measured; reducing the temperature difference between a finite number of points on opposite sides of the gap to zero may not necessarily ensure that there is zero net flow of heat across the gap. Improper position of the sensors will lead to systematic error. Spurious emfs within the circuitry will result in a systematic imbalance. The random component is due to short-term control fluctuations. After estimating the probable imbalance across the gap in terms of temperature (or sensor voltage) one needs to determine the effect of this imbalance on the measured heat flow through the metered area. This can be done by measuring the dependence of metered area power on intentionally introduced gap imbalance. A typical way of addressing this is to run three tests, one with the guard balanced and one each biased positive and negative. The results are plotted, lambda versus gap balance, and the zero intercept is determined. The imbalance introduced should be large enough to yield an easily measured change in Q, but small enough to remain in the region where the dependence of Q upon imbalance is approximately linear.

A1.7.3.1 It has been found that **(3,15,16)** the gap heat loss, Q_{gp} is linearly dependent on temperature unbalance across the gap, T_g, that is, $Q_{gp} = BT_g$. The proportionality constant, B, is dependent on the wires crossing the gap (number, size, and type), gap geometry (width and cross-sectional shape), the gap fill material (gas, insulation), the emittance of the gap surfaces and the material in the vicinity of the gap between the hot and cold plates. A reasonable approximation of this heat flow can be calculated from this information. It is recommended that this be done to confirm the value measured by the procedure described in the previous paragraph.

A1.7.4 *Effect of Drift of the Metered Area Heater*—A quasi-heat loss exists due to the changing heat content of the metered area heater as its temperature changes. Typical plates have a relatively high heat capacity and even for small drift rates can produce significant errors in measured heat flow. If the drift is monotonic, the error is systematic, $_sQ_d$; if not, the error is exhibited as random error, $_rQ_d$. Normally, the experiment is conducted so that there is no observable drift. Under this circumstance, the possible drift is determined by the detectability or control limit, dT/dt, of the system. One can compute the magnitude of this error, Q_d in watts, from a knowledge of the maximum possible dT/dt and the specific heats and masses of the various components of the metered section of the plate as follows:

$$Q_d = dT/dt\ C_iM_i \qquad \text{(A1.1)}$$

The specimen heat capacity also contributes to the drift error, but for low-density insulations the heat capacity of the specimen is small compared to the plate. This error also can be determined by measuring the dependence of drift rate on measured heater power. Comparison of the calculated and measured results is advised to increase confidence in the reported result.

A1.7.5 Power determination error, composed of both systematic, $_sQ_p$ and random, $_rQ_p$, components. With high quality instrumentation these errors can be reduced to an insignificant level. The manufacturers' specifications on bias and precision will normally suffice to define these errors.

A1.7.6 *Temperature and Temperature Difference*—Temperature error is composed of systematic, $_sT$, and random, $_rT$, components. In addition, these errors are further subdivided according to the source of the error:

A1.7.6.1 Calibration error, $_sT_c$, is entirely systematic as long as the same calibration is used. It is, however, not necessarily the same for each temperature sensor. In the case of thermocouples, calibration is frequently performed for each spool of wire, not for each piece of wire from that spool. Therefore, systematic differences can occur as one progresses through the spool. The calibration is frequently represented by an equation which approximates the experimental calibration data taken at selected temperatures. If a digital read-out device is used that yields temperature directly, the calibration formulation is built into the device and the same basis for error exists.

A1.7.6.2 Instrumentation measurement error, T_m, occurs when the sensor output is measured. This error contains both systematic and random components. Each component should be estimated from equipment manufacturer's specifications and from estimated spurious circuit effects. In addition, temperature errors are introduced by long and short-term control fluctuations. A helpful procedure to assess the magnitude of these errors is as follows. Place the guarded metered area and primary guard(s) in thermal contact with the adjacent cold plates (insert high conductance plates in place of the specimens if the plates cannot be placed physically together). Adjust the cold plates to the desired temperature; control this temperature until steady-state is reached. The metered area heater should be off. Periodically read the isothermal surface temperatures to detect systematic differences and random variations over an extended time.

A1.7.6.3 Sensor positioning, a potentially significant source of error in temperature measurement can be caused by improper positioning of the sensor or the disturbance caused by the presence of or finite size of the sensor itself. It is intended that the average temperature of each specimen surface be measured. If the sensor is mounted in the plate surface, thermal contact resistance between the plate and specimen is a source of error. If the sensor is mounted in the specimen surface, sensor separation (specimen thickness) is a source of error. If the specimen is inhomogeneous across the metered area, surface temperature variations exist and the indicated temperature will depend on its location on the surface. If heat flows along the sensor leads from the external environment, the measured temperature will be in error because of the presence of the sensor. For a single test on a given specimen, this source of error, $_sT_p$, is systematic. A performance check that is helpful to determine the potential temperature error due to temperature nonuniformity is as follows: Assemble a multijunction thermocouple and place it between the specimen and plate in question. Establish steady-state at the desired test condition. Determine the variation in temperature across the plate from the multijunction thermocouple outputs.

A1.7.6.4 A helpful technique to estimate interface temperature errors is to mount sensors both within the plate and within the specimen surface. Then perform a test and calculate the difference between the two sets of data.

A1.7.6.5 Temperature difference error is also composed of systematic, $_sT$, and random components, $_tT$. Care must be exercised in estimating these components compared to the error components for temperature itself. The results can depend strongly on whether a differential measurement or two absolute measurements are performed. Because T is frequently small, large percentage errors can occur if care is not observed. For example, at a mean specimen temperature of 300 K, an error of 1 K in the mean temperature, that corresponds to an error of about 0.2 % in thermal resistance for typical insulations. However, this same error of 1 K in measurement of a specimen temperature difference of 25 K corresponds to a 4 % error in both T and in the value of the thermal resistance, independent of the mean temperature. The ad hoc experiment described in 9.5.1.3 is recommended to provide estimates of these error components.

A1.7.7 Specimen thickness error, $_sL$, and meter area error, $_sA$, are both systematic errors. The specimen thickness error is determined by the ability to measure the plate spacing (including variations of this thickness over the metered area) or, in the case of rigid specimens, the specimen thickness and the changes due to thermal expansion. The effect of bowing or warping at operating temperatures should be given attention. At relatively large thicknesses (above 5 cm) this error can be maintained below 0.5 %. At small thicknesses (below 0.5 cm) this error may become a dominating factor in the overall accuracy. The meter area error is usually small except for the assumption about what proportion of the gap area to include. This error is difficult to estimate for very thin specimens or when a discontinuity in the specimen occurs at the gap. The specimen thickness error will contain a random component, $_rL$, due to assembly and disassembly.

A1.8 *Thermal Conductance or Thermal Resistance*—The relative uncertainty in thermal conductance, C, caused by either random errors or systematic errors of indeterminate sign, may be calculated from the following error propagation formula:

$$(\Delta C/C)^2 = (\Delta Q/Q)^2 + (\Delta T/T)^2 \qquad \text{(A1.2)}$$

where Q/Q and T/T are the total relative uncertainties of heat flux and temperature difference, respectively. The same equation applies to thermal resistance. Included in the total relative uncertainties are those due to the measurement as well as those discussed in Practice C 1045. For example for fibrous glass insulation at 24°C mean temperature and a 40°F temperature difference across the specimen the following errors can be realized:

$$(\Delta C/C)^2 = (0.5)^2 + (0.25)^2 = 0.31 \qquad \text{(A1.3)}$$

Therefore, the uncertainty in thermal conductance would be $\sqrt{0.31} = 0.56$ %.

A1.9 *Thermal Conductivity or Thermal Resistivity*—The relative uncertainty in thermal conductivity caused by either random or systematic errors may be calculated from the following error propagation formula:

$$\left(\frac{\Delta\lambda}{\lambda}\right)^2 = (\Delta Q/Q)^2 + (\Delta T/T)^2 + (\Delta A/A)^2 + (\Delta L/L)^2 \qquad \text{(A1.4)}$$

where A/A and L/L are the total relative uncertainties of area and thickness, respectively. Again, the above total relative uncertainties include not only the measurement uncertainty, but also the effect of material variability and deviations from the definitions as discussed in Practice C 1045. In addition, it should be noted that the temperature to which each measured property is assigned also contains a measurement error that affects the uncertainty of the final result. The effect of this error increases as the temperature dependence of the measured property increases.

A1.9.1 For example for fibrous glass insulation at 24°C mean temperature and a 22°C temperature difference across the specimen the following errors can be realized.

$$\left(\frac{\Delta\lambda}{\lambda}\right)^2 = (0.5)^2 + (0.25)^2 + (0.01)^2 + (0.1)^2 = 0.32 \quad \text{(A1.5)}$$

Therefore, the uncertainty in thermal conductivity would be $\sqrt{0.32} = 0.57\,\%$.

A1.10 It is recommended that the user periodically confirm these calculated uncertainties by measuring specimens of established standard reference materials or calibrated transfer specimens. Comparison of the measurement results with the accepted values will reveal whether the performance of the guarded hot plate is of acceptable quality. The results of such comparative measurements are not to be used to obtain an apparatus "calibration" or "correction" factor. For further information on this see Refs **(23–29)**.

REFERENCES

(1) Bankvall, C. G., "Mechanisms of Heat Transfer in Permeable Insulation and Their Investigation in a Special Guarded Hot Plate," *Heat Transmission Measurements in Thermal Insulations, ASTM STP 544*, R. P. Tye, Ed., 1974, pp. 34–48.

(2) Brendeng, E., and Frivik, P. E., "New Development in Design of Equipment for Measuring Thermal Conductivity and Heat Flow," *ASTM STP 544*, R. P. Tye, Ed., 1974, pp. 147–166.

(3) Smith, D. R., Hust, J. G., and Van Poolen, L. J., "A Guarded-Hot-Plate Apparatus for Measuring Effective Thermal Conductivity of Insulations Between 80 K and 360 K," *NBSIR 81-1657*, 1982, 49 pp.

(4) Pratt, A. W., Chapter 6: "Heat Transmission in Low Conductivity Materials," in *Thermal Conductivity*, Vol 1, R. P. Tye, Ed., Academic Press, 1969, pp. 301–405.

(5) De Ponte, F., and Di Filippo, P., "Design Criteria for Guarded Hot Plate Apparatus," *Heat Transmission Measurements in Thermal Insulations, ASTM STP 544*, R. P. Tye, Ed., 1974, pp. 97–117.

(6) Woodside, W., and Wilson, A. G., "Unbalance Errors in Guarded Hot Plate Measurements," *Symposium on Thermal Conductivity Measurements and Applications of Thermal Insulations, ASTM STP 217*, 1956, pp. 32–48.

(7) Woodside, W., "Analysis of Errors Due to Edge Heat Loss in Guarded Hot Plates," *Symposium on Thermal Conductivity Measurements and Applications of Thermal Insulations, ASTM STP 217*, 1957, pp. 49–64.

(8) Woodside, W., "Deviations from One-Dimensional Heat Flow in Guarded Hot Plate Measurements," *Review of Scientific Instruments*, Vol 28, 1957, pp. 1033–1037.

(9) Hust, J. G., "Thermal Anchoring of Wires in Cryogenic Apparatus," *Review of Scientific Instruments*, Vol 41, No. 5, May 1970, pp. 622–624.

(10) ASTM Subcommittee E20.04, *Manual on the Use of Thermocouples in Temperature Measurement, ASTM STP 470B*, 1981.

(11) Sparks, L. L., Powell, R. L., and Hall, W. J., *Cryogenic Thermocouple Tables, NBS Report 9712*, National Bureau of Standards, Boulder, Colorado, 1968.

(12) Bertasi, M., Bigolaro, G., and De Ponte, F., "Fibrous Insulating Materials as Standard Reference Materials at Low Temperatures," *Thermal Transmission Measurements of Insulation, ASTM STP 660*, R. P. Tye, Ed., 1978, pp. 30–49.

(13) Shirtliffe, C. J., "Establishing Steady-State Thermal Conditions in Flat Slab Specimens," *Heat Transmission Measurements in Thermal Insulations, ASTM STP 544*, R. P. Tye, Ed., 1974, pp. 13–33.

(14) Bomberg, M. and Solvason, K. R., "Precision and Accuracy of Guarded Hot Plate Method," *Proceedings of the Seventeenth International Thermal Conductivity Conference*, J. G. Hust, Ed., Plenum Press, 1983, pp. 393–410.

(15) Siu, M. C. I., "Comparison of Results of Measurements Made on a Line-Heat-Source and a Distributed Heat Source Guarded-Hot-Plate Apparatus," *Proceedings of the Seventeenth International Thermal Conductivity Conference*, J. G. Hust, Ed., Plenum Press, 1983, pp. 413–418.

(16) Siu, M. C. I., and Bulik, C., "National Bureau of Standards Line-Heat-Source Guarded-Hot-Plate Apparatus," *Review of Scientific Instruments*, Vol 52(11), 1981, pp. 1709–1716.

(17) Horlick, J., "National Voluntary Laboratory Accreditation Program, Proficiency Testing for Thermal Insulation Materials Laboratory Accreditation Program," *NBSIR 84-2890*, 1984.

(18) Donaldson, I. G., "A Theory for the Square Guarded Hot Plate-A Solution of the Heat Conduction Equation for a Two Layer System," *Quarterly of Applied Mathematics*, Vol XIX, 1961, pp. 205–219.

(19) Donaldson, I. G., "Computer Errors for a Square Guarded Hot Plate for the Measurement of Thermal Conductivities of Insulating Materials," *British Journal of Applied Physics*, Vol 13, 1962, pp. 598–602.

(20) Pratt, A. W., "Analysis of Error Due to Edge Heat Loss in Measuring Thermal Conductivity by the Hot Plate Method," *Journal of Scientific Instruments*, Vol 39, 1962, pp. 63–68.

(21) Somers, E. V., and Cybers, J. A., "Analysis of Errors in Measuring Thermal Conductivity of Insulating Materials," *Review of Scientific Instruments*, Vol 22, 1951, pp. 583–586.

(22) Troussart, L. R., "Three-Dimensional Finite Element Analysis of the Guarded Hot Plate Apparatus and its Computer Implementation," *Journal of Thermal Insulation*, Vol 4, 1981, pp. 225–254.

(23) Pelanne, C. M., "The Development of Low Density Glass-Fiber Insulation as Thermal Transmission Reference Standards," *Seventeenth International Thermal Conductivity Conference*, J. G. Hust, Ed., Plenum Press, 1983, pp. 763–776.

(24) Rennex, B. G., Jones, R. R., and Ober, D. G., "Development of Calibrated Transfer Specimens of Thick, Low-Density Insulations," *Proceedings of the Seventeenth International Thermal Conductivity Conference*, J. G. Hust, Ed., Plenum Press, 1983, pp. 419–426.

(25) Shirtliffe, C. J., "Effect of Thickness on the Thermal Properties of Thick Specimens of Low-Density Thermal Insulation," *Thermal Insulation Performance, ASTM STP 718*, D. L. McElroy and R. P. Tye, Eds., 1980, pp. 36–50.

(26) Siu, M. C. I., "Fibrous Glass Board as Standard Reference Material for Thermal Resistance Measurement Systems," *Thermal Insulation Performance, ASTM STP 718*, D. L. McElroy and R. P. Tye, Eds., 1980, pp. 343–360.

(27) Smith, D. R., and Hust, J. G., "Effective Thermal Conductivity of Glass-Fiber Board and Blanket Standard Reference Materials," *Proceedings of the Seventeenth International Thermal Conductivity Conference*, J. G. Hust, Ed., Plenum Press, 1983, pp. 483–496.

(28) Smith, D. R., and Hust, J. G., "Measurement of Effective Thermal Conductivity of a Glass Fiberboard Standard Reference Material," *Cryogenics*, Vol 21, No. 7, July 1981, pp. 408–410.

(29) Smith, D. R., Hust, J. G., and Van Poolen, L. J., "Measurement of Effective Thermal Conductivity of Glass Fibreblanket Standard

Reference Material," *Cryogenics*, Vol 21, No. 8, August 1981, pp. 460–462.

(30) Hust, J. G., and Smith, D. R., "Round-Robin Measurements of the Apparent Thermal Conductivity of Two Refractory Insulation Materials, Using High Temperature Guarded-Hot-Plate Apparatus," *NBSIR 88-3087*, 28 pp., U.S. National Institute of Standards and Technology, April 1988.

(31) Peavy, B. A., and Rennex, B. G., "Circular and Square Edge Effect Study for Guarded-Hot-Plate and Heat-Flow-Meter Apparatuses," *Journal of Thermal Insulation, 9*, 1986, pp. 254–300.

(32) International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty In Measurement*, ISBN 92-67-10188-9, 1993, First Edition.

(33) Bode, K.-H., "Thermal Conductivity Measurements with the Plate Apparatus: Influence of the Guard Ring Width on the Accuracy of Measurements," *Guarded Hot Plate and Heat Flow Meter Methodology, ASTM STP 879*, C. J. Shirtliffe, and R. P. Tye, Eds., 1985, pp. 29–48.

(34) ASTM, *Manual on Presentation of Data and Control Chart Analysis*, Manual 7, 6th Edition, 1991, Available from ASTM Headquarters, Request PCN:28-0070899-34.

(35) Eisenhart, C., "Realistic Evaluation of the Precision and Accuracy of Instrument Calibration Systems," *Journal of Research of the National Bureau of Standards*, Vol 67C, No. 2, April–June 1963, pp. 161–187.

(36) Pham, Q. T., and Smith, C. G., "Thermal Imbalance Errors and Effective Area in Guarded Hot Plates," *Review of Scientific Instruments*, Vol 57(1), January 1986, pp. 99–105.

(37) Orr, H. W., "A Study of the Effects of Edge Insulation and Ambient Temperature on Errors in Guarded-Hot-Plate Measurements," *Proceedings of the 7th Thermal Conductivity Conference*, D. R. Flynn, and B. A. Peavy, Eds., 1968, pp. 521–526.

(38) Robinson, H. E., and Watson, T. W., "Interlaboratory Comparison of Thermal Conductivity Determinations with Guarded-Hot-Plates," *Symposium on Thermal Insulating Materials, ASTM STP 119*, Philadelphia, 1951, pp. 36–44.

(39) Horlick, J., and Berger, H. W., "NVLAP and the Thermal Insulation Proficiency Testing Program," *Journal of Thermal Insulation*, Vol 8, April 1985, pp. 278–297.

(40) Ku, H. H., "Notes on the Use of Propagation Error Formulas," *Journal of Research of the National Bureau of Standards*, Vol 70C, No. 4, October–December 1966, pp. 263–273.

(41) Hust, J. G., and Pelanne, C. M., "Round Robins on the Apparent Thermal Conductivity of Low Density Glass Fiber Insulations Using Guarded Hot Plate and Heat Flow Meter Apparatus," *NBSIR 85-3026*, National Bureau of Standards, Boulder, Colorado, 1985.

(42) Pelanne, C. M., "Experiments on the Separation of Heat Transfer Mechanisms in Low-Density Fibrous Insulation," *8th Conference on Thermal Conductivity*, Plenum Press, 1969, pp. 897–911.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name:	ASTM C236: Standard Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box
CFR Section(s):	10 CFR 434.402.1.2.1(a)
Standards Body:	American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: C 236 – 89 (Reapproved 1993)$^{\epsilon 1}$

Standard Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box[1]

This standard is issued under the fixed designation C 236; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

$^{\epsilon 1}$ NOTE—Section 12 was added editorially in September 1993.

1. Scope

1.1 This test method, known as the guarded hot box method, covers the measurement of the steady-state thermal transfer properties of panels. In distinction to Test Method C 177, which is primarily applicable to homogeneous samples, the guarded hot box method provides for the evaluation of thermal performance of building assemblies. This test method is suitable for building construction assemblies, building panels, and other applications of nonohomogeneous specimens at similar temperature ranges. It may also be used for homogeneous specimens.

1.2 This test method may be applied to any building construction for which it is possible to build a reasonably representative specimen of size appropriate for the apparatus.

NOTE 1—A calibrated hot box, Test Method C 976, may also be used for the described measurements and may prove more satisfactory for testing assemblies under dynamic conditions (nonsteady-state) and to evaluate the effects of water migration and air infiltration. The choice between the calibrated or the guarded hot box should be made only after careful consideration of the contemplated use.

1.3 In applying this test method, the general principles outlined must be followed; however, the details of the apparatus and procedures may be varied as needed.

1.3.1 The intent of this test method is to give the essential principles and the general arrangement of the apparatus. Any test using this apparatus must follow those principles. The details of the apparatus and the suggested procedures that follow are given not as mandatory requirements but as examples of this test method and precautions that have been found useful to satisfy the essential principles.

1.3.2 Persons applying this test method shall be trained in the methods of temperature measurement, shall possess a knowledge of the theory of heat flow, and shall understand the general requirements of testing practice.

1.3.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 2—While various units may be found for thermal properties, the International System of units is used exclusively in this test method. For conversion factors to inch-pound and kilogram-calorie systems, see Table 1.

2. Referenced Documents

2.1 *ASTM Standards:*

C 168 Terminology Relating to Thermal Insulating Materials[2]
C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus[2]
C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[2]
C 578 Specification for Preformed Cellular Polystyrene Thermal Insulation[2]
C 976 Test Method for Thermal Performance of Building Assemblies by Means of a Calibrated Hot Box[2]
C 1045 Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements[2]
E 178 Practice for Dealing With Outlying Observations[3]
E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[4]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Terminology

3.1 *Definitions*— For definitions of terms used in this test method, refer to Terminology C 168.

3.2 *Symbols:*

3.2.1 The symbols used in this test method have the following significance:

λ = thermal conductivity, W/(m·K),
C = thermal conductance, W/(m^2·K),
h = surface conductance, W/(m^2·K),
U = thermal transmittance, W/(m^2·K),
q = heat flux (time rate of heat flow through Area A), W/m^2,

---

[1] This test method is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurement.

Current edition approved Sept. 29, 1989. Published June 1990. Originally published as C 236 – 60. Last previous edition C 236 – 87.

[2] *Annual Book of ASTM Standards*, Vol 04.06.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

TABLE 1 Conversion Factors for Thermal Conductivity[A]

	W/(m·K)[B]	W/(cm·K)	cal/(s·cm·K)	kg-cal/(h-m·K)	Btu/(h·ft·°F)	Btu·in./(h·ft²°F)
1 $W \cdot m^{-1} \cdot K^{-1}$ =	1.000	1.000×10^{-2}	2.388×10^{-3}	0.8598	0.5778	6.933
1 $W \cdot cm^{-1} \cdot K^{-1}$ =	100.0	1.000	0.2388	85.98	57.78	693.3
1 $cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}$ =	418.7	4.187	1.000	360.0	241.9	2903.00
1 $kg\text{-}cal \cdot h^{-1} \cdot h^{-1} \cdot K^{-1}$ =	1.163	1.163×10^{-2}	2.778×10^{-3}	1.000	0.6720	8.064
1 $Btu \cdot h^{-1} \cdot ft^{-1} \cdot °F^{-1}$ =	1.731	1.731×10^{-2}	4.134×10^{-3}	1.488	1.000	12.00
1 $Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$ =	0.1442	1.442×10^{-3}	3.445×10^{-4}	0.1240	8.333×10^{-2}	1.000

Thermal Conductance[A]

	W/(m²·K)[B]	W/(cm²·K)	cal/(s·cm²·K)	kg-cal/(h·m²·K)	Btu/h·ft²·°F)
1 $W \cdot m^{-2} \cdot K^{-1}$ =	1.000	1.000×10^{-4}	2.388×10^{-5}	0.8598	0.1761
1 $W \cdot cm^{-2} \cdot K^{-1}$ =	1.000 × 104	1.000	0.2388	8598	1761
1 $cal \cdot s^{-1} \cdot cm^{-2} \cdot K^{-1}$ =	4.187×10^{4}	4.187	1.000	3.600×10^{4}	7373
1 $kg\text{-}cal \cdot h^{-1} \cdot m^{-2} \cdot K^{-1}$ =	1.163	1.163×10^{-4}	2.778×10^{-3}	1.000	0.2048
1 $Btu \cdot h^{-1} \cdot ft^{-2} \cdot °F^{-1}$ =	5.678	5.678×10^{-4}	1.356×10^{-4}	4.882	1.000

[A] Units are given in terms of (*1*) the absolute joule per second or watt, (*2*) the calorie (International Table) = 4.1868 J, or the British thermal unit (International Table) = 1055.06 J.

[B] This is the SI unit.

Q = time rate of heat flow, total input to the metering box, W,
A = metering area normal to heat flow, m^2,
L = length of path of heat flow (thickness of specimen), m,
N = minimum number of thermocouples (see Eq 1, 6.5.1.1),
r = surface resistance, $K \cdot m^2/W$,
R = thermal resistance, $K \cdot m^2/W$,
R_u = overall thermal resistance, $K \cdot m^2/W$,
t_h = average temperature of air 75 mm or more from the hot surface, K,
t_1 = area weighted average temperature of hot surface, K,
t_2 = area weighted average temperature of cold surface, K, and
t_c = average temperature of air 75 mm or more from cold surface, K.

4. Summary of Test Method

4.1 To determine the conductance, C, the thermal transmittance, U, and the thermal resistance, R, of any specimen, it is necessary to know the area, A, the heat flux, q, and the temperature differences, all of which must be determined under such conditions that the flow of heat is steady. The hot box is an apparatus designed to determine thermal performance for representative test panels and is an arrangement for establishing and maintaining a desired steady temperature difference across a test panel for the period of time necessary to ensure constant heat flux and steady temperature, and for an additional period adequate to measure these quantities to the desired accuracy. The area and the temperatures can be measured directly. The heat flux, q, however, cannot be directly measured, and it is to obtain a measure of q that the hot box has been given its characteristic design. In order to determine q, a five-sided metering box is placed with its open side against the warm face of the test panel. If the average temperature across the walls of the metering box is maintained the same, then the net interchange between the metering box and the surrounding space is zero, and the heat input to the metering box is a measure of the heat flux through a known area of the panel. The portion of the panel outside the meter area, laved by the air of the surrounding guard space, constitutes a guard area to minimize lateral heat flow in the test panel near the metering area. Moisture migration, condensation, and freezing within the specimen can cause variations in heat flow; to avoid this, the dew point temperature on the warm side must be kept below the temperature of the cold side when the warm surface is susceptible to ingress of moisture vapor. It is expected that, in general, tests in the guarded hot box apparatus will be conducted on substantially dry test panels, with no effort made to impose or account for the effect of the vapor flow through or into the panel during the test.

4.2 Since the basic principle of the test method is to maintain a zero temperature difference across the metering box walls, adequate controls and temperature-monitoring capabilities are essential. It is recognized that small temperature gradients could occur due to the limitations of controllers. Since the total wall area of the metering box is often more than twice the metering area of the panel, small temperature gradients through the walls may cause heat flows totaling a significant fraction of the heat input to the metering box. For this reason, the metering box walls may also be equipped to serve as a heat flow meter so that heat flow through them can be estimated and minimized by adjusting conditions during tests, and so that a heat flow correction can be applied in calculating test results.

5. Significance and Use

5.1 When the guarded hot box is constructed to test assemblies in the vertical orientation, it is suited for evaluating walls and other vertical structures. When constructed to test assemblies in the horizontal orientation, it is suited for evaluating roof, ceiling, floor, and other horizontal structures. Other orientations are allowable. The same apparatus may be used for both vertical and horizontal testing if it can be rotated or reassembled in either orientation.

NOTE 3—Horizontal structures that incorporate attic spaces between a ceiling and a sloping roof are highly complex constructions, and testing in the guarded hot box would be extremely difficult. Proper consideration must be given to specimen size, natural air movement, ventilation effects, radiative effect, baffles at the guard-meter demarcation, etc. All of these special conditions must be included in the report (10.1.1). Consideration should be given to the use of the calibrated hot box for such large, complex constructions.

5.2 For vertical specimens with air spaces that significantly affect thermal performance, the metering box height should ideally match the construction height. If this is not possible, horizontal convection barriers must be installed to prevent air exchange between meter and guard areas, unless it can be shown that the omission of such barriers does not significantly affect results.

5.3 For all specimens it is necessary to maintain a near zero lateral heat flow between the guard area and the meter area of the specimen. This can be achieved by maintaining a near zero temperature difference on the specimen surface between the metered and guard areas. In specimens incorporating an element of high lateral conductance (such as a metal sheet), it may be necessary to separate the metered and the guard areas of the highly conductive element by a narrow gap such as a saw cut.

5.4 Since this test method determines the total flow of heat through the test area demarcated by the metering box, it is possible to determine the heat flow through a building element smaller than the test area, such as a window or representative area of a panel unit, if the parallel heat flow through the remaining surrounding area or mask is determined (see Annex A1).

6. Apparatus

6.1 *Arrangement*—Fig. 1 (*a*) shows a schematic arrangement of the test panel and of various major elements of the apparatus; Fig. 1(*b*) and (*c*) show alternative arrangements. Still other arrangements, accomplishing the same purpose, may be preferred for reasons of convenience or ease of installing panels. In general, the size of the metering box determines the minimum size of the other elements.

6.2 *Metering Box:*

6.2.1 *Size*—The size of the metering box is largely governed by the metering area required to obtain a representative test area of panel. For example, for panels incorporating air spaces or stud spaces, the metering area, preferably, should exactly span an integral number of spaces. The height of the metering box should be not less than the width and is subject to the limitations as described in 5.2. The depth of the metering box should be not greater than that required to accommodate its necessary equipment.

6.2.2 *Thermal Resistance*—The metering box walls shall have a thermal resistance of not less than 0.83 m^2 K/W. In order that the resistance of the box wall shall be uniform over the entire box area, a construction without internal ribs shall be used, for example, a glued balsa wood or a sandwich construction with aged urethane foam core. The edge in contact with the panel shall, if necessary, be narrowed on the outside only, to hold a gasket not more than 13 mm wide. If necessary, a wood nosepiece can be used to carry the gasket. The metering area of the panel shall be taken as the area included between the center lines of the gaskets. All surfaces that can exchange radiation with the specimen must have a total hemispherical emittance greater than 0.8.

6.2.3 *Heat Supply and Air Circulation*—Fig. 2 shows a possible arrangement of equipment in the metering box to assure an even, gentle movement of air over the metering

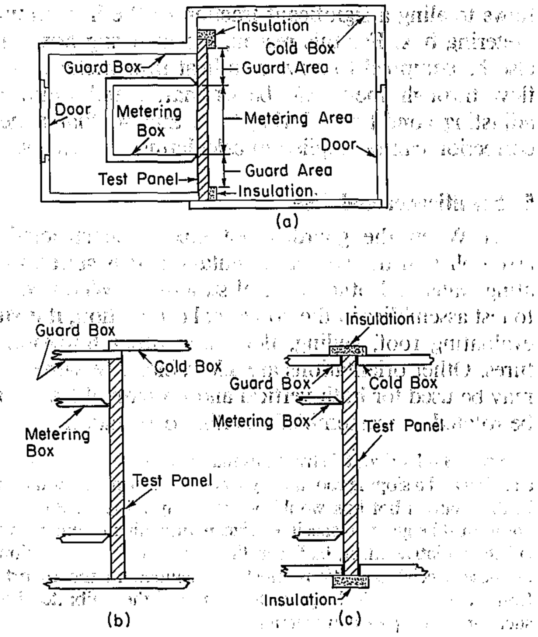


FIG. 1 General Arrangements of Test Box, Guard Box, Test Panel, and Cold Box

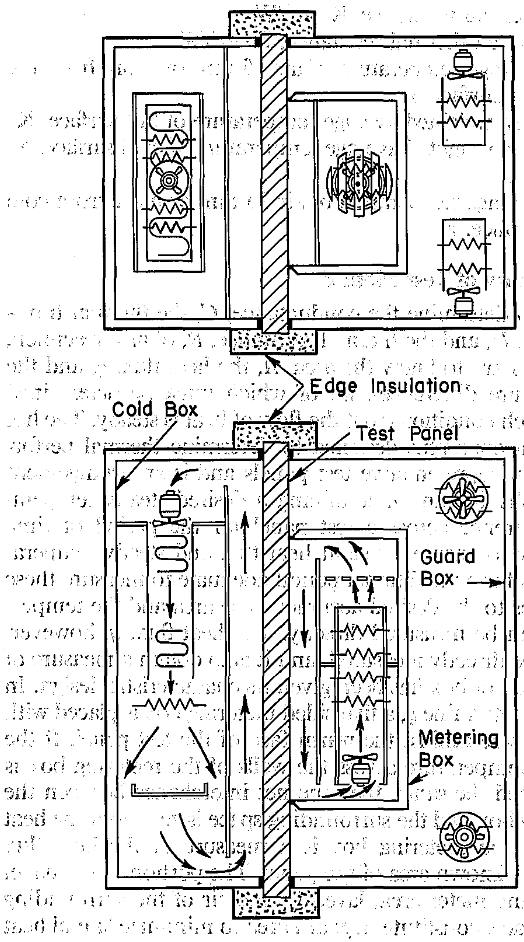


FIG. 2 Arrangement of Equipment During the Test

area of the panel. The electric heaters are mounted in a housing with walls of resistance not less than 0.83 m^2 K/W, and with a low emittance outside surfacing to minimize radiation heat transfer to the metering box walls. In this arrangement air is continuously circulated by a small fan upward through the cylindrical housing and downward between the baffle and the panel in accordance with the motion that would result from natural convection forces. A slat-type baffle is placed some distance above the outlet of the cylindrical housing to prevent impingement of a jet of heated air against the top inner surface of the metering box. For large meter boxes the cylindrical housing may cause concentrations of air flow. To direct the air properly across the specimen, other fan arrangements may be preferable. A curved vane is mounted at the top of the baffle to smooth the entrance of air into the baffle space. In a hot box apparatus used for testing panels in a vertical position only, the moderate circulation of air resulting from natural convection may be sufficient without the use of a fan. The change in temperature of the air as it moves along the surface of the panel will, in general, be greater with natural circulation than with a fan. If a fan is used, its motor should be within the metering box, its electrical input should be as small as feasible, and the input should be carefully measured. If it is necessary to locate the motor outside the metering box, the heat equivalent of the shaft power must be accurately measured, and air leakage into or out of the metering box around the shaft must be zero.

6.2.4 *Temperature Control*—To obtain reliable test results, accurate temperature control equipment must be utilized. Temperature controllers must be capable of controlling temperature within 0.25 K during the test period. The heaters should be the open-wire type of minimal heat capacity and lag.

6.2.5 *Gaskets*—The contact edges of the metering box should ensure, by a gasket or other means, a tight air seal against the surface of the test panel. For some panels special provisions may be necessary. The metering box should be pressed tightly against the panel by suitable means. Some gasket materials age with time and service. Periodic inspection of gaskets is recommended in order to confirm their ability to provide a tight seal under test conditions.

6.2.6 *Heat Flux Transducer*—To equip the metering box walls to serve as a heat flux transducer, a means of detecting the temperature difference across the metering box walls or the heat flux through the metering box walls shall be provided. One method found satisfactory for this purpose is to apply a number of differential thermocouples connected in a series to the inside and outside surfaces of the metering box walls to form a thermopile. Precautions must be taken when determining the number of differential thermocouples. Based on a survey of guarded hot box operators, the number of differential thermocouple pairs located on metering box walls shall be five pairs per square metre of specimen metered area located on the metering box sides. At no time shall there be less than 1 pair of differential thermocouples on each of the five sides of the metering box **(1)**.[5] Precautions must also be taken when determining locations of the differential thermocouples, as temperature gradients on the inside and outside of the metering box walls are likely to exist and have been found to be a function of metering and guard box air velocities and temperature. The junctions and the thermocouple wires for at least a 100-mm distance from the junctions shall be flush with, and in thermal contact with, the surface of the wall. The output of the thermocouple pairs shall be averaged.

6.2.7 *Thermopile emf and Heat Flow Relationship*—The relationship between thermopile emf and heat flow through the metering box walls shall be determined. This relationship shall be determined for each set of metering box conditions (temperature and air velocity). A suggested method of accomplishing this objective is outlined in Appendix X1.

6.3 *Guard Box:*

6.3.1 *Size*—It is recommended that the guard box be large enough so that there is a clear distance between its inner wall and the nearest surface of the metering box of not less than the thickness of the thickest panel to be tested, but in no case less than 150 mm.

6.3.2 *Thermal Conductance*—To assure that there shall be a temperature difference of no more than a few degrees between the guard box air and its inner surfaces, the walls shall have a thermal conductance not greater than 0.6 W/(m^2·K). A low conductance is also desirable for operating reasons, to assure that the heat flow into or out of the guard box from outside will be only a small fraction of the heat flow through the guard area of the test panel.

6.3.3 *Heat Supply and Air Circulation*—One or more reflective-surfaced cylindrical heater units with a fan may be used to supply heat to the guard box air and also to circulate the air to avoid stratification. The fan air intake of at least one such heater unit should be located at the lowest point in the guard box, to prevent pooling of cool air at the bottom. The air discharged from the heater cylinder shall not impinge directly against either the metering box or the test panel.

6.3.4 *Temperature Control*—The guard box air temperature and heat input can be controlled by a differential thermopile such as that used on the metering box for a heat flow meter, or by a sensitive bridge circuit with opposed temperature-sensitive arms located in the guard and metering boxes. To avoid hunting due to the small periodic temperature variations of the metering box air, as its thermostat functions, it is desirable to put the temperature-sensitive element of the differential control in the metering box in good thermal contact with the inside surfaces of the metering box. The temperature-sensitive element in the guard box should be placed to avoid being directly in the air stream of the heater units and should be of minimum thermal lag. The control equipment used to maintain guard box temperatures must be capable of controlling to within 0.25 K.

6.4 *Cold Box:*

6.4.1 *Size*—The size of the cold box is governed by the size of the test panel or by the arrangement of boxes used, as illustrated in Fig. 1.

6.4.2 *Insulation*—The cold box should be heavily insulated to reduce the required capacity of the refrigerating equipment, and the exterior of the cold box should be provided with a good vapor barrier to prevent ingress of

---

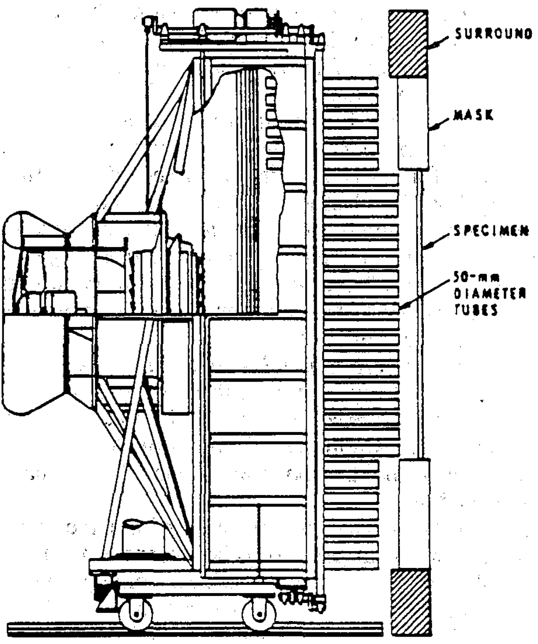
[5] The boldface numbers in parentheses refer to the list of references at the end of this test method.

vapor and heavy frost accumulations on the cooling coils.

6.4.3 *Temperature Control*—The cold box may be cooled in any manner that is capable of the close control of air temperature necessary during a test. An arrangement of equipment similar to that in the metering box may be used with a fan to force air downward through the enclosed refrigerating coils and upward through the space between a baffle and the test panel as indicated in Fig. 2. It has been found satisfactory with an arrangement of this sort to operate a unit refrigeration system continuously, with the evaporation temperature of the coil held constant by an automatic back-pressure regulating valve, and refrigerant supplied to the coil through an automatic expansion valve. An alternative method is to use an exterior located refrigeration system and insulated ducts to supply chilled air to the cold box. Liquid nitrogen in connection with a solenoid valve regulating its flow may also be used. For fine control of the cold box, installation of open wire electrical heaters in the blower duct or other fast moving part of the air circulation system and controlling these heaters by a sensor located in the discharge of the air circulation system is recommended. The use of desiccants to remove excessive moisture in the recirculating cold air may be useful. Temperature controllers for steady-state tests must be capable of controlling temperatures within ±0.25 K.

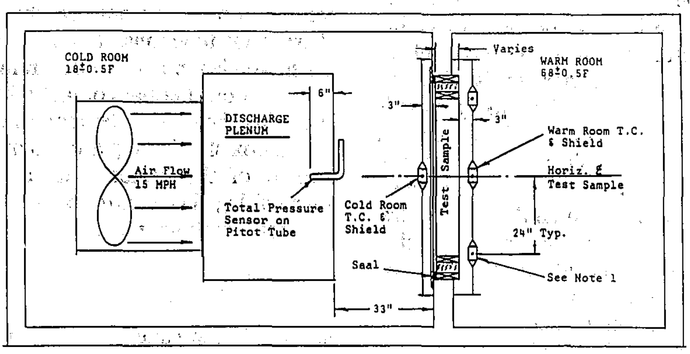
6.4.4 *Air Circulation*—High air velocities are permissible when their effect upon heat flow is to be determined. This may be accomplished by directing the airflow either parallel or perpendicular to the specimen cold surface. One method of obtaining parallel uniform velocity is to force air through a space between the specimen and a parallel baffle whose spacing may be adjustable to aid in changing the air curtain velocity. Parallel velocities, as provided in this test method, aid in obtaining uniform specimen surface temperatures and simulate the effect of cross winds. Velocities commonly used to simulate cross wind conditions are 3.35 m/s for summer conditions and 6.70 m/s for winter conditions. Perpendicular velocities, simulating direct wind impingment require moving larger amounts of air than most parallel situations, with corresponding larger power requirements. Also, the baffle should be placed further from the specimen surface and should have a porous section (a screen or honeycomb flow straightener) that directs the wind at the specimen surface (see Fig. 3 and 4). Velocities commonly used to simulate wind conditions are 3.35 m/s for summer conditions and 6.70 m/s for winter conditions. Air leakage through the specimen should be eliminated by sealing all cracks and joints with tape, caulk, or foam strips.

6.4.4.1 After construction of the air circulation system a velocity scan across the air curtain is required to verify that a uniform air curtain is formed. The apparatus should provide a means for determining air velocity past the specimen surface. One method is to locate velocity sensors directly in the air curtain.



NOTE—One inch is equal to 25.4 mm.

FIG. 3 DBR Wind Machine



NOTE 1—Thermocouples and shields on the warm side are movable to maintain 3 in. spacing to test sample.
NOTE 2—Overall chamber length may vary.
NOTE 3—One inch is equal to 25.4 mm.
NOTE 4—Thirty-two degrees farenheit is equal to 0°C.

FIG. 4 Thermal Chamber Diagram

6.5 *Temperature-Measuring Equipment:*

6.5.1 *Surface Temperatures*—Thermocouples of wire not larger in size than 0.25 mm (No. 30 AWG gage), and which meet or are calibrated to the special limits of error specified in Tables E 230, are recommended for measuring surface temperatures in the apparatus (larger thermocouples can be used if it can be shown that there is no difference in bias); for this purpose the thermocouple junction and the adjoining lead wires for a distance of at least 100 mm should be taped, or preferably cemented, tightly to the surface. The emittance of the surfacing material tape or cement should be close to the emittance of the surface.

6.5.1.1 If the specimen (and therefore its thermal resistance) is uniform, or nearly so, over the area and thus the surface temperatures vary only slightly at lower air velocities, a minimum number of thermocouples spaced uniformly and symmetrically over the surface is sufficient. This minimum number depends on the specimen size. Experience has shown that the required minimum number of thermocouples, N, can be determined from the relation that:

$$N = A/(0.07 + 0.08\sqrt{A}) \qquad (1)$$

where A is the metering area in m^2. If the number of thermocouples used is within 10 % of the number determined by this relation, then the requirements of this section are judged to be met.

6.5.1.2 If the specimen is of nonuniform construction, the number of thermocouples specified in 6.5.1.1 may still be sufficient. In this case the thermocouples shall be judiciously located to represent each of the construction elements. Such representation shall be distributed approximately uniformly and symmetrically over the specimen surface.

6.5.1.3 If the surface temperatures are expected to be greatly nonuniform, additional thermocouples must be used to sample adequately the different temperature areas so that reliable weighted mean temperatures may be obtained.

6.5.1.4 With some nonhomogeneous walls, such as concrete, it may be advisable to use copper shim stock under the thermocouples to average the temperature. Large aggregates in the concrete can create biased temperature readings.

6.5.1.5 At least two surface thermocouples shall be placed on the guard area of the specimen at suitable locations to indicate the effectiveness of the guard area.

6.5.1.6 Surface temperatures on the cold side of the test panel shall be measured by surface thermocouples placed directly opposite those on the warm side.

6.5.2 Air temperatures may be measured by thermocouples, temperature sensitive resistance wires, or other sensors. Air thermocouples shall be made of wire not larger than 0.51 mm (No. 24 AWG).

6.5.2.1 If thermocouples or other point sensors are used, they shall be located in the metering box area in the same quantity and spacing as that specified for surface thermocouples in 6.5.1.1. The thermocouple shall be located midway between the face of the panel and the baffle, if one is used, but in no case less than 75 mm from the face of the panel. The junctions of the thermocouples shall have bright metallic surfaces and shall be as small as possible to minimize radiation effects. Another method is to shield the thermocouple junction. The thermocouples may be placed directly opposite the surface thermocouples; in any case they should be located as uniformly as possible over the metering area.

6.5.2.2 Thermocouples shall also be placed in the guard space at suitable locations, to indicate the degree of uniformity of guard space air temperatures; preferably, one should be placed opposite each guard area surface thermocouple, but not less than 75 mm from the panel.

6.5.2.3 Air temperatures on the cold side of the panel shall be measured by one thermocouple placed directly opposite each of the warm side air temperature thermocouples and located in a plane parallel to the specimens surface and spaced far enough away that they are unaffected by temperature gradients in the boundary layer. The thermocouples shall be located midway between the face of the panel and the baffle, if one is used. For low velocities, a minimum spacing of 75 mm from the specimen surface is required. At higher velocities the required minimum spacing is less but in no case less than 20 mm. No thermocouples need be placed in the cold space opposite guard space thermocouples remote from the panel surface.

6.5.2.4 If air temperatures are to be measured by means of resistance wire grids, the wire shall be distributed uniformly to indicate approximately the average temperature of the air on both sides of the panel at a plane midway between the baffle and the panel but in no case less than 75 mm from the panel.

6.5.2.5 It is recommended that the surface temperature of the baffles on the hot and cold sides be measured by placing thermocouples on all surfaces the specimen can see.

NOTE 4—This is not a requirement of this test method but is highly recommended. There are several reasons for the recommendation: (*1*) this indicates any difference between the baffle surface and air temperatures; (*2*) it will allow corrections to be made to the radiation component of the surface conductances due to differences in these temperatures; and (*3*) it is necessary to do this for specimens such as glass which have a high-thermal conductance.

6.6 *Instruments:*

6.6.1 All thermocouples or other temperature sensors for observing surface and air temperatures shall have their leads brought out individually to suitable measuring instruments capable of indicating temperatures to within ±0.05 K.

6.6.2 Total average power (or integrated energy over a specified time period) for all energy to the meter box shall be accurate to within ±0.5 % of reading under conditions of use. Power measuring instruments must be compatible with the power supplied whether ac, dc, on-off proportioning, etc. Voltage stabilized power supplies are strongly recommended.

6.6.3 Velocities of air over both surfaces of the panel should be measured with suitable instruments or be calculated from a heat balance between the rate of loss or gain of heat as it moves through the baffle space, as indicated by its temperature change, and the rate of heat flow through the test panel, average values of which can be determined from the test data. It should be recognized that radiant transfer between the baffle and the specimen can affect the calculation if the radiation is significant. For this reason direct velocity measurement is desirable.

NOTE 5—It is recommended that a central control location be established, that automatic scanning and recording equipment for unattended operation be used, and that data be computer processed.

6.7 *Verification*—When a new or modified apparatus is constructed, verification tests shall be conducted on panels made from materials of known conductance that does not exceed 1.5 W/(m²·K) as determined in Test Methods C 177 or Test Method C 518[6]. Any differences in results should be carefully analyzed and corrective measures taken. Further periodic checks are recommended.

7. Sampling and Test Specimens

7.1 Specimens shall be representative of the construction to be investigated but may be modified if necessary for test purposes as mentioned in 5.2 and 5.3. It must be recognized that modifications to the construction may result in conditions that do not represent true field conditions. In many cases conduction and convection paths have considerable effect on the performance of the specimen and must be left intact. Other considerations are:

7.1.1 *Sensors*—Install temperature sensors as directed in 6.5. When desired, temperature and other sensors may be installed throughout the interior of the specimen for special investigations.

7.1.2 *Conditioning*—The usual pre-test conditioning is in ambient air long enough to come to practical equilibrium. Assemblies that may have significant moisture content, which can influence test results, must be allowed to reach steady-state moisture conditions. Since the specimen size will probably preclude oven drying, concrete wall specimens may require 6 to 8 weeks of room temperature aging.

7.1.3 *Edge Insulation*—When a test panel is installed, its edges shall, if necessary, be insulated to prevent edge effect from overtaxing the guarding effect of the guard area of the panel. For this purpose, the edges of the panel may be protected against heat loss or gain by a thickness of insulation with an *R* of 1 or 1.25 K·m²/W. It may be necessary to vapor-proof the insulation to prevent condensation of moisture in the edges of the panel, if a test arrangement similar to that shown in Fig. 1(*c*) is used. The edge of the specimen should be well sealed to prevent air infiltration between the guard and the cold box.

8. Procedures

8.1 Test conditions of temperature and orientation should be chosen to correspond as closely as possible to the circumstances of use of the construction to be tested. This test method is primarily designed for the temperatures encountered in normal building use; however, it is recognized that the method may find application in testing conditions that are outside this normal range. It is recommended that a minimum temperature differential of approximately 25 K be maintained for accurate measurement.

8.2 The required stabilization and test periods are as follows:

8.2.1 Impose steady-state conditions for at least 4 h prior to final data collection. This condition is satisfied when, over this 4-h period, the average surface temperature did not vary by more than ±0.06°C (±0.1°F) and the average power in the meter area did not vary by more than ±1 % and the data did not change unidirectionally. During this period, data shall be collected at intervals of 1 h or less.

8.2.2 After the conditions in 8.2.1 have been satisfied, continue the test period at least 8 h, but do not terminate the test until two or more successive 4-h periods produce results that do not differ by more than 1 %. During this period take data at intervals of 1 h or less. The average of the data for the two or more successive 4-h periods that agree within 1 % are used in calculating the final results. In testing panels that are heavily insulated, very massive, or both, it may be necessary to extend the duration of the test beyond the minimum period of two consecutive 4-h periods in order to be assured that conditions are steady, as it has been observed that continuing but small incremental changes can give a premature appearance of stability.

8.2.3 The calculation of a time constant, generated from apparatus measurements (Note 6) combined with an estimate of the thermal properties of the specimen, will help in estimating the time required for the test set-up to reach equilibrium. (2) It is also suggested that *C* and *U* values be calculated for the test specimen, utilizing known properties of the components. This will serve as general check of the measured results and avoid serious errors in measurement.

NOTE 6—The thermal mass of the apparatus may be the major factor contributing to the time constant of the system.

8.3 Data to be determined include:

8.3.1 The total net energy or average power through the specimen during a measurement interval. This includes all meter box heating and power to fans or blowers, and any corrections for meter box wall heat flow.

8.3.2 All air and surface temperatures specified in 6.5.1 and 6.5.2 (Note 7).

8.3.3 The effective dimensions of the metered area.

NOTE 7—In 6.5 the locations of thermocouples or temperature-measuring elements at various points are stipulated, for example, in the guard space and on the guard area of the test panel. The temperatures indicated by such thermocouples are of great value in evaluating the uniformity of temperatures prevailing in the guard space and on the test panel surfaces, but it is not feasible to stipulate generally the limits within which certain of these measured temperatures must agree. It must, therefore, be the responsibility of the test engineer to observe and weigh the significance of these temperatures to ascertain their effect upon the validity of a particular test measurement.

9. Calculation

9.1 Calculate the final test results by means of the following equations using the average data obtained in 8.2.2 for the two 4-h periods that agree within 1 %:

$$
\begin{aligned}
U &= Q/A(t_h - t_c)\\
C &= Q/A(t_1 - t_2)\\
R &= (t_1 - t_2)A/Q\\
R_u &= (t_h - t_c)A/Q = r_c + R + r_h\\
r_h &= (t_h - t_1)A/Q\\
r_c &= (t_2 - t_c)A/Q\\
h_h &= Q/A\,(t_h - t_1)\\
h_c &= Q/A\,(t_2 - t_c)\\
\lambda &= QL/A\,(t_1 - t_2)
\end{aligned}
$$

9.1.1 For a relatively uniform but nonhomogeneous specimen such as normal walls, floors, ceilings, etc., the properties that may be calculated are transmittance *U*, conductance *C*, resistance *R*, overall resistance R_u, surface resistances and surface conductances, *h*.

9.1.2 For uniform and homogeneous specimens all of the

---

[6] Practice C 1045 must be used in conjunction with Test Methods C 177 and Test Method C 518.

properties listed in 9.1.1 may be calculated plus thermal conductivity λ.

9.1.3 For elements smaller than the metering area, the properties that apply to the element, according to the distinctions of 9.1.1 and 9.1.2 may be calculated if tests have been run that allow the element heat flow to be determined. Annex A1 presents considerations for these calculations.

10. Report

10.1 Report the following information:

10.1.1 Name, and any other identification or description of the test construction, including if necessary a blueprint showing important details, dimensions, and all modifications made to the construction, if any, and specimen orientation. Description of the test construction and a complete and detailed description of all materials. This includes the generic name of the material and its density. (For hygroscopic materials, such as some concrete materials and wood, the moisture content should also be given). If the thermal conductivities of these materials, at the test conditions, have been measured in a hot box facility (Test Method C 236 or Test Method C 976), a guarded hot plate (Test Method C 177) or a heat flow meter (Test Method C 518), these values should also be included.

NOTE 8—By generic description, the name of the material in addition to the brand name should be given (for example, preformed, cellular polystyrene Type VIII with a density of 22 kg/m^3; spruce-pine-fir with a moisture content of 12 % and a dry density of 486 kg/m^3).

10.1.2 Pertinent information in regard to preconditioning of the test panel.

10.1.3 Size and dimensions of the metering and guard areas of the test panel.

10.1.4 Average values during the test period of the temperatures and velocities of the air on both sides of the metering area of the panel, and of the temperature of the surfaces on both sides. (If significant, give the average values of the temperature of specific areas of the surface of the panel.)

10.1.5 Average rate of net heat input to the metering box.

10.1.6 Any thermal transmission properties calculated in 9.1 and the known precision of the equipment. Precision of the equipment should be checked using the propagation of errors theory.

NOTE 9—Discussions of this method can be found in many textbooks on engineering experimentation and statistical analysis **(3)**.

10.1.7 Test duration and date.

10.2 All values shall be reported in both SI and inch-pound units unless specified otherwise by the client.

10.3 Where this test method is specifically referenced in published test reports and published data claims, and where deviations from the specifics of the test method existed in the tests used to obtain said data, the following statement shall be required to accompany such published information: "This test did not fully comply with the following provisions of Test Method C 236" (followed by a listing of specific deviations from this test method and any special test conditions that were applied).

11. Precision and Bias

11.1 *Background*—A round robin for guarded and calibrated hot boxes was conducted in accordance with Practice E 691. This round robin involved 21 different laboratories of which 16 had guarded hot boxes **(4)**. Data were reported for 100-mm (4-in.) thick homogeneous specimens of expanded polystyrene board (Specification C 578). Each laboratory received material from a special manufacturer's lot that was controlled to maintain a uniform density. Data reduction and analysis using Practice E 178 identified one of the 16 laboratories as a statistical outlier. Results from the other 15 laboratories showed that at a mean temperature (t) of 24°C (75°F), the average R value was determined to be 2.78 $K\cdot m^2/W$ (15.77 F ft^2 h/Btu). The regression equation for the data set was:

$$R = 3.146 - 0.016\,t\ (R \text{ in } K\cdot m^2/W \text{ and } t \text{ in } ^\circ C) \qquad (2a)$$

$$R = 17.867 - 0.028\,t\ (R \text{ in } F\,ft^2h/Btu \text{ and } t \text{ in } ^\circ F) \qquad (2b)$$

over the mean temperature range from 4°C to 43°C (40°F to 110°F). The mean specimen density ranged from 20.2 to 23.9 kg/m^3 (1.26 to 1.49 lbs/ft^3).

11.2 *Precision*—At a specimen thermal resistance of R = 2.78 $K\cdot m^2/W$ (15.76 F ft^2h/Btu) and on the basis of test error alone, the difference in absolute value of two test results obtained in different laboratories on the same specimen materials will be expected to exceed the reproducibility interval only 5 % of the time according to Table 2. For example, measurements from two different laboratories on the same specimen could differ by up to ±7.8 % at a mean temperature of 24°C (75°F) 95 % of the time.

11.3 *Bias*—Based on guarded hot plate data (Test Method C 177) from the National Institute of Standards and Technology—Center for Building Technology and supported by measurements from other laboratories, the true value for the round-robin specimen is a thermal resistance of 2.81 $K\cdot m^2/W$ (15.94 F ft^2h/Btu). The mean value measured by the guarded hot box differed by −1.07 %.

NOTE 10—Another test series was conducted on homogeneous common lot specimens in three guarded hot boxes at different laboratories. **(5, 6)** R-values of the specimens ranged from approximately 0.5 to 2.1 $K\cdot m^2/W$ (3 to 11.8 F ft^2h/Btu) at mean temperatures of 4, 24, and 43°C (40, 75, and 110°F). This series indicated that results with precision of ±5 % may be achieved.

NOTE 11—Both round robins used a homogeneous specimen, an ideal wall section. Actual wall sections will be nonhomogeneous. The precision and bias has not yet been determined for nonhomogeneous specimens. The above statements provide a bound.

12. Keywords

12.1 building assemblies; guarded hot box; test method; thermal performance; thermal resistance

TABLE 2 Precision for Test Method C 236

Mean Temperature, °C (°F)	Thermal Resistance, $K\cdot m^2/W$ (Fft^2h/Btu)	Reproducibility Interval, %	Change in R, $K\cdot m^2/W(Fft^2h/Btu)$
4 (40)	2.95 (16.75)	±7.3	±0.22 (±1.23)
24 (75)	2.78 (15.77)	±7.8	±0.22 (±1.23)
43 (110)	2.60 (14.79)	±8.6	±0.22 (±1.27)

ANNEX

(Mandatory Information)

A1. USING THE GUARDED HOT BOX TO DETERMINE HEAT TRANSFER THROUGH A BUILDING ELEMENT SMALLER THAN THE METERING AREA

A1.1 General Considerations

A1.1.1 In this use, the building cement area (A_a) is located centrally in the metering area (A_b) demarcated by the hot box, and is surrounded by a masking partition which extends homogeneously beyond the area A_b into the guard area. The area of the mask (A_m) within the metering area equals ($A_b - A_d$). The total heat flow rate Q_b determined by the hot box measurement consists of two heat flow rates in parallel, in accordance with the equation

$$Q_b = Q_r + Q_m \qquad \text{(A1.1)}$$

where Q_r is the total through the building element area A_∞ and Q_m is that through the mask area A_m.

A1.1.2 In conducting a test to ascertain Q_r for a particular building element, Q_b is determined by the hot box measurement, and Q_m is inferred from the results of calibration measurements. The calibration is made by means of hot box tests of the masking partition either before the aperture for the building element is cut out or with a blank of known thermal conductance installed in place of the building element. The error in Q_e is evidently equal to the difference of the algebraic errors in Q_b and Q_m. The fractional error is given by

$$\begin{aligned}\Delta Q_e/Q_e &= (\Delta Q_h - \Delta Q_m)/(Q_b - Q_m) \\ &= [(\Delta Q_h/Q_h) - (\Delta Q_m/Q_b)]/(1 - Q/Q_b)\end{aligned} \qquad \text{(A1.2)}$$

where (ΔQ_e) is the algebraic error in $Q\infty$ etc. An estimate of the fractional error $(\Delta Q_m)/Q_b$ is dependent upon the method which is used to calibrate the mask. If the calibration is made before the aperture for the building element is cut out then

$$\Delta Q_m/Q_h = (\Delta Q'_h/Q_h) \times (A_m/A_b) \qquad \text{(A1.3)}$$

where ($\Delta Q'_h$) is the error in heat flow measured during the calibration test. If a blank of known thermal conductance is used to calibrate the mask then

$$\Delta Q_m/Q_h = (\Delta Q'_h - \Delta Q_{b1})/Q_b \qquad \text{(A1.4)}$$

where (ΔQ_{b1}) is the algebraic error in determination of heat flow through the blank. Little can be said in general about the magnitudes of the algebraic fractional errors $(\Delta Q_h)/Q_b$ and $(\Delta Q_m)/Q_b$, since these depend on the quality and management of the particular hot box apparatus and upon the accuracy of determination of heat flow through the blank, but is is evident that the systematic portion of the error $(\Delta Q_r)/Q_r$ is reduced as Q_m/Q_b is made small. Also, as Q_m is made small, the term $(\Delta Q_m/Q_h)$ is presumably also made less significant. Thus, the fractional systematic error possible in the determination of Q_r is reduced by increasing either the area of the building element (if feasible), or the total thermal resistance of the mask.

A1.1.3 The need to infer the mask heat flow Q_m accurately requires that the mask be designed to act as a heat flow meter with an emf output and temperature difference of Δ_t proportional to the total heat flow through it. This consideration is the basis for the specific recommendations which follow.

NOTE A1.1—Additional error may arise due to the possible influences of the building element in causing two or three dimensional heat flow at the boundary with the mask and thus affecting the mask heat flow in regions adjacent to the element. Thus mask heat flow, determined under a given set of conditions with a calibration standard in place, may change when the building element is installed, even though the test conditions remain unchanged. The user of this procedure should be aware of such possible errors and should attempt to evaluate their magnitude in relation to the desired accuracy of the test.

A1.2 Recommendations

A1.2.1 It is recommended that the mask be made of a suitable uniform thickness of a homogeneous and stable material of low thermal conductivity having adequate strength to support the weight of the building elements to be tested. Suitable materials are faced high-density glass fiber or polystyrene boards laminated together as necessary. Stronger masks can be fabricated by sandwiching layers of insulation between layers of rigid materials such as plywood. Such masks, though nonhomogeneous, are uniform in the direction perpendicular to the direction of heat flow and are calibrated in the same manner as homogeneous masks. It may be necessary in some cases to incorporate framing in the mask to support heavy building elements such as heavy-duty metal frame windows or masonry sections. Such nonuniform masks are necessarily calibrated using blanks of known thermal conductance. Framing members must be kept away from the juncture with the building element and with the boundary of the metering area so as not to contribute excessively to lateral heat transfer at these points. It is important that the mask be low in hygroscopicity to minimize changes in its thermal resistance with ambient humidity conditions, and that it be substantially impervious to air flow through it.

A1.2.2 Thermocouples for measuring the temperature difference across the mask should be permanently installed uniformly flush with or just under its surfaces. These may be connected in series-differential for determination of the mask temperature difference, or as individual thermocouples for exploring the temperature distributions on the faces of the mask. It is recommended that there be at least eight thermocouple junctions on each face of uniform masks: four at positions bisecting the four lines from the corners of the building element aperture to the corresponding corners of the metering area, and four at positions bisecting the sides of the rectangle having the first four thermocouples at its corners. A suitable thermocouple arrangement would have to be chosen for nonuniform masks that would provide representative average surface temperatures. This is particularly important when natural convection is used and air temperatures and film coefficients vary over the metering surface. If framing members are used, an area-weighted average of temperatures measured over the members and away from them is necessary. The mask, as a heat flow meter, should be calibrated and used in terms of the average temperature (or thermocouple emf) difference across it indicated by the permanently installed thermocouples.

A1.2.3 To protect the surfaces of the mask and the permanently installed thermocouples, and if necessary to render the surfaces impervious to air, a permanent coating or thin facing on each face of the mask is desirable. However, the coating or facing must be of low conductance laterally so that it does not contribute excessively to lateral heat transfer at the juncture with the building element or at the boundary of the metering area. The emittance of the mask surfaces should be uniform, and unchanged after calibration of the mask; in cases where the transmittance (rather than the conductance) of the building element is of particular interest, it is preferable that the emittance of the mask surfaces be large.

A1.2.4 In view of the desirability of high thermal resistance of the mask relative to that of the building element, the uniform thickness of the mask should in general not be less than that of the building elements to be tested, and may be greater than that of the thinner elements. Mask thickness greatly exceeding that of the building element is to be avoided if possible because of lateral heat flow in the mask due to its exposure at uncovered areas of its aperture. (In special instances, for example, a window designed to be set a few inches outward from the plane of the inner surface of a wall, a special calibration of the mask as a heat flow meter may be necessary using a blank of known thermal conductance in the precise position of the window at the juncture with the mask aperture.)

A1.2.5 The mask aperture in which the building element is installed for test should fit the element specimen snugly. Cracks between them should be minimal in width, and should be filled completely with a good fibrous insulation and caulked or otherwise sealed at the mask surfaces to prevent air leakage. It is desirable that the insulation used to fill cracks have approximately the same conductivity as the mask material; it would then be possible, if the cracks aggregate an area significant in relation to the mask area, to compensate roughly for the increased virtual mask area by increasing the mask heat flow indicated by its temperature drop in proportion to the increase of area.

A1.2.6 It is probable that many building elements to be tested are inhomogeneous or nonuniform in construction for structural reasons, and in consequence that the local thermal conductances differ considerably at different frontal areas of the element. The variations are inherent, and the result of the test is an average conductance or transmittance value for the total construction, provided that the conductance variations at edges do not seriously impair the validity of using the mask as an adequate heat flow meter. This is a matter which varies with the case, and therefore must rest on the judgment and discretion of those conducting the test measurement. A useful guiding principle is that nothing should be incorporated in, or omitted from, a building element specimen being tested that would make it not representative of the assembly that would be found in actual installation in service. For example, if a metal window ordinarily is installed with inset wood framing, the test specimen should include just so much of the wood framing as is properly chargeable to it.

A1.3 Calibration of the Mask as a Heat Flow Meter

A1.3.1 The calibration of the mask is made by means of hot box tests either before the aperture for the building elements is cut out or with a blank of known thermal conductance installed in place of the building element. The mask must be fully prepared with the permanent differential thermocouples installed and any final facings or coatings applied. Several tests are made, adequately covering the range of mask mean temperatures (and perhaps mask temperature drops and box air velocities) at which the mask will be operated in tests of building elements. In each test, under steady-state conditions, the metering box heat flow Q'_t and the corresponding mask temperature drop Δ_t, indicated by its permanently installed thermocouples, are determined. The net mask heat flow Q'_m corresponding to Δt is calculated as $Q'_b(A_m/A_b)$ when the calibration is made before the aperture is cut, where A_m and A_b are as defined earlier, and as $(Q'_i - Q_{b1})$ for the calibrated-blank method where Q_{b1} is the calculated heat flow through the blank. In the latter method of calibration, a suitable blank must first be prepared and calibrated.

APPENDIX

(Nonmandatory Information)

X1. THERMOPILE EMF AND HEAT FLOW RELATIONSHIP (7, 8)

X1.1 The procedure given in this Appendix outlines the steps suggested to obtain the relationship between heat flow and thermopile output. This method addresses the technique that will yield the heat flow relationship as a function of the thermopile output and a thermopile offset, if present.

X1.1.1 It is essential that the number of fans and power input in the metering, guard, and environmental boxes be held constant along with all temperatures throughout the calibration (and measurement) phase. By holding the fan number and input along with the surface temperatures constant, the operator assures a constant heat transfer film coefficient to the specimen throughout testing. The E_o value associated with negligible net heat flow across the meter box walls is then obtained from the relationship between Q_m and E. The equation that describes the total heat flow drawn schematically in Fig. 4 is:

$$Q_F + Q_H + Q_m = Q_s = \frac{A \Delta T}{R} \qquad \text{(X1.1)}$$

where:

Q_F = heat flow due to fan, W,
Q_H = heat flow due to heater, W,
Q_m = heat flow through the metering box walls, W,
Q_s = heat flow through specimen, W,
R = thermal resistance of specimen, $m^2 \cdot K/W$,
A = heat flow metered area, m^2,
ΔT = temperature difference across specimen, K,
E_o = thermopile emf when net heat flow through metering box walls is negligible, and
E = thermopile emf.

The goal is to make Q_m equal to zero. Q_m can be described by:

$$Q_m = fn\,(E) = m\,E + b \qquad \text{(X1.2)}$$

X1.1.2 To quantify m, at least two test runs must be performed with differing levels of E. E must be held constant within each test. The specimen surface to surface temperature difference for all tests must be constant and of the same value. Q_s can be approximated by assuming the design R. It is not necessary to know the R of the specimen. Plot Q_m calculated from Eq X1.1 versus E. The slope of the line is m.

The next step is to quantify b in Eq 2. Set the temperature difference across the specimen surface equal to zero ($Q_s = 0$). Substituting Eq X1.2 into Eq X1.1 and setting $Q_s = 0$ reduces Eq X1.1 to:

$$Q_F + Q_H = -(m\,E + b) \qquad \text{(X1.3)}$$

X1.1.3 Set E to a value such that the fan wattage is at operational conditions and the heater wattage is at the minimum value that maintains temperature control. This will assure that no heat is flowing anywhere except through the meter box walls. During this test, lateral heat flow must still be negligible. Using m that was determined, Eq X1.3 will yield b. The thermopile emf value that pertains to negligible net heat flow through the meter box walls E_o can then be calculated using Eq X1.2:

$$E_o = -(b/m) \qquad \text{(X1.4)}$$

REFERENCES

(1) Miller, R. G., "Hot Box Operating Techniques and Procedures: A Survey," *Journal of Testing and Evaluation*, Vol 15, No. 3, May 1987, pp. 153–166.

(2) Rohsenow, W. M., Choi, H. I., *Heat, Mass and Momentum Transfers*, Prentice-Hall, Inc., Englewood Cliffs, NJ, 1961, p. 112.

(3) Schenck, Hilbert, *Theory of Engineering Experimentation*, McGraw-Hill, NY, NY, Second Ed., 1968, Chap. 3, p. 46.

(4) Bales, E., "ASTM/DOE Hot Box Round Robin," ORNL/Sub/84-97333/2, Oak Ridge National Laboratory, Oak Ridge, TN, November 1988.

(5) Miller, R. G., Perrine, E. L., and Linehan, P. W., "A Calibrated/Guarded Hot Box Test Facility," *Thermal Transmission Measurements of Insulation, ASTM STP 660*, 1978, p. 329.

(6) Sherman, M., "Aged Thermal Resistance (*R*-value) of Foil-Faced Polyisocyanurate Foam," Thermal Insulation Board Proceedings of the ASHRAE/DOE-DEHL Conference on *Thermal Performance of External Envelopes of Buildings*, ASHRAE SP 28, 1981, pp. 952–964.

(7) Gerace, R. R., Derderian, G. D., Cirignano, P. C., Orlandi, R. D. and Shu, L. S., "An Operational Procedure for Guarded Hot Box Testing," *Journal of Testing and Evaluation*, Vol 15, No. 3, May 1987, pp. 138–144.

(8) Orlandi, R. D., Howanski, J. W., Derderian, G. D., and Shu, L. S., "Development of a Testing Procedure for a Guarded Hot Box Facility," *Symposium on Thermal Insulation, Materials, and Systems for Energy Conservation in the 80's, ASTM STP 789*, 1982, pp. 205–214.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM C330: Standard Specification for Lightweight Aggregates for Structural Concrete

CFR Section(s): 30 CFR 250.901(a)(18)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: C 330 – 99

Standard Specification for Lightweight Aggregates for Structural Concrete[1]

This standard is issued under the fixed designation C 330; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers lightweight or sand aggregates intended for use in structural concrete in which prime considerations are reducing the density while maintaining the compressive strength of the concrete. Procedures covered in this specification are not intended for job control of concrete.

1.2 The values stated in SI units are to be regarded as the standard. The values shown in parentheses are for information purposes only.

1.2.1 With regard to other units of measure, the values stated in inch-pound units are to be regarded as standard.

1.3 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

NOTE 1—This specification is regarded as adequate to ensure satisfactory lightweight aggregates for most concrete. It is recognized that it may be either more or less restrictive than needed for some conditions and for special purposes, such as fire resistance, fill, and concrete constructions, the use of which is based on load tests rather than conventional design procedures.

2. Referenced Documents

2.1 *ASTM Standards:*

C 29/C 29M Test Method for Unit Weight and Voids in Aggregate[2]

C 33 Specification for Concrete Aggregates[2]

C 39 Test Method for Compressive Strength of Cylindrical Concrete Specimens[2]

C 40 Test Method for Organic Impurities in Fine Aggregates for Concrete[2]

C 114 Test Methods for Chemical Analysis of Hydraulic Cement[3]

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates[2]

C 142 Test Method for Clay Lumps and Friable Particles in Aggregates[2]

C 151 Test Method for Autoclave Expansion of Portland Cement[3]

C 157 Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete[2]

C 192 Practice for Making and Curing Concrete Test Specimens in the Laboratory[2]

C 496 Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens[2]

C 567 Test Method for Unit Weight of Structural Lightweight Concrete[2]

C 641 Test Method for Staining Materials in Lightweight Concrete Aggregates[2]

C 666 Test Method for Resistance of Concrete to Rapid Freezing and Thawing[2]

D 75 Practice for Sampling Aggregates[4]

3. General Characteristics

3.1 Two general types of lightweight aggregates are covered by this specification, as follows:

3.1.1 Aggregates prepared by expanding, pelletizing, or sintering products such as blast-furnace slag, clay, diatomite, fly ash, shale, or slate, and

3.1.2 Aggregates prepared by processing natural materials, such as pumice, scoria, or tuff.

3.2 The aggregates shall be composed predominately of lightweight-cellular and granular inorganic material.

4. Chemical Composition

4.1 Lightweight aggregates shall not contain excessive amounts of deleterious substances, as determined by the following limits:

4.1.1 *Organic Impurities (Test Method C 40)*—Lightweight aggregates that, upon being subjected to test for organic impurities, produce a color darker than the standard shall be rejected, unless it is demonstrated that the discoloration is due to small quantities of materials not harmful to the concrete.

4.1.2 *Staining (Test Method C 641)*—An aggregate producing a heavy or very heavy stain shall be rejected when the

---

[1] This specification is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C 09.21 on Lightweight Aggregates.

Current edition approved March 10, 1999. Published March 1999. Originally published as C 330 – 53 T. Last previous edition C 330 – 98.

[2] *Annual Book of ASTM Standards,* Vol 04.02.

[3] *Annual Book of ASTM Standards,* Vol 04.01.

[4] *Annual Book of ASTM Standards*, Vol 04.03.

material making up the stain is found upon chemical analysis to contain an iron content, expressed as Fe_2O_3, equal to or greater than 1.5 mg/200 g of sample.

4.1.3 *Loss on Ignition (Methods C 114)*—The loss on ignition of lightweight aggregates shall not exceed 5 %.

NOTE 2—Certain processed aggregates may be hydraulic in character, and may be partially hydrated during production; if so, the quality of the product is not usually reduced thereby. Therefore, consideration should be given to the type of material when evaluating the product in terms of ignition loss.

5. Physical Properties

5.1 Lightweight aggregate under test shall meet the following requirements:

5.1.1 *Clay Lumps*—The amount of clay lumps shall not exceed 2 % by dry weight.

5.1.2 *Grading*—The grading shall conform to the requirements shown in Table 1.

5.1.3 *Uniformity of Grading*—To ensure reasonable uniformity in the grading of successive shipments of lightweight aggregate, fineness modulus shall be determined on samples taken from shipments at intervals stipulated by the purchaser. If the fineness modulus of the aggregate in any shipment differs by more than 7 % from that of the sample submitted for acceptance tests, the aggregate in the shipment shall be rejected, unless the supplier demonstrates that it will produce concrete of the required characteristics.

5.1.4 *Bulk Density (Loose)*—The bulk density (loose) of the lightweight aggregates shall conform to the requirements shown in Table 2.

5.1.5 *Uniformity of Bulk Density (Loose)*—The reported bulk density (loose) of lightweight aggregate shipments, sampled and tested, shall not differ by more than 10 % from that of the sample submitted for acceptance tests, but the dry loose bulk density shall not exceed the limits in Table 2.

5.2 Concrete specimens containing lightweight aggregate under test shall meet the following requirements:

TABLE 2 Bulk Density (Loose) Requirements of Lightweight Aggregates for Structural Concrete

Size Designation	Maximum Dry Loose Bulk Density kg/m³ (lb/ft³)
Fine aggregate	1120 (70)
Coarse aggregate	880 (55)
Combined fine and coarse aggregate	1040 (65)

5.2.1 *Compressive Strength (Test Method C 39), Density (Test Method C 567), and Splitting Tensile Strength (Test Method C 496)* —Compressive strength and density shall be an average of three specimens and the splitting tensile strength shall be the average of eight specimens. It shall be possible to produce structural concrete using the lightweight aggregates under test, so that from the same batch of concrete one or more of the compressive strength requirements and splitting tensile strength requirements in the following table will be satisfied without exceeding the corresponding maximum unit weight values.

Average Air Dry 28-day Density max, kg/m³ (lb/ft³)	Average 28-day Splitting Tensile Strength, min, (MPa) psi	Average 28-day Compressive Strength, min, (MPa) psi
	All Lightweight Aggregate	
1760 (110)	2.2 (320)	28 (4000)
1680 (105)	2.1 (300)	21 (3000)
1600 (100)	2.0 (290)	17 (2500)
	Sand/Lightweight Aggregate	
1840 (115)	2.3 (330)	28 (4000)
1760 (110)	2.1 (310)	21 (3000)
1680 (105)	2.1 (300)	17 (2500)

NOTE 3—Intermediate values for strength and corresponding density values shall be established by interpolation. Materials that do not meet the minimum average splitting tensile strength requirement may be used provided the design is modified to compensate for the lower value.

5.2.2 *Natural Sand*— Natural sand, when used to replace part, or all, of the lightweight-aggregate fines shall comply with the applicable requirements of Specification C 330. The test report shall record the proportion of all ingredients and the characteristics of the natural sand to ensure compliance with these minimum requirements.

5.2.3 *Drying Shrinkage*— The drying shrinkage of concrete specimens prepared and tested as described in the method for preparation of samples for shrinkage of concrete shall not exceed 0.07 %.

5.2.4 *Popouts*—Concrete specimens prepared as described in the method for preparation of sample for shrinkage of concrete and tested in accordance with Test Method C 151 shall show no surface popouts.

5.2.5 *Resistance to Freezing and Thawing*—When required, the aggregate supplier shall demonstrate by test or proven field performance that the lightweight aggregate when used in

TABLE 1 Grading Requirements for Lightweight Aggregates for Structural Concrete

Size Designation	Percentages (Mass) Passing Sieves Having Square Openings								
	25.0 mm (1 in.)	19.0 mm (¾) in.)	12.5 mm (½ in.)	9.5 mm (⅜ in.)	4.75 mm (No. 4)	2.36 mm (No. 8)	1.18 mm (No. 16)	300 µm (No. 50)	150 µm (No. 100)
Fine aggregate:									
4.75 mm to 0	...	...	...	100	85–100	...	40–80	10–35	5–25
Coarse aggregate:									
25.0 m to 4.75 mm	95–100	...	25–60	...	0–10	...	...	...	...
19.0 mm to 4.75 mm	100	90–100	...	10–50	0–15	...	...	...	...
12.5 mm to 4.75 mm	...	100	90–100	40–80	0–20	0–10	...	...	...
9.5 mm to 2.36 mm	...	...	100	80–100	5–40	0–20	0–10	...	...
Combined fine and coarse aggregate:									
12.5 mm to 0	...	100	95–100	...	50–80	...	...	5–20	2–15
9.5 mm to 0	...	...	100	90–100	65–90	35–65	...	10–25	5–15

concrete, had the necessary resistance to freezing and thawing to perform satisfactorily in its intended use.

6. Sampling

6.1 Sample lightweight aggregates in accordance with Practice D 75.

7. Number of Tests

7.1 *Tests on Aggregate*—One representative sample is required for each test for organic impurities, staining, loss on ignition, grading, unit weight, and clay lumps.

7.2 *Tests on Concrete*—At least three specimens are required for each of the following tests of concrete: compressive strength, shrinkage, density, resistance to freezing and thawing, and presence of popout materials. At least eight concrete specimens are required for splitting tensile strength tests.

8. Test Methods

8.1 *Compressive Strength (Test Method C 39)*—Make test specimens in accordance with Practice C 192. Cure specimens in accordance with Practice C 192 until the time of test, or follow the curing procedures for the air dry density (Test Method C 567). When the latter procedure is used, remove the specimens from the moist curing at the age of 7 days and store at 23 ± 2°C (73.5 ± 3.5°F) with a relative humidity of 50 ± 5 % until the time of test.

8.2 *Splitting Tensile Strength*—Make 152 by 305 mm (6 by 12 in.) cylindrical test specimens in accordance with Practice C 192, cure, and test in accordance with Test Method C 496.

8.3 *Density of Concrete (Test Method C 567)*—Follow the procedures in Test Method C 567.

8.4 *Shrinkage of Concrete (Test Method C 157)*—Follow the procedures of Test Method C 157 with the following exceptions:

8.4.1 Prepare the concrete mixture using 335 kg of cement/m^3 (564 lb/yd^3), admixture (if any), and with an air content of 6 ± 1 %. Adjust the water content so as to produce a slump of 50 to 100 mm (2 to 4 in.). Thoroughly consolidate the concrete in steel molds not smaller than 50 by 50 mm (2 by 2 in.) nor larger than 100 by 100 (4 by 4 in.) in cross section, and long enough to provide a 250 mm (10 in.) gage length. The surface of the concrete shall be steel troweled.

8.4.2 *Curing*—To prevent evaporation of water from the unhardened concrete, cover the specimen with a nonabsorptive, nonreactive plate or sheet of tough, durable, impervious plastic or wet burlap. When wet burlap is used for covering, the burlap must be kept wet until the specimens are removed from the molds (see Note 4). Remove specimens from the molds not less than 20 nor more than 48 h after casting and store in a moist room maintained at 23 ± 2°C (73.5 ± 3.5°F) with a relative humidity of not less than 95 %. At the age of 7 days, remove the specimens from the moist room, measure for length, and store in a curing cabinet maintained at 37.8 ± 1.1°C (100 ± 2°F) with a relative humidity of 32 ± 2 %.

NOTE 4—Placing a sheet of plastic over the burlap will facilitate keeping it wet.

NOTE 5—The air immediately above a saturated solution of magnesium chloride ($MgCl_2$) at 37.8°C (100°F) is approximately 32 % relative humidity.

8.4.3 *Report*—After storage in the cabinet for 28 days, determine the change in length of each specimen to the nearest 0.01 % of the effective gage length. Report the change in length as the drying shrinkage of the specimen; report the average drying shrinkage of the specimens as the drying shrinkage of the concrete.

8.5 *Test for Popout Materials*—Prepare concrete specimens for the test for popout materials as described in method for preparation of samples for shrinkage of concrete. Cure and autoclave the specimens in accordance with Test Method C 151. Visually inspect the autoclaved specimens for the number of popouts that have developed on the surface. Report the average number of popouts per specimen.

8.6 *Test for Freezing and Thawing*— Make freezing and thawing tests of concrete, when required, in accordance with Test Method C 666, with the following modification to the section on Test Specimens and on Procedure. Unless otherwise specified, remove the lightweight aggregate concrete specimens from moist curing at an age of 14 days and allow to air dry for another 14 days exposed to a relative humidity of 50 ± 5 % and a temperature of 23 ± 2°C (73.5 ± 3.5°F). Then submerge the specimens in water for 24 hours, prior to the freezing and thawing test.

8.7 *Grading (Method C 136)*—Follow the procedures of Method C 136, except that the mass of the test sample for fine aggregate shall be in accordance with Table 3. The test sample for coarse aggregate shall consist of 2830 cm^3 (0.1 ft^3) or more of the material used for the determination of bulk density. Mechanical sieving of aggregate shall be for 5 minutes..

8.8 *Bulk Density (Loose) (Test Method C 29)*—The aggregate shall be tested in an oven dry condition utilizing the shoveling procedure.

8.9 *Clay Lumps and Friable Particles in Aggregates*—Test Method C 142.

9. Rejection

9.1 Material that fails to conform to the requirements of this specification shall be subject to rejection. Rejection shall be reported to the producer or supplier promptly and in writing.

10. Certification

10.1 When specified in the purchase order or contract, a producer's or supplier's certification shall be furnished to the purchaser that the material was manufactured, sampled, and tested in accordance with this specification and has been found to meet the requirements. When specified in the purchase order

TABLE 3 Mass of Sieve Test Sample for Fine Lightweight Aggregates

Range of Nominal Bulk Density (Loose) of Aggregate		Weight of Test Sample, g
kg/m^3	lb/ft^3	
80–240	5–15	50
240–400	15–25	100
400–560	25–35	150
560–720	35–45	200
720–880	45–55	250
880– 1040	55–65	300
1040–1120	65–70	350

or contract, a report of the test results shall be furnished.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM C509: Standard Specification for Elastomeric Cellular Preformed Gasket and Sealing Material

CFR Section(s): 24 CFR 200, Subpart S

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: C 509 – 84

Standard Specification for
CELLULAR ELASTOMERIC PREFORMED GASKET AND SEALING MATERIAL[1]

This standard is issued under the fixed designation C 509; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification applies to those cellular elastomeric compounds of a firm grade that are manufactured in preformed shapes for use as gaskets and for use as sealing materials, in the form of compression seals or gaskets, or both, for glazing purposes.

NOTE 1—For softer cellular elastomeric materials used in secondary sealing applications, refer to Specification D 1056.

1.2 The following precautionary caveat pertains only to the test method portion, Section 11, of this specification: *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Documents

2.1 *ASTM Standards:*

D 395 Test Methods for Rubber Property—Compression Set[2]

D 412 Test Methods for Rubber Properties in Tension[2,3]

D 746 Test Method for Brittleness Temperature of Plastics and Elastometers by Impact[3]

D 865 Test Method for Rubber Deterioration by Heating in Air (Test Tube Enclosure)

D 925 Test Methods for Rubber Property—Staining of Surfaces (Contact, Migration, and Diffusion)[2]

D 1056 Specification for Flexible Cellular Materials—Sponge or Expanded Rubber[3]

D 1149 Test Method for Rubber Deterioration—Surface Ozone Cracking in a Chamber (Flat Specimens)[2]

3. Terminology

3.1 *Definition:*

3.1.1 *cellular elastomer*—a cured elastomeric material containing cells or small voids.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *compression seal*—a type of joint seal in which weathertightness is maintained by the exertion of compressive pressure on the gasket or sealing material.

3.2.2 *gasket glazing*—a method of setting glass or panels in prepared openings, using a preformed gasket to obtain a weathertight seal.

3.2.3 *preformed gasket*—an elastomeric compound molded in the form of a continuous strip, channel, or other shape, for use in filling joints and providing weathertight seals in glazing or between building components.

3.2.4 *sealing material*—any material intended for use in providing weathertight seals in building applications.

4. Significance and Use

4.1 *Flame Propagation:*

---

[1] This specification is under the jurisdiction of ASTM Committee C-24 on Building Seals and Sealants and is the direct responsibility of Subcommittee C24.72 on Compression Seal Gaskets.

Current edition approved Oct. 26, 1984. Published February 1984. Originally published as C 509 – 63 T. Last previous edition C 509 – 79.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

[3] *Annual Book of ASTM Standards*, Vol 09.02.

4.1.1 This specification has two options:

4.1.1.1 *Option I*—Flame propagation test is required.

4.1.1.2 *Option II*—Flame propagation test is not required.

4.1.2 In case no option is specified, Option I will apply.

4.1.3 *The sections of this specification applicable to flame propagation should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.*

4.2 This specification has two classifications as related to ozone resistance. These are Type I and Type II, with the latter having the greater resistance to ozone. The type should be specified when making reference to this specification but in the event that the type is not specified, Type I shall apply.

NOTE 2—Type II is included in this specification for use where greater ozone resistance is required.

5. Materials and Manufacture

5.1 Cellular elastomeric materials furnished to this specification shall be manufactured from natural rubber, synthetic rubber, rubber-like materials, or mixtures of these, with added compounding ingredients of such nature and quality that, with proper curing, the finished product will comply with this specification.

5.2 The cured compounds shall be suitable for use where resistance to sunlight, weathering, oxidation, and permanent deformation under load are of prime importance.

5.3 The manufacturing process shall be such to ensure a homogeneous cellular material free of defects that may affect serviceability.

5.4 Although under this specification the manufacturer is permitted to choose constituent materials, there is no implication that the several compounds are equivalent in all physical properties. Any special characteristics other than those required by this specification, which may be needed for specific applications, shall be specified by the purchaser, since such characteristics may influence the choice of base materials and other ingredients.

6. Physical Properties

6.1 The material shall conform to the requirements prescribed in Table 1.

7. Dimensional Tolerances

7.1 Permissible variations in dimensions of the various forms shall be ±6 % unless otherwise agreed upon between the purchaser and the supplier.

8. Workmanship, Finish, and Appearance

8.1 The cellular elastomeric materials shall be manufactured and processed in a careful and workmanlike manner in accordance with the best commercial practices.

8.2 The surfaces of the finished material shall be reasonably smooth and free of excessive talc or bloom.

8.3 Unless otherwise specified, the material shall be black. When colored material is desired, it is recommended that other tests, agreed upon between the purchaser and the supplier, be conducted to ensure color stability.

9. Sampling

9.1 When possible, the completed manufactured product of a suitable section thereof shall be used for the tests specified. Representative samples of the lot being examined shall be selected at random as required.

9.2 When the finished product does not lend itself to testing or to the taking of test specimens because of complicated shape, small size, metal or fabric inserts, or other reasons, standard test strips shall be prepared. The standard extruded specimens for testing, except where a specific specimen size is defined by a particular test method, shall be 6.4 mm (¼ in.) thick by 32 mm (1¼ in.) wide in rectangular cross section. The test pieces for flame propagation tests shall be as specified in 11.8.2. All test pieces shall be made from the same compound and shall have the same apparent density and state of cure as the product they represent.

9.3 The tests for dimensional stability, ozone resistance, water absorption, and nonstaining may be made on samples from the material to be shipped or on samples representative of it. Tests for compression deflection, compression set, heat aging, flame propagation, and low-temperature brittleness may be made on standard samples previously prepared in accordance with 9.2.

10. Number of Tests and Retests

10.1 Any material that fails in one or more of the test requirements may be retested by making two additional tests for the requirements in which failure occurs. Failure in one such retest shall be cause for final rejection.

10.2 Rejected material shall be disposed of as directed by the supplier.

11. Test Methods

11.1 *Compression - Deflection* — Specification D 1056. Base calculations of compression-deflection on the original thickness of the specimens.

11.2 *Compression Set*—Test Methods D 395, Method B.

11.3 *Compression Deflection After Heat Aging:*

11.3.1 A 152-mm (6-in.) length of the finished extrusion shall be heat aged along with the specimen for Specification D 1056 and shall pass the requirements of Table 1, Footnote B.

11.3.2 Test for compression-deflection by first aging the specimen (a piece of appropriate size for the compression-deflection test, instead of the dumbbell-shaped tension specimen) in accordance with Test Method D 865, then measuring the compression-deflection value in accordance with Specification D 1056.

11.3.3 The specimen for heat aging shall be large enough to allow the taking of the appropriate number and six size of specimens as defined by Specification D 1056. The cutting of specimens for Specification D 1056 shall be done after the heat aging has been performed.

11.4 *Dimensional Stability After Heat Aging*—Determine the dimensional stability by subjecting a 150-mm (6-in.) length of the extruded shape to heat aging for 70 h at 100°C (212°F) in accordance with Test Method D 865. After aging, the changes in length and breadth dimensions of the specimen shall not exceed 4 %.

11.5 *Ozone Resistance*—Test Method D 1149. The concentration of ozone shall be 100 MPa for Type I and 300 MPa for Type II. The time of test shall be 100 h at 40°C (104°F) with a specimen as defined by 9.2 with a length of 152 mm (6 in.) and with a specimen elongation of 40 %.

11.6 *Low - Temperature Brittleness*—See Appendix X1.

11.7 *Water Absorption*—Test for water absorption by placing ten 460-mm (18-in.) strips of the elastomer, bent in a U-form, in water at 21.1°C (70°F) for a period of 24 h. Submerge only the center 406 mm (16 in.) of the specimen with the bottom of the loop 152 mm (6 in.) below the water line, and the cut ends of the specimen out of the water. After removal from the water, blot the specimens with lint-free paper before weighing. The test value is the average weight of water absorbed by the ten specimens.

11.8 *Flame Propagation*—This method determines whether or not the gasket will propagate flame, with no significance being attached to such matters as fuel contribution, rate of flame spread, smoke generation, or nature and temperature of products of combustion.

11.8.1 *Apparatus:*

11.8.1.1 The test chamber may be any enclosure that will permit normal gravity circulation of air past the specimen during burning. A hood or ventilated spray booth is recommended in order to remove any noxious products of combustion, provided the velocity of air past the specimen does not exceed 18.3 m (60 ft)/min.

NOTE 3—Air velocities greater than 18.3 m/min will have an extinguishing effect upon any flame and will present an unrealistic evaluation of the flame resistance of the material.

11.8.1.2 The burner shall be a bunsen burner with a barrel nominally 9.5 mm (0.38 in.) in diameter.

11.8.1.3 The fuel shall be ordinary fuel gas at a normal pressure.

11.8.2 The test specimen shall be 13 mm (½ in.) thick, 25 mm (1 in.) wide, and 460 mm (18 in.) long made in accordance with 9.2. It shall be free of any permanent set producing a curved section within the specimen that will not permit it to hang vertically, and it shall be free of abnormally porous sections and foreign materials.

NOTE 4—This method recognizes the fact that every flame, no matter how hot, has a kindling height above which its temperature is too low to kindle the specimen. For the specified flame, this height is considerably less than 460 mm (18 in.), so if a specimen burns above this kindling height, it does so on its own heat of combustion. Therefore it is considered to propagate flame.

11.8.3 *Procedure:*

11.8.3.1 Secure the specimen in a vertical position within the test chamber using a ring stand with a clamp positioned at the top of the specimen. Locate the specimen high enough to permit the burner to be placed beneath it. Use two wire loops to retain the position of the sample over the flame. Place one wire loop 51 mm (2 in.) from the end to be ignited, the other 127 mm (5 in.) from the end to be ignited. Fasten the wire loops to the stand holding the specimen.

11.8.3.2 Light and adjust the burner so as to produce a blue flame approximately 38 mm (1.5 in.) high.

11.8.3.3 Place the lighted burner directly below the specimen so that the tip of the inner cone of the flame just touches the lowest part of the specimen. Allow the burner to remain in this position for 5 min, then remove it.

11.8.3.4 After removal of the burner, remove loose char with a stiff brush and measure the remaining length of the specimen. Consider the unburned length to be that remaining after removal of loose char.

11.8.4 *Report*—The report should state the distance of flame propagation expressed in millimetres (or inches). The distance of flame propagation equals original length less unburned length.

11.9 *Nonstaining*—Test Methods D 925, Method B. The surface against which stain is to be tested and the acceptable degree of staining shall be specified by the purchaser.

12. Inspection

12.1 All tests and inspections shall be made at the place of manufacture prior to shipment unless otherwise specified. The supplier shall provide the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with this specification.

13. Certification

13.1 When required, the supplier shall furnish the purchaser with a certified test report giving the results of the tests required to determine conformance with all requirements specified herein.

14. Packaging and Package Marking

14.1 All material shall be properly separated according to compound, size, etc., and shall be packaged and labeled in accordance with the best commercial practice with ample protection against damage in shipment.

Table 1 Physical Requirements of Cellular Elastomeric Materials

Property	Limit	ASTM Test Method[A]
Compression-deflection, 25 % deflection limits:		
kPa	91 to 168	D 1056
psi	13 to 24	
Compression set, 22 h @ 70°C (158°F) max, %	30	D 395, Method B
Heat aging[B], 70 h @ 100°C (212°F), change in compression-deflection values:		
kPa	0 to +70	D 865 and D 1056
psi	0 to +10	
Dimensional stability, change, max %, after heat aging, 70 h @ 100°C (212°F)	4	11.4
Ozone resistance[C] at 40 % elongation, 100 h @ 40°C (104°F):		
Type I 100 MPa ozone	no cracks	D 1149
Type II 300 MPa ozone	no cracks	D 1149
Low-temperature brittleness @ −40°C (−40°F)	pass	see Appendix X1
Water absorption, max, % weight	5.0	11.7
Flame propagation:		
Option I	100 mm (4 in.) max.	11.8
Option II	no limit	
Nonstaining[D]	no migratory stain	D 925

[A] See Section 11.

[B] After heat aging, surfaces of the specimen shall be neither hard nor brittle. A 150-mm (6-in.) length of the finished extrusion shall exhibit no surface cracks when bent on itself 180°.

[C] The specimen shall exhibit no surface cracks when in the extended condition.

[D] This requirement may be waived, subject to agreement between the purchaser and the supplier.

APPENDIX

(Nonmandatory Information)

X1. TEST METHOD FOR LOW-TEMPERATURE BRITTLENESS OF RUBBER AND RUBBER-LIKE MATERIALS[4]

X1.1 Scope

X1.1.1 This test method is intended to determine the ability of compounds made from rubber or rubber-like materials to resist the effect of low temperatures that may cause them to become brittle and fracture or crack when bent. Standard specimens are exposed to specified low temperatures for definite periods after which the specimens are bent in a prescribed manner and any fracture or cracking noted. The procedure is commonly called the "Thiokol" method.

NOTE X1.1—Results obtained by this test method are influenced by the rate of flexing of the cooled specimens which can not be closely controlled in the prescribed apparatus. They are therefore of a qualitative nature and may not be closely reproducible over a range of several degrees of temperature depending on the speed of flexure. For more accurate determination of brittle temperature, and particularly in new specifications, Test Method D 746 is recommended.

X1.2 Apparatus

X1.2.1 *Cold Chamber,* of sufficient size to contain the flexing fixture when loaded with specimens, and so arranged as to permit the operation of the fixture to bend to specimens without removal from the chamber. It shall be capable of maintaining within it a uniform atmosphere of cold, dry air or a mixture of air and carbon dioxide at specified temperatures within a tolerance of ±1°C (2°F).

NOTE X1.2—Temperatures of −40°C (−40°F) and −55°C (67°F) are commonly used.

X1.2.2 *Flexing Fixture,* consisting of two parallel plates each having a width of at least 50 mm (2 in.) so supported in guides that they may be rapidly moved from a position 63 mm (2½ in.) apart until they are separated by a distance of 25 mm (1 in.). Suitable clamping bars or devices shall be provided for holding the ends of the specimens for a distance of 6.4 mm (¼ in.) at the corresponding edge of each plate so that when mounted, the specimens form similar bent loops between the plates. A satisfactory flexing fixture is shown in Fig. X1.1.

X1.3 Test Specimens

X1.3.1 The test specimens shall conform in shape to Die C as specified in Methods D 412 and shall have a thickness of 2.032 ± 0.254 mm (0.080 ± 0.010 in.).

X1.3.2 At least two specimens from each compound shall be tested.

X1.4 Procedure

X1.4.1 Mount the test specimens in loop position between the plates of the flexing fixture with the enlarged ends spaced at least 3.2 mm (⅛ in.) apart and held in the clamps for a distance of 6.4 mm (¼ in.). With the plates in the open position separated 63 mm (2½ in.), place the fixture containing the specimens in the cold chamber and expose it for the specified period to cold, dry air or a mixture of air and carbon dioxide at the specified temperature. The standard exposure period shall be 5 h (Note X1.3). At the termination of the exposure period and while still in the cold chamber, move the plates of the flexing fixture as rapidly as possible from the 63-mm (2½-in.) distance of separation to a position where they are 25 mm (1 in.) apart. Then examine the specimens for fracture or visible cracks.

NOTE X1.3—Previously two periods were specified, 96 h for natural-rubber compounds and 5 h for synthetic-rubber compounds. It was found that 5 h is adequate for either class of compounds within the intent of this test method.

X1.5 Results

X1.5.1 When two specimens are tested and neither one fractures nor shows cracks after being tested, the compound shall be considered as having passed the brittleness test. If both specimens crack, the compound shall be considered to have failed.

X1.5.2 If only one specimen fractures or cracks, the result is inconclusive and two additional specimens shall be tested. If either one of these cracks, the compound shall then be considered to have failed.

X1.6 Report

X1.6.1 Report the following:

X1.6.1.1 The results of the test expressed as "passed" or "failed,"

X1.6.1.2 The temperature of the cold chamber,

X1.6.1.3 The duration of the exposure period,

X1.6.1.4 Identification of the material tested including description of any special treatment prior to test, and

X1.6.1.5 Data of manufacture of the material, if known, and date of test.

---

[4] This test method was originally issued in 1943 under the designation D 736 which was discontinued in April 1967.

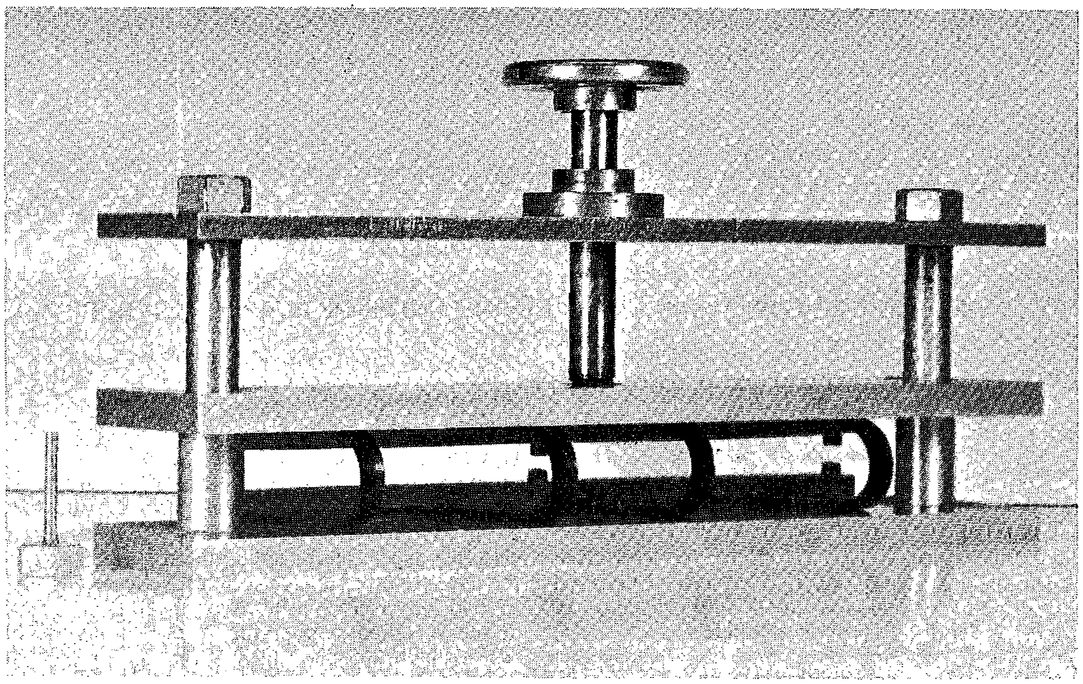


FIG. X1.1 Flexing Fixture for Low-Temperature Brittleness Test

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM C516: Standard Specification for Vermiculite Loose Fill Thermal Insulation

CFR Section(s): 24 CFR 200, Subpart S

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: C 516 – 80 (Reapproved 1996)$^{\epsilon 1}$

Standard Specification for Vermiculite Loose Fill Thermal Insulation[1]

This standard is issued under the fixed designation C 516; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

$^{\epsilon 1}$ NOTE—Keywords were added editorially in December 1996.

1. Scope

1.1 This specification covers the composition and physical properties of expanded or exfoliated vermiculite loose fill insulation. The specification also includes the testing procedures by which the acceptability of the material may be determined. These testing procedures deal primarily with material performance in the temperature range associated with the thermal envelope of buildings; however, the commercially usable temperature range for this insulation is from −459 to 1400°F (−272 to +760°C). For specialized applications, refer to manufacturer's instructions.

1.2 The specification also covers the composition and properties of vermiculite that has been surface-treated to produce water repellency for installations where liquid moisture may be a factor.

1.3 When the installation or use of thermal insulation materials, accessories, and systems may pose safety or health problems, the manufacturer shall provide the user with appropriate current information regarding any known problems associated with the recommended use of the company's products, and shall also recommend protective measures to be employed in their safe utilization.

1.4 The following safety hazards caveat pertains only to the test methods portion, Section 9, of this specification: *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 12.

2. Referenced Documents

2.1 *ASTM Standards:*

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates[2]

C 168 Terminology Relating to Thermal Insulating Materials[3]

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus[3]

C 390 Criteria for Sampling and Acceptance of Preformed Thermal Insulation Lots[3]

C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[3]

C 520 Test Methods for Density of Granular Loose Fill Insulations[3]

E 84 Test Method for Surface Burning Characteristics of Building Materials[4]

E 136 Test Method for Behavior of Materials in a Vertical Tube Furnace at 750°C[4]

3. Terminology

3.1 The terms used in this specification are defined in Terminology C 168.

4. Classification

4.1 Vermiculite insulation shall be specified by two type designations and five classes, as follows:

4.1.1 *Vermiculite Loose Fill Insulation—Type I*—The product that results from the expanding or exfoliating of natural vermiculite ore by grading and heating to meet the requirements of this specification.

4.1.2 *Vermiculite Loose Fill Insulation—Type II*—Expanded vermiculite that has been surface-treated to produce water repellency and limit absorption of moisture from both liquid and vapor phase.

4.1.3 Vermiculite loose fill insulation has five grade designations established by range of particle size distribution as shown in Table 1.

5. Ordering Information

5.1 All purchase orders should designate both type and grade of insulation desired. If type designation is omitted, Type I will be furnished. The type and grade classifications in this specification differ from the classifications in earlier issues. Purchasers referencing this specification should include the date of issue.

6. Materials and Manufacture

6.1 Vermiculite is a micaceous mineral which is mined and processed to produce a high-purity concentrate. The

---

[1] This specification is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.23 on Blanket and Loose Fill Insulation.

Current edition approved Oct. 31, 1980. Published March 1981. Originally published as C 516 – 63 T. Last previous edition C 516 – 75.

[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.06.

[4] *Annual Book of ASTM Standards*, Vol 04.07.

TABLE 1 Density Specifications

Grade Designation	Bulk Density, lb/ft^3 (kg/m^3)	
	min	max
0—Premium	3.0 (48)	5.0 (80)
1—Large	3.7 (59)	5.5 (88)
2—Medium	4.0 (64)	6.0 (96)
3—Fine	4.5 (72)	7.0 (112)
4—Super Fine	5.5 (88)	8.0 (128)

TABLE 2 Thermal Resistance

Grade Designation	Thermal Resistance[A] for 1 in. (25.4 mm) Thickness °F·h·ft^2/Btu (K·m^2/W) Mean Temperature, °F (°C)		
	−119 (−84)	40 (4)	75 (24)
0—Premium	...	...	2.3 (0.41)
1—Large	...	...	2.3 (0.41)
2—Medium	...	...	2.3 (0.41)
3—Fine	...	...	2.3 (0.41)
4—Super Fine	3.4 (0.58)	2.5 (0.43)	2.3 (0.41)

[A] The thermal values in this table are given at 1 in. (25.4 mm) of thickness with a gradient of 50°F (28°C). Test data that cover the thickness range from 1 to 6 in. show that thermal resistance per inch of thickness is essentially linear with respect to thickness. Test values do not deviate on the low side from the value in the table by more than 5 %.

concentrate, in the form of flakes of varying size and thickness weighing 55 lb/ft^3 (880 kg/m^3), is expanded in high-temperature furnaces to densities in the range from 3.0 to 8.0 lb/ft^3 (148 to 128 kg/m^3). As a naturally occurring mineral, it is classifiable as an elementary building material. It is noncombustible as determined by Test Method E 136. Material must pass combustion test criteria of Test Method E 136.

7. Physical Requirements

7.1 The physical requirements listed in this section are defined as Inspection Requirements (see Criteria C 390, 5.1.2; see also Terminology C 168). The insulation shall conform to the following requirements:

	Type I	Type II
Bulk density, lb/ft^3 (kg/m^3)	See Table 1	
Grading (particle size)	See Table 3	
Water properties, max g wicked in 5 min	N.A.	3

7.2 The physical properties listed in this section of the specification are defined as Qualification Requirements (see Criteria C 390, 5.1.1). The insulation shall conform to the following requirements:

Thermal resistance, °F·h·ft^2/Btu (K·m^2/W)	See Table 2	
Moisture absorption, max, % by weight/ 14 days	3.5	3.5
Combustibility	No flaming, glowing, or smoking	
Surface-burning characteristics (Test Method E 84):		
Flame spread, max	0	0
Smoke developed, max	0	0
Water properties, min, mL of water repelled	N.A.	175

8. Sampling

8.1 For purposes of standard tests, sampling shall be in accordance with Criteria C 390.

9. Test Methods

9.1 The physical properties, as enumerated in Section 7, shall be determined in accordance with the following methods:

9.1.1 *Bulk Density*—Test Methods C 520, Method A.

9.1.2 *Grading*—Test Method C 136, except that when a mechanical sieving device is used, the sieving time shall be 5 min and the test specimen shall be 50 g of material.

9.1.3 *Thermal Resistance*—Tests for thermal resistance may be made in accordance with Test Methods C 177 or C 518. Test at the design density. The thermal resistance of the various types shall not be lower than the values listed in Table 2, except that the average thermal resistance of any four specimens may fall up to 5 % below the value in the table. Determine the thermal resistance (*R*-value) at mean temperature of 75°F (24°C) and 40°F (4°C), at design density and in accordance with the current editions of Test Methods C 177 or C 518. Report the direction of heat flow. Thermal resistance at other mean temperatures may be determined if required.

9.1.4 *Moisture Absorption*—The test specimen shall be a sample of approximately 50 g. Loose fill the sample and level into a sample holder 9 by 9 by 5 in. (228 by 228 by 127 mm) deep.

9.1.4.1 Condition with minimum air movement across the sample surface. Condition at 50 ± 2 % relative humidity and 120°F (48.9°C) to constant weight and record. State the density of the sample conditioned to constant weight in the report of results.

9.1.4.2 Increase the relative humidity to 90 ± 2 %. Condition to constant weight by check-weighing at 24-h intervals. Determine the moisture pickup as a percent of conditioned weight.

9.1.4.3 Record percent absorption at 14 days.

9.1.4.4 *Precision and Bias*—This test establishes a typical property of vermiculite. It is an inherent characteristic. It is only intended to indicate absorption under high humidity conditions which are known to be characteristic of its usual insulation end use.

9.1.5 *Water Repellency (Type II Only)*—Determine the water repellency of Type II vermiculite insulation in accordance with the following procedure:

9.1.5.1 *Apparatus*—(*a*) Rigid plastic tube 50 mm inside diameter by 300 mm long with a 150-μm (100-mesh) screen

TABLE 3 Grading Cumulative Percent Retained

Grade Designation	U. S. Sieve No.						
	3/8 in. (9.5 mm)	4 (4.75 mm)	8 (2.36 mm)	16 (1.18 mm)	30 (600 μm)	50 (300 μm)	100 (150 μm)
0—Premium	30–80		80–100				
1—Large	0–10			90–100			
2—Medium		0–10	45–90		95–100		
3—Fine			0–10		90–100		
4—Super Fine				0–5		60–98	90–100

covering firmly fastened or adhered to the bottom. The tube shall be marked at 400 mL from the screen-covered end. (*b*) No. 15 rubber stopper. (*c*) 250-mL graduated cylinder. (*d*) 500-mL beaker.

9.1.5.2 *Sample Preparation*—Spoon a representative sample into the test cylinder to a level slightly above the 400-mL mark. Compact it by dropping the tube from a height of approximately 75 mm on a large rubber stopper (No. 15 recommended) for a total of ten drops. As the sample compacts to a level below the 400-mL mark, add additional material so that after the tenth drop, the level of the sample is within 3 mL of the 400-mL mark.

9.1.5.3 *Procedure*—With the tube supported in a vertical position and a beaker positioned under the tube, rapidly pour 250 mL of cold tap water onto the vermiculite. Take care while pouring, that the stream hits the middle of the surface of the bed of vermiculite and does not merely slide down the side of the test cylinder. Allow the water to drain through the bed of vermiculite for exactly 3 min. Tilt the tube at approximately 45° to drain water collected on the screen. Tilt it only momentarily for this purpose. Measure the collected water in the 250-mL graduate.

9.1.5.4 *Calculation*—Report the amount of collected water as "millilitres repelled." Report results as the mean of three independent tests.

9.1.5.5 *Precision and Bias*—The purpose of this test is to confirm that the intended degree of surface treatment has been accomplished to achieve water repellency. The specified characteristic is a minimum (no range or maximum). Quantitative values for conforming products exceeding the limits have no commercial significance.

9.1.6 *Wickability—Vermiculite Insulation (Type II Only):*

9.1.6.1 *Apparatus*—(*a*) Rigid plastic tube 50 mm in inside diameter by 300 mm long with a 150-µm (100-mesh) screen covering firmly fastened or adhered to the bottom. (*b*) 50-mm long compaction plug, fitting snugly inside the sample holder to give standard compaction to the sample. (*c*) Tubing rack capable of supporting the sample tube. (*d*) Water immersion tray.

9.1.6.2 *Sample Preparation*—Spoon a representative sample into the test cylinder to a level slightly below the top of the cylinder. Compact it with the plug until a moderate resistance pressure is felt. In no event shall compaction be more than 24 mm.

9.1.6.3 *Procedure*—Place the sample tube, screen end down, on a rack in the water immersion tray. The water level in the tray shall be 50 mm above the bottom of the tube. Allow the sample to remain in the position and allow to wick water for 5 min. Remove the tube from the tray onto a tared 75-mm watch glass so that all water that is allowed to drain, is caught by the watch glass and is included in the weight of water picked up by the sample. Determine the weight of the water picked up by the sample in grams.

9.1.6.4 *Calculation*—Report the amount of water picked up by the vermiculite sample through this wicking action as grams. Report results as a mean of three independent tests.

9.1.6.5 *Precision and Bias*—See 9.1.5.5.

10. Inspection

10.1 Inspection of the materials shall be made as agreed upon between the purchaser and the manufacturer as part of the purchase contract.

11. Packaging and Marking

11.1 *Packaging*—Unless otherwise specified, the insulation shall be packed in the manufacturer's standard commercial container. The insulation shall be packed to ensure carrier acceptance and safe delivery at destination in containers complying with the rules and regulations applicable to the mode of transportation.

11.2 *Marking*—Shipping containers shall be marked with the name of the insulation, minimum weight of container, and the name of the manufacturer. For residential insulation, a chart shall also be affixed or printed on the container listing the minimum thickness, maximum net coverage area, and minimum weight per square foot at R values of 11, 19, and 22. In addition to this chart, the following statements must be added: 1. "R means resistance to heat flow. The higher the R-value, the greater the insulation power. 2. To get the marked R-value, it is essential that this insulation be installed properly. If you do it yourself, follow the instructions carefully."

12. Health and Safety Precautions

12.1 *Preinstallation*—The insulation material should be handled and stored in accordance with manufacturer's instructions. It should be kept dry and free of extraneous materials. It is noncombustible.

12.2 *Installation:*

12.2.1 The insulating material should be poured into the spaces and cavities to be insulated in a manner that minimizes free-fall and impact. This will minimize crushing and breakdown of insulation particles and the unnecessary formation of dust.

12.2.2 Pourable loose fill insulation, which is commonly installed in confined, poorly ventilated attic spaces, may generate a buildup of airborne dust. Inhalation of dust can be injurious to health. Refer to manufacturer's instructions regarding recommended installation practices.

13. Keywords

13.1 loose fill; thermal insulation; thermal resistance; vermiculite; water repellency

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM C518: Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus

CFR Section(s): 46 CFR 160.174-17(f)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
The Executive Director
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: C 518 – 91

Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[1]

This standard is issued under the fixed designation C 518; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.

INTRODUCTION

This test method presents a comprehensive assemblage of information and requirements on the complex problem of the measurement of the thermal transmission properties of insulations using a heat flow meter apparatus. For convenience, it is organized in four major parts:

General Considerations (Sections 1 through 6)
Apparatus and Calibration (Sections 7 through 9)
Test Procedures and Report (Sections 10 through 14)
Annexes (A1 and A2)

This test method shall be used in conjunction with Practice C 1045.

Many advances have been made in thermal insulation technology, both in measurement techniques and in improved understanding of the principles of heat flow through such materials. These advances have prompted revisions in the conceptual approaches to the measurement of the thermal transmission properties, see References **(1)**, **(2)**, **(3)**, and **(4)**.[2] All users of this test method should be aware of these concepts.

1. Scope

1.1 This test method covers the measurement of steady state thermal transmission through flat slab specimens using a heat flow meter apparatus.

1.2 This is a comparative (or secondary) method of measurement since specimens of known thermal transmission properties must be used to calibrate the apparatus. Properties of the calibration specimens must be traceable to an absolute measurement method, and should be obtained from or traceable to a recognized national standards laboratory.

1.3 The test method is applicable to the measurement of thermal transmission through a wide range of specimen properties and environmental conditions. The method has been used at ambient conditions of 10 to 40°C with thicknesses up to approximately 250 mm, and with plate temperatures from −195°C to 540°C at 25 mm thickness (Refs. **(5, 6)**).

1.4 This test method may be used to characterize material properties which may or may not be representative of actual conditions of use. Other test methods such as Test Methods C 236 or C 976 should be used if needed.

1.5 To meet the requirements of this test method the thermal resistance of the sample must be greater than 0.10 $K \cdot m^2/W$ in all directions.

1.6 It is not practical in a test method of this type to try to establish details of construction and procedures to cover all contingencies that might offer difficulties to a person without pertinent technical knowledge. Thus users of this test method shall have sufficient knowledge to satisfactorily fulfill their needs. For example, knowledge of heat transfer principles, low level electrical measurements, and general test procedures is required.

1.7 Standardization of this test method is not intended to restrict in any way the future development of improved or new methods or procedures by research workers.

1.8 Since the design of a heat meter apparatus is not a simple matter, a procedure for qualifying an apparatus is given in 7.8.

1.9 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 167 Test Methods for Thickness and Density of Blanket or Batt Thermal Insulations[3]

---

[1] This test method is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurement.

Current edition approved July 15, 1991. Published September 1991. Originally published as C 518 – 63T. Last previous edition C 518 – 85.

[2] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[3] *Annual Book of ASTM Standards*, Vol 04.06.

C 168 Terminology Relating to Thermal Insulating Materials[3]
C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties By Means of the Guarded-Hot-Plate Apparatus[3]
C 236 Test Method for Steady-State Thermal Performance of Building Assemblies by Means of a Guarded Hot Box[3]
C 519 Test Method for Density of Fibrous Loose-Fill Building Insulations[3]
C 687 Practice for Determination of the Thermal Resistance of Loose-Fill Building Insulation[3]
C 976 Test Method for Thermal Performance of Building Assemblies by Means of a Calibrated Hot Box[3]
C 1045 Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements[3]
C 1132 Practice for Calibration of the Heat Flow Meter Apparatus[3]
E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[4]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

3. Terminology

3.1 *Definitions*—The terms and symbols are as defined in Definitions C 168 with exceptions included as appropriate.

3.2 *Symbols and Units*—The symbols used in this test method have the following significance:

λ = thermal conductivity, W/m · K,
C = thermal conductance, W/m^2 · K,
R = thermal resistance, m^2 · K/W,
q = heat flux, W/m^2,
Q = heat flow, W,
A = area measured on a selected isothermal surface, m^2,
L = thickness of specimen measured along a path normal to isothermal surfaces, m,
T_m = mean temperature, K,
ΔT = temperature difference, K,
ρ = density of the material as tested, kg/m^3,
S = sensitivity of heat flow transducer (W/m^2)/V,
E = heat flow transducer output, V,
T_h = temperature of hot plate surface, K, and
T_c = temperature of cold plate surface, K.

4. Summary of Test Method

4.1 The heat flow meter apparatus establishes steady state unidirectional heat flux through a test specimen between two parallel plates at constant but different temperatures. By appropriate calibration of the heat flux transducer(s) with calibration standards and by measurement of the plate temperatures and plate separation, Fourier's law of heat conduction is used to calculate thermal conductivity, thermal resistance, or resistivity.

4.2 The accurate use of the test method is limited by the capability of the apparatus to reproduce unidirectional constant heat flux density in the specimens, and by the precision in the measurement of temperature, thickness, EMF produced by heat flux transducer, etc.

5. Significance and Use

5.1 This test method provides a rapid means of determining thermal properties with high precision and as such is useful in research and quality control work.

5.2 The thermal transmission properties of specimens of a given material or product may: (*1*) vary due to variability of the composition of the material; (*2*) be affected by moisture or other conditions; (*3*) change with time; (*4*) change with mean temperature and temperature difference, and (*5*) depend upon the prior thermal history. It must be recognized, therefore, that the selection of typical values of thermal transmission properties representative of a material in a particular application should be based on a consideration of these factors and will not necessarily apply without modification to all service conditions. As an example, this test method provides that the thermal properties shall be obtained on specimens that do not contain any free moisture although in service such conditions may not be realized. Even more basic is the dependence of the thermal properties on variables such as mean temperature and temperature difference. These dependencies should be measured or the test made at conditions typical of use.

5.3 Special care must be taken in the measurement procedure for specimens exhibiting appreciable inhomogeneities, anisotropies, rigidity, or especially high or low resistance to heat flow (see Practice C 1045). Special considerations are necessary when the measurements are conducted at either high or low temperatures, in a vacuum or in hazardous gases.

5.4 The determination of the accuracy of the method for any given test is a function of the apparatus design, of the related instrumentation and of the type of specimens under test (see Section 9), but this method is capable of determining thermal transmission properties within ±2 % of those determined by Test Method C 177 when the ambient temperature is near the mean temperature of the test (T (ambient) = T (mean) ±1°C), and in the range of 10 to 40°C.

5.4.1 Where certification of measurement by this test method is required, the apparatus shall have the capabilities required in 7.8 and one of the following procedures must be followed:

5.4.1.1 The testing laboratory apparatus shall be calibrated within 24 h before or after the certification test using calibration standards that have been issued or whose values have been established by a recognized National Standards Laboratory not more than five years prior to the certification date. The average of two calibrations shall be used as the calibration factor and the specimen(s) certified with this average value. When the change in calibration factor is greater than one percent, the standard specimen shall be retested and a new average calculated. If the change in calibration factor is still greater than 1 % the apparatus must be calibrated using the procedure in Section 8.

5.4.1.2 Where both the short and long term stabilities of the apparatus have been proven to be better than 1 % of the reading, see 8.4, the apparatus may be calibrated at less frequent intervals, not exceeding 30 days. The specimens so tested cannot be certified until after the calibration following the test and then only if the change in calibration factor from

---

[4] *Annual Book of ASTM Standards*, Vol 14.03.
[5] *Annual Book of ASTM Standards*, Vol 14.02.

the previous calibration test is less than 1 %. When the change in calibration is greater than one percent, test results from this interval shall be considered void and the tests repeated in accordance with 5.4.1.1.

5.4.2 The precision (repeatability) of measurements made by the heat flow meter apparatus calibrated as in 5.4.1 are normally much better than ±1 % of the mean value. This precision is required to identify changes in calibration and is desirable in quality control applications.

6. Limitations to Test Method

6.1 *Limitations Due to Apparatus:*

6.1.1 The apparatus shall not be used at temperatures, thicknesses, or resistances, other than those within the range of the calibration, unless it can be shown that there is no difference in accuracy.

6.1.2 The apparatus must be capable of maintaining at least a 10°C temperature difference across the specimen for the duration of the test, unless a smaller ΔT is a requirement of a particular test.

6.2 *Limitations Due to the Specimens:*

6.2.1 The specimens under test may also limit the use of the test method and these limitations are outlined in Practice C 1045.

7. Apparatus

7.1 The construction details given in this section should be understood by the user of this test method. While it is mandatory that these details be carefully followed when constructing an apparatus, it behooves the user to verify that the equipment was built as specified. Serious errors of measurement may result from this oversight.

7.2 *General:*

7.2.1 The general features of a heat flow meter apparatus with the specimen or the specimens installed are discussed in Annex A1. A heat flow meter apparatus consists of two isothermal plate assemblies, one or more heat flux transducers, equipment to measure and control temperature, thickness, and the output of the heat flux transducers and equipment to control the environmental conditions when needed. Each configuration will yield equivalent results if used within the limitations stated in this test method. There are distinct advantages for each method in practice and these are discussed in Annex A1.

7.2.2 The working surfaces of the plates and the heat flux transducer (that is, the surfaces making contact with the specimens) shall be painted or otherwise treated to have a total hemispherical emittance of greater than 0.84 at operating temperatures (Note 1).

NOTE 1—Hard anodizing of aluminum produces a surface with a total hemispherical emittance of approximately 0.85. Several paints are available which when applied as directed, produce a total hemispherical emittance of approximately 0.86.

7.3 *Plate Assemblies, Hot and Cold*—The two plate assemblies provide isothermal surfaces in contact with either side of the test specimen. The assemblies consist of heat source or heat sink, a high conductivity surface, means to measure surface temperature, and means of support. A heat flux transducer may be attached to one, both or neither plate assembly, depending upon the design. In all cases the area defined by the dimensions of the heat flux transducer is called the metering area and the remainder of the plate is the guard area.

7.3.1 The portion of each plate assembly in contact with the heat flux transducer, or if a heat flux transducer is not mounted on the plate assembly, the portion in contact with the sample, shall consist of a high conductivity material, usually metal, known as the isothermal plate. The isothermal plate shall be supported so as to minimize temperature gradients across its surface. For a 2 % total uncertainty, deviations from isothermal conditions over the plate surface of no more than ±0.5 K shall be allowed.

7.3.2 A means shall be provided to maintain the temperature of the isothermal plates at the desired level. Examples are fluid baths, electrical heaters and combinations of these (see Refs (**7, 8, 9**)).

7.3.3 If a heat flux transducer is mounted at the midplane of the specimen then means shall be provided to determine the average temperature of the transducer in order to apply temperature corrections to the calibration, except when test temperatures are equal to those used in calibration, in which case no correction is required.

7.3.4 Means shall be provided to measure the temperature of the surfaces of the heat flux transducer or plates in contact with the specimen.

7.3.5 The surface plates shall be sufficiently rigid to maintain flatness and parallelism to within one-tenth the accuracy required of the final result. For measurements on non-rigid materials and an allowable total uncertainty of ±2 %, a flatness and parallelism of 0.2 % of the maximum linear dimension of the heating unit is thus required (Note 2). One recommended way to check this is to generate a L-map over the metering area. This is discussed fully in Ref (**10**).

NOTE 2—The planeness of the surface can be checked with a straightedge, of a length greater than the width or diameter of the unit, held against the surface and viewed with a light behind the straightedge. Departures as small as 25 µm are readily visible, and larger departures can be measured using shimstock or thin paper.

7.3.5.1 The parallelism of the plates must be maintained for several reasons. In most cases it is the plate separation which is measured in order to determine specimen thickness. Furthermore the plate parallelism is important in maintaining consistent surface contact with specimens in repeat testing (such as calibration), and is required to maintain a uniform temperature difference across the specimen(s).

7.3.6 The rigidity, flatness, and parallelism of the plates may impede the testing of rigid specimens where it is not possible to obtain good surface contact. In such cases the use of a thin sheet of suitable homogeneous material may be interposed between the specimen and the plate surfaces. This thin sheet should have a low thermal resistance relative to the specimen. The resistance of the thin sheet should be determined using a Test Method C 177 apparatus. The resistance of the composite sandwich (sheet-rigid specimen-sheet) is then determined and the value of the sheet resistance (determined in a separate test) subtracted from the total resistance measured.

7.4 *Temperature Measuring and Control Systems:*

7.4.1 Any proven device possessing adequate accuracy may be used for measurement and control of the temperatures in the apparatus.

7.4.2 Surface temperatures are often measured by permanently mounting temperature sensors, such as thermocouples, RTD's or thermistors, in grooves or flush with the working surfaces on both plates.

7.4.3 Many existing systems utilize a minimum of two temperature sensors to determine the average temperature of each isothermal surface. However, to determine the actual surface temperature variations under typical operating conditions, the temperature must be sampled at considerably more than two points. This is especially true when the specimen surface temperature itself must be measured or when the specimen is highly inhomogeneous. Therefore, no strict specification is given as to the number of temperature sensors that must be used for each surface. However, the user must report the uncertainty of the temperature measurement, including the component, due to temperature nonuniformity across the surface. This determination is probably best done with a multijunction thermocouple in ad-hoc experiments and not part of every measurement. (See Ref **(10)**.) In some cases where temperature mapping of the plate surfaces has indicated high uniformity under all conditions of use, one thermal sensor per surface has been satisfactorily used.

7.4.3.1 Special precautions should be taken to ensure that the temperature sensors are thermally anchored to the surface to be measured and that the temperature gradients along the wires leading to the sensors are minimized. If sensors on opposing surfaces are connected differentially, they must be electrically insulated from the plates with a resistance of 1MΩ or greater **(11, 12)**.

7.4.3.2 Thermocouples mounted in the surfaces of the plates should normally be made of wire not larger than 0.51 mm in diameter (No. 24 B & S gage). Thermocouples placed against or set into the surfaces of the specimens should be made of wire not larger than 0.25 mm in diameter (No. 30 B & S gage). For information concerning sensitivity and accuracy of thermocouples in the cryogenic temperature range, and installation, see Refs **(6)** and **(13)**.

7.4.3.3 Temperature sensors should be calibrated to an accuracy equivalent to that for thermocouples conforming to Tables E 230. The temperature measuring system shall have an equivalent accuracy. The precision of the temperature measuring system may need to be better than this to detect the effect of drift on the results as discussed in 7.9. Tables E 230 lists both "standard" and "special" limits on the accuracy of thermocouples. The accuracy required by a heat flow meter apparatus can best be determined by carrying out an error analysis (see Section 9), and then calibrating the temperature sensors to the degree required.

7.5 *Heat Flux Transducer*—The heat flux transducer is a device including both the metering and guard areas which measures heat flux by sensing the temperature difference across a core of appropriate thermal resistance and thermal stability. The metering area usually consists of a number of differentially connected temperature sensors placed on each face of the core and surface sheets to protect the assembly. The guard area is usually constructed of the same material as the core. A properly designed transducer will have a sensitivity that is essentially independent of the specimens' thermal properties **(14, 15)**.

7.5.1 *Types of Heat Flux Transducers*—The type of heat flux transducer described in this test method is called a gradient type. This type consists of a slab of material, the "core," across which the temperature gradient is measured, normally with a thermopile.

7.5.2 *Temperature Difference Detector*—The temperature difference across the core material shall be measured with a sensitive stable temperature detector. The main transducer surfaces are assumed to be isothermal, so that heat flow will be normal to them. Precautions must be taken to limit the effect of heat flow through the leads on the output of the temperature difference detector. Often the heat flux transducer is also instrumented to measure one of the surface temperatures of the specimen(s).

7.5.3 *Surface Sheets*—To prevent damage to the temperature difference detector that will affect its calibration, both surfaces shall be covered with a layer of material as thin as is compatible with protection from thermal shunting of the temperature difference detector wires. The working surfaces of the heat flux transducer shall be smoothly finished to conform to the desired geometric shape to within the limits of 7.3.

7.6 *Plate Separation, Specimen Thickness*—A means shall be provided to determine the average separation between the heating and cooling plate surfaces during operation. Rigid specimens generally act as the spacers themselves and plate separation is determined by their thickness at operating temperature. In this case a small constant force is generally applied to hold the plates against the specimen. It is unlikely that a pressure greater than 2.5 kPa (approximately 50 lbf/ft^2) will be required. For easily compressible specimens, small stops interposed between the corners of the hot and cold plates, or some other positive means shall be used to limit the compression of the specimens, (Note 3). Provision must be made for checking the linearity of any thickness measuring system.

NOTE 3—Because of the changes of specimen thickness possible as a result of temperature or compression by the plates, it is recommended that specimen thickness be measured in the apparatus, at the existing test temperature and compression conditions whenever possible.

7.7 *Edge Insulation*—Heat loss from the outer edges of the heat flow meter apparatus and specimens shall be restricted by edge insulation or by governing the surrounding air temperature or by both methods. The three different configurations differ in their sensitivity to edge heat losses as is discussed in Annex A1 **(2, 4, 16)**.

7.7.1 For all three configurations, the sensitivity to edge heat losses is strongly related to the sensitivity of the transducer to temperature differences along its main surfaces and therefore only experimental checks while changing environmental conditions can confirm, for each operating condition, the magnitude of the effect of edge heat losses on measured heat flux. This error should be smaller than 0.5 %. An approximate guideline is to maintain power lost through the edges below 5 % of the power flowing through the specimen.

7.8 *Measuring System Requirements for Certification*—When the apparatus is used for certification testing, the measuring system shall have the following capabilities:

7.8.1 The accuracy of the measurement of the temperature difference across the specimens shall be within ±0.5 %. The accuracy of the measurement of the output from the heat flux transducer shall be within ±0.6 %.

7.8.2 A voltage sensitivity of better than 0.2 % of the minimum output (from the transducer) to be measured.

7.8.3 Sufficient linearity so that it contributes less than 0.2 % error at all outputs.

7.8.4 Sufficient input impedance so that it contributes less than 0.1 % error for all readings. One megohm has been found adequate for many apparatuses.

7.8.5 Sufficient stability so that it contributes less than 0.2 % error during the period between calibrations, or 30 days, whichever is greater.

7.8.6 Adequate noise immunity so that less than 0.2 % rms noise occurs in the readings.

7.8.7 Where direct readout equipment is used, adequate provision shall be made for calibration of the electronic circuitry, independent of the remainder of the apparatus, and shall contribute no more than 0.2 % error, for each variable.

7.9 *Proven Performance*—Any heat flow meter apparatus that is new or has been modified must be tested for the following characteristics and corrections shall be made where a change of greater than one percent occurs in the transducer output due to the effects of 7.9.1 to 7.9 over the range of operation and are recommended for changes of 0.3 % over the range of operation.

7.9.1 *Zero Offset*—If there is any output from the transducer for zero heat flux, this may be due to:

7.9.1.1 Electrical contact resistance on a transducer with low output: improve connections to eliminate the problem as this type of output may be temperature dependent.

7.9.1.2 Also check grounding circuits because such a signal may be due to AC pickup in the leads from the transducer. If after checking 7.9.1.1 and 7.9.1.2 there is still a zero off-set, it may be possible to correct for this by assuring that the calibration curve of output versus heat flux is linear over the range of operating conditions.

7.9.2 Sensitivity to warm or cold plate temperature non-uniformity. Check for temperature non-uniformity under all operating conditions.

7.9.3 Drift in the transducer due to material aging or delamination. This can be used to determine the required calibration frequency.

7.9.4 Temperature coefficient of the transducer sensitivity, as this depends on the type of temperature detectors used in the temperature difference detector (thermocouple materials used in the thermopile) and the type of material used for the transducer core. If it is found that the sensitivity is temperature dependent, a curve of sensitivity versus temperature shall be developed and used to correct measurement data.

7.9.5 Heat flow down the leads. One possible way to check for this is to move one's hand across the surface of the transducer between the metering area to the point where the leads exit the plate assembly, while observing the transducer output. In a well designed plate or transducer assembly there should be no observable output from the transducer except in the metering area.

7.9.6 Effect of the thermal conductivity of the specimen on the sensitivity of the transducer. A "thermal shorting" effect between elements caused by low thermal resistance between the sensors of the thermopile or a funneling of heat through the sensors can change the sensitivity of the meter. This can best be tested by running specimens with known widely different thermal conductivities.

7.9.7 Effect of loading pressure on the transducer sensitivity, if the transducer core is flexible.

7.9.8 The test results obtained by this method can only be assured if the limitations of the apparatus are known. To establish the limitations, one must prove the performance by comparing the results with materials of similar thermal properties previously tested on a guarded hot plate apparatus as those to be evaluated.

7.9.8.1 A single point of reference may lead to serious errors. It is best to select a range of transfer standards having known thermal transmission properties which cover the range of values to be tested, in both resistance and thickness.

7.9.8.2 If the apparatus is to be used at thicknesses greater than that of the available reference materials, a series of calibration measurements must be performed to insure that the equipment does not introduce additional errors which may be due to lateral heat losses or gains brought about by insufficient guarding **(4, 17)**.

7.10 *Environmental Control*—In many applications, it is desirable to control the environment surrounding the test specimen to reduce edge heat losses and it is especially important if the mean test temperature must be below the ambient temperature, in order to avoid condensation on the cold plate.

7.10.1 A cabinet or enclosure surrounding the heat flow meter assembly to maintain the ambient temperature at the mean temperature of the specimen may also be used as a means to maintain the dew point temperature at least 5 K lower than the temperature of the cold plates, that will prevent condensation and moisture pickup by the specimen. This is required when conducting tests at mean temperatures substantially lower than the laboratory air temperature.

7.10.2 Any environmental control system employed in conjunction with a heat flow meter apparatus shall be capable of maintaining its set point condition within ±1°C in temperature, and ±2 % in humidity.

8. Calibration

8.1 The calibration of a heat flow meter apparatus is a very critical operation. Since lateral heat losses or gains of heat are not automatically controlled or eliminated, but only lessened by increasing the size of the guard area and edge insulation, there is no guarantee that the heat losses or gains are negligible under all testing conditions. To ensure that the equipment is performing properly with specimens of different thermal resistances, the apparatus must be calibrated with materials having similar characteristics as the materials to be evaluated.

8.2 See Practice C 1132.

9. Error Analysis

9.1 A complete error analysis is complex. However, it is mandated for compliance with this test method. The performance of this analysis involves consideration of:

9.1.1 Estimates of errors in each individual measurement procedure and propagation of these errors to the final result,

9.1.2 Measurements to determine apparatus sensitivity to intentional deviations from normal operations, and

9.1.3 Measurements on reference materials and participation in round-robin programs.

9.1.4 For a more complete discussion of this error analysis the reader is directed to Test Method C 177, and Ref **(10)**.

9.2 To illustrate a procedure of error analysis estimation, consider the operational definition of thermal conductivity:

$$\lambda = Q \cdot L/A \cdot \Delta T$$

The uncertainties in Q, A, L, and ΔT (δQ, δA, δL, and $\delta \Delta T$) can be used to form the uncertainty $S\lambda$ by the usual error propagation formula where the total uncertainty is calculated from the square root of the sums of the squares of the individual standard deviations.

$$S\lambda^2 = (\delta Q/Q)^2 + (\delta A/A)^2 + (\delta L/L)^2 + (\delta \Delta T/\Delta T)^2$$

This equation assumes that the errors in Q, A, etc., are random and independent of each other since the covariance terms are omitted. This is valid here if different instrumentation is used for measurements on each of the variables.

9.2.1 An alternative approach, which generally leads to a larger calculated uncertainty, is to sum the upper limits of the individual uncertainties **(10)**.

9.2.2 Care must be taken to properly evaluate all of the uncertainties in the variables Q, A, etc. For example, the obvious sources of error in Q are those causing extraneous transverse heat flow along leads and deviations from one dimensional heat flow. However, an often neglected but important quasi-heat leak is that caused by a temperature drift of the transducer itself, or in the heater, or both.

9.2.2.1 This can readily be estimated from the heat capacity of the transducer assembly and the drift detection limit of the measurement system. The error in ΔT, $\delta \Delta T$, can be caused by calibration errors and measurement errors but also by incorrect placement, incorrect thermal anchoring, and disturbances introduced by the thermocouple itself.

9.3 Numerous experiments can be performed to determine the sensitivity of the measurement results to deviations from normal operating conditions. This sensitivity combined with the probable deviation under normal operating conditions can be used to estimate the error from this source. As one example, the effect of imperfect guard balance control can be determined by purposely offsetting the guard (if this is possible) by a sufficient ΔT in both directions and measuring the differences in the measured output.

9.3.1 Care should be taken to not use such large offsets that nonlinear effects occur in the specimens. These results combined with the probable value of the offset during normal operation yield the error due to imperfect guard balance.

9.4 Finally, measurements should be performed on accepted reference materials, if available, to qualify the apparatus. Care should be taken here to ensure that the reference materials have characteristics similar to the specimens to be measured, and that the uncertainties of the standards themselves are known.

9.5 The total estimated imprecision can be listed in a table of errors such as shown in Table 1. This table is shown as an example only and does not represent any one particular heat flow meter apparatus since the errors will be specific to each apparatus.

10. Test Procedures

10.1 *Foreword on Testing Procedures*—The simplicity of this test procedure may lead one to overlook very important factors which may affect the results. To ensure accurate measurement, the operator must be fully instructed in the operation of the equipment. Furthermore, the equipment must be properly calibrated with reference materials having similar heat transfer characteristics. It is also necessary that the specimen be properly prepared for the evaluation.

10.2 *Sampling and Preparation of Specimens:*

10.2.1 *Test Specimens*—One or two-piece specimens may be used, depending on the configuration selected for the test. Where two pieces are used, they shall be selected from the same sample to be essentially identical in construction, thickness, and density. For loose fill materials, the method specified in the material specification or in Practice C 687 shall be used to produce a specimen or specimens of the desired density.

10.2.2 *Selection of Specimens*—The specimen or specimens shall be of such size as to cover the heat flux transducer, cold plate and warm plate surfaces and shall either be of the actual thickness to be applied, or of sufficient thickness to give a true average representation of the material to be tested.

10.3 *Specimen Conditioning*—Details of the specimen selection and conditioning are preferably specified in the material specification. Where such specifications are not given, the specimen preparation shall be conducted in accordance with the requirement that materials shall not be exposed to temperatures that will change the specimens in an irreversible manner. Typically the material specifications call for sample conditioning at 22°C (72°F) and 50 % R.H. for a

TABLE 1 Estimates of Random and Systematic Errors at Room Temperature

Variable	Absolute Variations		Percent Variations	
	Random	Systematic	Random	Systematic
Mean Temperature, T	0.02 K	0.2 K	0.007	0.07
ΔT	0.01 K	0.2 K	0.04	0.40
ΔL	0	0.1 mm	0	0.40
Transducer area	0	50 mm^2	0	0.10
Heat flux				
drift	1 mW	1 mW	0.2	0.2
lateral	1 mW	2 mW	0.2	0.4
calibration	0	2 mW	0	0.2
Temperature				
drift	0.05 K	0.05 K	0.01	0.01
calibration	0	0.1 K	0	0.4
λ	0.2 mW/m·K	0.3 mW/m·K	0.4	0.6

period of time until less than a 1 % weight change is observed.

10.4 *Measurements on Specimens:*

10.4.1 *Blanket and Batt Type Materials*—When specified, the test thickness of blankets and batt type materials shall be determined before testing in accordance with Test Methods C 167, provided that good contact is maintained between the specimen and the isothermal plates.

10.5 *Limitations on Specimen Thickness:*

10.5.1 *General*—The combined thickness of the specimen or specimens, the heat flux transducer and any damping material, which in total equals the distance between the cold and hot plates, must be restricted in order to limit the effect of edge losses on the measurement of heat flux. The edge losses are affected by the edge insulation and the ambient temperature, so the requirements on both the these parameters must be met as well.

10.5.2 *Maximum Spacing Between Hot and Cold Plates*—The maximum allowable distance between the hot and cold plates during the test, is related to the dimensions of the heat flux transducer, the heat metering area, the width of the non-metering plate, the construction of the heat meter apparatus, and the properties of the specimen. No suitable theoretical analysis is available to predict the maximum allowable thickness of specimens. It is possible to use the results of an analysis for a similarly sized guarded hot plate as a guide **(8, 18)**.

11. Procedure of Measurement

11.1 *Temperature Difference and Gradient*—For any test, make the temperature difference across the specimen not less than 10 K. For specimens that are expected to have a large thermal resistance, a larger temperature difference in the specimen is recommended. The actual temperature difference or gradient is best specified in the material specifications or by agreement of the parties concerned.

11.2 *Edge Insulation*—Wrap the edges of the specimens with the required thermal insulation to reduce edge heat losses to an acceptable level (see 7.7).

11.3 *Settling Time and Measurement Interval*—Check for thermal equilibrium by observing and if possible recording, the mean temperature and the emf output of the heat flux transducer, the mean temperature of the specimens, and the temperature drop across the specimen. Make observations at time intervals of at least 10 minutes unless experience has shown that a shorter time interval may be used until five successive observations yield values of thermal conductance which fall with one-half percent of their mean value. For high density specimens ($\rho > 40$ Kg/m^3) or for low conductance specimens ($C < 0.05$ W/K·m^2) the time between readings may have to be increased to 30 minutes or longer **(19)**. If the five readings show a unidirectionally increasing or decreasing trend the data is suspect and additional sets of readings shall be taken.

12. Calculation

12.1 *Density and Change in Mass*—When required, calculate the density of the dry specimen as tested, ρ, the loss of mass due to conditioning of the material, and the mass regain of the specimen during test.

12.1.1 *Density of Batt and Blanket Specimens*—It has been found that in characterizing the thermal performance of low density materials it is important to measure the mass of the specimens in contact with the heat flux transducer metering area. For these materials, the area of the specimen directly measured must be cut out and its mass determined after testing. This procedure is desirable for all specimens.

12.2 *Thermal Properties for One Specimen*—When only one specimen is used, calculate the thermal conductance of the specimens as follows:

$$C = S \cdot E/\Delta T$$

and where applicable, calculate the thermal conductivity, as follows:

$$\lambda = S \cdot E \cdot (L/\Delta T)$$

12.3 *Thermal Properties for Two Specimens*—When two specimens are used, calculate the total thermal conductance, C, as follows:

$$C = S \cdot E/(\Delta T_1 + \Delta T_2)$$

The λ factor, that is, the average thermal conductivity of the specimen is calculated as follows:

$$\lambda_{ave} = \frac{S \cdot E}{2} \frac{L_1 + L_2}{\Delta T_1 + \Delta T_2}$$

where the subscripts refer to the two specimens.

12.4 Other derived thermal transmission properties may be calculated but only under the provisions given in Practice C 1045.

12.5 *Thermal Properties for Two Transducers*—All pertinent specifications of 12.2 apply on this configuration, provided S·E will be replaced by $(S' \cdot E' + S'' \cdot E'')/2$, where the superscripts $'$ and $''$ refer to the first and second heat flux transducer, respectively.

13. Precision and Bias

13.1 The precision and bias of this test method cannot be stated quantitatively for all apparatus variations and test conditions. However, it is mandated that for compliance with this test method, any result must be accompanied with its uncertainty. Further details on doing this are given in Section 9.

13.2 It should be noted that the only statistically valid procedure to determine uncertainty is to force all sources of systematic bias to be exhibited as random errors and then uncertainty can be directly estimated from imprecision. However, this is impractical since it involves variation of all measurement variables (Global set) and cost constraints make this impossible. For example, this implies the repetition of an experiment with differently calibrated thermocouples, different sizes and configurations of the apparatus, different operators, different environment, differently calibrated instrumentation, etc. For any one given apparatus a careful error analysis as outlined in Section 9 will, in most cases, show up any major difficulties, which may need correcting in order to improve the measurement accuracy of the heat flow meter apparatus.

13.3 A series of three round robins was conducted between 1976 and 1983, as reported by Hust and Pelanne **(21)**, and employed low density fiberglass specimens from 2.54 to 10.2 cm thick with densities ranging from 10 to 33 kg/m^3. A total of twelve laboratories was involved in these studies. The imprecision, at the two standard deviation level, was found to vary from 1.92 % to 3.54 % between 2.54 cm and 10.2 cm.

13.4 Inter-laboratory testing carried out between nine laboratories under the National Voluntary Laboratory Accreditation Program is currently showing an imprecision of 2.12 % at the two standard deviation level based on testing of similar but not identical specimens.

13.5 A round robin, conducted in 1987, was reported by Adams and Hust with eleven participating laboratories testing a fiberglass blanket and several types of loose-fill insulations **(22)**. The blanket insulation had an imprecision of 3.7 % at the two standard deviation level. The loose-fill imprecision was found to be >10 % for different materials at the two standard deviation level. It has been suggested that the principal cause for the significant differences observed was the various sample preparation techniques used by the various laboratories.

14. Report

14.1 The report of the results of each test shall include the following information with all data to be reported in both SI and inch-pound units unless specified otherwise:

14.1.1 Name and any other pertinent identification of the material including a physical description.

14.1.2 Description of the specimen and its relationship to the sample, including a brief history of the specimen, if known.

14.1.3 Thickness of the specimen as received and as tested. In a two piece specimen configuration this is the thickness of one piece only.

14.1.4 Method and environment used for conditioning, if used.

14.1.5 Density of the conditioned specimen as tested, kg/m^3.

14.1.6 Mass loss of the specimen during conditioning and testing, in percentage of conditioned mass, if measured.

14.1.7 Mass regain of the specimen during test, in percentage of conditioned mass, if measured.

14.1.8 Average temperature gradient in the specimen during test as computed from the temperatures of the hot and cold surfaces, K/m.

14.1.9 Mean temperature of the test, K or °C.

14.1.10 Heat flux per unit area through the specimen, W/m^2.

14.1.11 Thermal conductance, W/m^2·K.

14.1.12 Orientation and position of the heat meter apparatus during test (vertical, horizontal, etc.) and whether the meter was against the hot or cold surface of the specimen and whether the edges of the specimen(s) were sealed or open to the ambient.

14.1.13 Duration of the measurement portion of the test, min or h.

14.1.14 For loose-fill materials report the sample preparation followed.

14.1.15 Date of the test, the date of the last heat meter calibration, and the type or types of materials used.

14.1.16 For certification testing only, the specimens used in calibration must be identified as to the type, thermal resistance, date of specimen certification, source of certification, expiration date of calibration and the certification test number.

14.1.17 Statement of Compliance or, where circumstances or requirements preclude complete compliance with the procedures of the test, agreed exceptions. A suggested wording is: "This test conformed with all requirements of ASTM C 518 – ____ with the exception of (a complete list of exceptions follows)."

14.1.18 For direct reading apparatus, the results of the calibration of electronic circuitry and equipment, or a statement of compliance including date, and a statement of compliance on linearity requirements.

14.1.19 Estimated or calculated uncertainty in reported values.

15. Keywords

15.1 thermal insulation; thermal resistance; thermal conductivity; test method; heat flux; instrument verification; test procedures; error analysis; heat flow meter apparatus

ANNEXES

(Mandatory Information)

TABLE A1.1 Limits of Error for Thermocouples

NOTE 1—Limits of error in this table apply to new thermocouple wire, normally in the size range 0.25 to 3 mm in diameter (No. 30 to No. 8 Awg) and used to temperatures not exceeding the recommended limits of Table 2. If used at higher temperatures these limits of error may not apply.

NOTE 2—Limits of error apply to new wire as delivered to the user *and do not allow for calibration drift during use*. The magnitude of such changes depends on such factors as wire size, temperature, time of exposure, and environment.

NOTE 2—NOTE 3—Where limits of error are given in percent, the percentage applies to the temperature being measured when expressed in degrees Celsius. To determine the limit of error in degrees Fahrenheit multiply the limit of error in degrees Celsius by 9/5.

Thermo-couple Type	Temperature Range		Limits of Error-Reference Junction 0°C (32°F)			
			Standard		Special	
	°C	°F	°C (whichever is greater)	°F	°C (whichever is greater	°F
T	0 to 350	32 to 700	±1 or ±0.75 %	Note 3	±0.5 or 0.4 %	Note 3
J	0 to 750	32 to 1400	±2.2 or ±0.75 %		±1.1 or 0.4 %	
E	0 to 900	32 to 1600	±1.7 or ±0.5 %		±1 or ±0.4 %	
K	0 to 1250	32 to 2300	±2.2 or ±0.75 %		±1.1 or ±0.4 %	
R or S	0 to 1450	32 to 2700	±1.5 or ±0.25 %		±0.6 or ±0.1 %	
B	870 to 1700	1600 to 3100	±0.5 %		—	
T[A]	−200 to 0	−328 to 32	±1 or ±1.5 %		[B]	
E[A]	−200 to 0	−328 to 32	±1.7 or ±1 %		[B]	
K[A]	−200 to 0	−328 to 32	±2.2 or ±2 %		[B]	

[A] Thermocouples and thermocouple materials are normally supplied to meet the limits of error specified in the table for temperatures above 0°C. The same materials, however, may not fall within the sub-zero limits of error given in the second section of the table. If materials are required to meet the sub-zero limits, the purchase order must so state. Selection of materials usually will be required.

[B] Little information is available to justify establishing special limits of error for sub-zero temperatures. Limited experience suggests the following limits for Types E and T thermocouples:

Type E −200 to 0°C ±1°C or ±0.5 % (whichever is greater)
Type T −200 to 0°C ±0.5°C or ±0.8 % (whichever is greater)
These limits are given only as a guide for discussion between purchaser and supplier.

Due to the characteristics of the materials, sub-zero limits of error for Type J thermocouples and special sub-zero limits for Type K thermocouples are not listed.

A.2 Response of Heat Flux Transducers

A2.1 *Configurations*—The general features of a heat flow meter apparatus with the specimen or the specimens installed are shown in Fig. A2.1. A heat flow meter apparatus consists of two isothermal plate assemblies, one or more heat flux transducers, equipment to measure and control temperature, thickness and the output of the heat flux transducers and equipment to control the environmental conditions when needed. Configuration a is called single transducer, one face; the heat flux transducer may be placed against either plate. The Configuration b is called two transducer, both faces. The Configuration c is called single transducer, mid-plane; in this case, the specimens should be halves of the same specimen **(20)**. Each configuration will yield equivalent results if used within the limitations stated in this method. For a particular heat flux transducer, the test configuration that has the fastest response, that is shortest settling time, is best determined by experiment. Four specimens of different materials, such as urethane, ceramic fiber board, a rubber, and a high conductivity, low-thermal capacity material, each of which is conditioned to the mean test-temperature, should be tested in each configuration. A study of these will allow either the selection of the proper configuration for each type of material, or the selection of a reasonable configuration for all types.

A2.1.1 In order to predict settling times for all types of specimens, each of the above specimens must be retested after being conditioned to temperatures both below and above the mean temperature of the test.

A2.2 *Time Responses of Heat Flux Transducers:*

A2.2.1 *High Thermal Resistance Transducer*—A transducer with a high thermal resistance is generally used in Configuration a. When the specimens are preconditioned to the mean temperature of the test and when the plates are capable of both heating and cooling the specimen, the time response of a high resistance transducer will be more rapid than a comparable guarded hot plate apparatus. However, if the heat flux transducer has appreciable mass the response will not be rapid.

A2.2.1.1 The temperature drop across a high thermal resistance transducer may be greater than desirable, that is, one percent of the total, when used in Configuration c, and thus its use in this configuration is not recommended.

A2.2.1.2 Configuration b can be used to obtain very rapid response times if both plates are capable of heating and cooling and if the outputs of both transducers are summed, **(15)**. Configuration b is always faster than Configuration a with the same transducers and specimens and can be comparable to Configuration c if a low thermal resistance transducer is employed.

A2.2.2 *Low Thermal Resistance Transducers*—The low-thermal resistance, gradient-type heat flux transducer is better suited to the Configuration c. The temperature drop across the low-resistance transducer is small enough so that the two specimens can be considered as halves of a single specimen. When the specimens are first conditioned to the mean temperature of the test and when the specimens are identical, the response is sufficiently rapid to be used for quality control work.

TABLE A1.2a Conversion Factors (Thermochemical)

NOTE—Conversion factors for thermal resistivity and thermal conductance can be found by using these tables in the reverse direction.

Thermal Conductivity[A]						
	$\mathrm{W \cdot m^{-1} \cdot K^{-1}}$ ([B])	$\mathrm{W \cdot cm^{-1} \cdot K^{-1}}$	$\mathrm{cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}}$	$\mathrm{kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}}$	$\mathrm{Btu \cdot h^{-1} \cdot ft^{-1} \cdot {}^{\circ}F^{-1}}$	$\mathrm{Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot {}^{\circ}F^{-1}}$
$1\ \mathrm{W \cdot m^{-1} \cdot K^{-1}} =$	1.000	1.000×10^{-2}	2.390×10^{-3}	0.8604	0.5782	6.938
$1\ \mathrm{W \cdot cm^{-1} \cdot K^{-1}} =$	100.0	1.000	0.2390	86.04	57.82	693.8
$1\ \mathrm{cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}} =$	418.4	4.184	1.000	360.0	241.9	2903
$1\ \mathrm{kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}} =$	1.162	1.162×10^{-2}	2.778×10^{-3}	1.000	0.6720	8.064
$1\ \mathrm{Btu \cdot h^{-1} \cdot ft^{-1} \cdot {}^{\circ}F^{-1}} =$	1.730	1.730×10^{-2}	4.134×10^{-3}	1.488	1.000	12.00
$1\ \mathrm{Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot {}^{\circ}F^{-1}} =$	0.1441	1.441×10^{-3}	3.445×10^{-4}	0.1240	8.333×10^{-2}	1.000

Thermal Resistance[A]					
	$\mathrm{K \cdot m^2 \cdot W^{-1}}$ ([B])	$\mathrm{K \cdot cm^2 \cdot W^{-1}}$	$\mathrm{K \cdot cm^2 \cdot s \cdot cal^{-1}}$	$\mathrm{K \cdot m^2 \cdot kg\text{-}cal^{-1}}$	$\mathrm{{}^{\circ}F \cdot ft^2 \cdot h \cdot Btu^{-1}}$
$1\ \mathrm{K \cdot m^2 \cdot W^{-1}} =$	1.000	1.000×10^{4}	4.184×10^{4}	1.162	5.674
$1\ \mathrm{K \cdot cm^2 \cdot W^{-1}} =$	1.000×10^{-4}	1.000	4.184	1.162×10^{-4}	5.674×10^{-4}
$1\ \mathrm{K \cdot cm^2 \cdot s \cdot cal^{-1}} =$	2.390×10^{-5}	0.2390	1.000	2.778×10^{-5}	1.356×10^{-4}
$1\ \mathrm{K \cdot m^2 \cdot h \cdot kg\text{-}cal^{-1}} =$	0.8604	8.604×10^{3}	3.600×10^{4}	1.000	4.882
$1\ \mathrm{{}^{\circ}F \cdot ft^2 \cdot h \cdot Btu^{-1}} =$	0.1762	1.762×10^{3}	7.373×10^{3}	0.2048	1.000

[A] Units are given in terms of (*1*) the absolute joule per second or watt, (2) the calorie (thermochemical) = 4.184 J, or the British thermal unit (thermochemical) = 1054.35 J.

[B] This is the SI (International System of Units) unit.

TABLE A1.2b Conversion Factors (International Table)

NOTE—Conversion factors for thermal resistivity and thermal conductance can be found by using these tables in the reverse direction.

Thermal Conductivity[A]						
	$\mathrm{W \cdot m^{-1} \cdot K^{-1}}$ ([B])	$\mathrm{W \cdot cm^{-1} \cdot K^{-1}}$	$\mathrm{cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}}$	$\mathrm{kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}}$	$\mathrm{Btu \cdot h^{-1} \cdot ft^{-1} \cdot {}^{\circ}F^{-1}}$	$\mathrm{Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot {}^{\circ}F^{-1}}$
$1\ \mathrm{W \cdot m^{-1} \cdot K^{-1}} =$	1.000	1.000×10^{-2}	2.388×10^{-3}	0.8598	0.5778	6.933
$1\ \mathrm{W \cdot cm^{-1} \cdot K^{-1}} =$	100.0	1.000	0.2388	85.98	57.78	693.3
$1\ \mathrm{cal \cdot s^{-1} \cdot cm^{-1} \cdot K^{-1}} =$	418.7	4.187	1.000	360.0	241.9	2903.
$1\ \mathrm{kg\text{-}cal \cdot h^{-1} \cdot m^{-1} \cdot K^{-1}} =$	1.163	1.163×10^{-2}	2.778×10^{-3}	1.000	0.6720	8.064
$1\ \mathrm{Btu \cdot h^{-1} \cdot ft^{-1} \cdot {}^{\circ}F^{-1}} =$	1.731	1.731×10^{-2}	4.134×10^{-3}	1.488	1.000	12.00
$1\ \mathrm{Btu \cdot in. \cdot h^{-1} \cdot ft^{-2} \cdot {}^{\circ}F^{-1}} =$	0.1442	1.442×10^{-3}	3.445×10^{-4}	0.1240	8.333×10^{-2}	1.000

Thermal Resistance[A]					
	$\mathrm{K \cdot m^2 \cdot W^{-1}}$ ([B])	$\mathrm{K \cdot cm^2 \cdot W^{-1}}$	$\mathrm{K \cdot cm^2 \cdot s \cdot cal^{-1}}$	$\mathrm{K \cdot m^2 \cdot h \cdot kg\text{-}cal^{-1}}$	$\mathrm{{}^{\circ}F \cdot ft^2 \cdot h \cdot Btu^{-1}}$
$1\ \mathrm{K \cdot m^2 \cdot W^{-1}} =$	1.000	1.000×10^{4}	4.187×10^{4}	1.163	5.678
$1\ \mathrm{K \cdot cm^2 \cdot W^{-1}} =$	1.000×10^{-4}	1.000	4.187	1.163×10^{-4}	5.678×10^{-4}
$1\ \mathrm{K \cdot cm^2 \cdot s \cdot cal^{-1}} =$	2.388×10^{-5}	0.2388	1.000	2.778×10^{-5}	1.356×10^{-4}
$1\ \mathrm{K \cdot m^2 \cdot h \cdot kg\text{-}cal^{-1}} =$	0.8598	8.598×10^{3}	3.600×10^{4}	1.000	4.882
$1\ \mathrm{{}^{\circ}F \cdot ft^2 \cdot h \cdot Btu^{-1}} =$	0.1761	1.761×10^{3}	7.373×10^{3}	0.2048	1.000

[A] Units are given in terms of (*1*) the absolute joule per second or watt, (2) the calorie (International Table) = 4.1868 J, or the British thermal unit (International Table) = 1055.06 J. These tables are meant for use in Canada and Great Britain.

[B] This is the SI (International System of Units) unit.

A2.2.2.1 Where half thicknesses of the normal specimen can be used, it can be more rapid than Configuration a, especially when each of the specimens is first conditioned to the mean temperature at which it will operate in the test. The use of the low-resistance transducer in Configuration a is ideal if Plate 1 can provide both heating and cooling, but unless the temperature of Plate 1 is extremely well controlled, a damping layer will be required between the transducer and the plate. The advantage of the fast response time is then partially lost, but the response time is likely rapid enough in any event. The transducer can be mounted permanently in this configuration.

A2.3 *Sensitivity of Configurations to Edge Losses:*

A2.3.1 The Configuration a is similar to the guarded hot plate apparatus regarding edge heat losses through the specimen. The edge heat losses in the transducer may be much more significant than those in the guarded hot plate apparatus because they may produce errors due to the temperature non-uniformity on the side in contact with the specimen.

A2.3.2 The Configuration b is the most insensitive to edge conditions if the average of the readings of the two meters is assumed to be the measured heat flux per unit area through the specimen. If the plates were perfectly uniform in temperature, if the two transducers were exactly equal in the layout of the thermopile junctions, and if the specimen had thermal conductivity independent of temperature, this configuration would be insensitive to edge conditions. However, even under these ideal conditions the use of Configuration b does not eliminate edge losses but only reduces the apparatus sensitivity to variations in the boundary temperatures. Also, the settling time is short if the average of the two readings is used.

A2.3.3 Configuration c is very sensitive to edge heat losses on the heat flux transducer as the power that flows through the edges is supplied, not by a heavy nearly isothermal metal plate, but by the specimens, so that their surface temperatures may not be uniform. If the transducer is sensitive to temperature differences along its main surfaces, edge heat

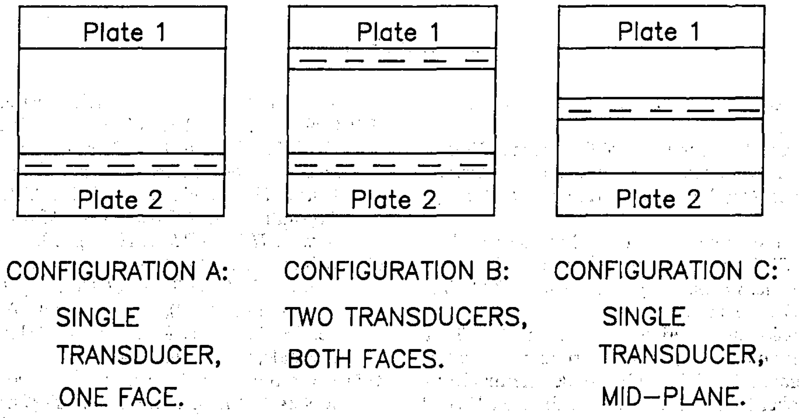


FIG. A2.1 Typical Heat Flow Meter Apparatus Configurations

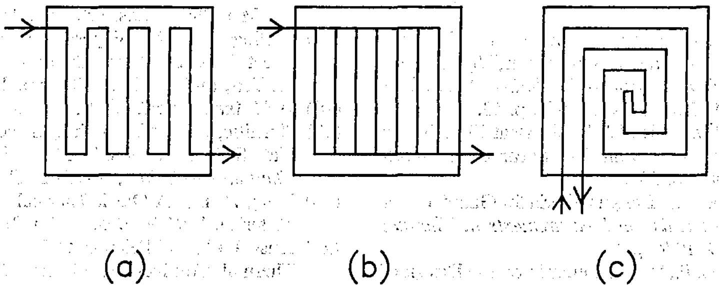


FIG. A2.2 Some Layouts of the Liquid Paths in Heating or Cooling Plates

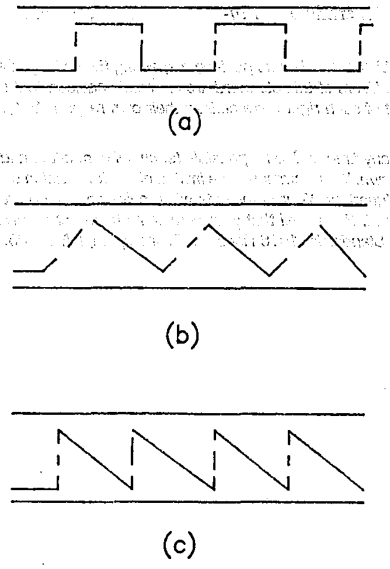


FIG. A2.3 Schematic Designs of Transducers

losses may now create serious errors. Edge heat losses within the specimens are similar to those in the guarded hot plate when the surrounding temperature is that of the hot or cold plate.

REFERENCES

(1) Tye, R. P., *Thermal Conductivity Vols I and II*, Academic Press, London and N.Y., 1969.

(2) Degenne, Klarsfeld, Barthe, "Measurement of the Thermal Resistance of Thick Low-Density Mineral Fiber Insulation," *Thermal Transmission Measurements of Insulation, ASTM STP 660*, 1978, p. 130.

(3) Pelanne, C. M., "Does the Insulation Have a Thermal Conductivity? The Revised ASTM Test Standards Require an Answer," *Thermal Transmission Measurements of Insulation, ASTM STP 660*, R. P. Tye, Ed., 1978, pp. 60–70.

(4) Pelanne, C. M., "Discussion on Experiments to Separate the Effect of Thickness' from Systematic Equipment Errors in Thermal Transmission Measurements," DOE/ASTM C16.00 Thermal Insulation Conference October 22–25, 1978, Tampa, FL, *ASTM STP 718*, McElroy and Tye Eds, 1980, pp. 322, 334.

(5) Zabawsky, Z., "Construction and Calibration of a Heat Flow Meter for Thermal Conductivity Measurements," *Paper No. 68-520*, ISA.

(6) Hollingsworth, M., Jr., "An Apparatus for Thermal Conductivity at Cryogenic Temperatures Using a Heat Flow Meter," *Symposium on Thermal Conductivity Measurements of Insulating Materials at Cryogenic Temperatures, ASTM STP 411*, 1967, p. 43.

(7) Pelanne, C. M., and Bradley, C. B., "A Rapid Heat Flow Meter Thermal-Conductivity Apparatus," *Materials Research and Standards*, Vol 2, No. 7, July 1962, p. 549.

(8) De Ponte, F., and Di Filippo, P., "Design Criteria for Guarded Hot Plate Apparatus," *Heat Transmission Measurements in Thermal Insulations, ASTP STP 544*, 1974, p. 97.

(9) De Ponte, F., and Di Filippo, P., "Some Remarks on the Design of Isothermal Plates," Prec. Meeting, Comm. B1, 11R, Zurich 1943–1944, pp. 145–155.

(10) Rennex, B. "Error Analysis for the National Bureau of Standards 1016 mm Guarded Hot Plate," *NBSIR 83-2674*, Washington, DC, 1983.

(11) Marechal, J. C., "Metrologie et Conductivite Thermique," *Materiaux et Constructions*, Janvier-Fevrier 1974, No. 37, pp. 61–65.

(12) Clulow, A., and Rees, W. H., "The Transmission of Heat through Textile Fabrics, Part III, A New Thermal Transmission Apparatus," *Journal of the Textile Institute*, 1968, pp. 286–294.

(13) Kinzer, G. R., and Pelanne, C. M., "A Cryogenic Heat-Flow-Meter Apparatus," *Heat Transmission Measurement in Thermal Insulations, ASTM STP 544*, 1974, p. 13.

(14) Baines, D. J., "A Comparative Theoretical Evaluation of Five Commonly Used Types of Unsteady Heat Flux Sensor," *Report HSA* (prior to 1970), Dept. of Supply, Australian Defense Scientific Service, Weapons Research Establishment, Box 1424H, G.P.O., Adelaide, South Australia, 5001.

(15) De Ponte, F. and Maccato, W. "The Calibration of Heat Flow Meters," *Thermal Insulation Performance, ASTM STP 718*, McElroy and Tye Eds, 1980, pp. 237–254.

(16) Bomberg, M. and Solvason, K. R., "Comments on Calibration and Design of a Heat Flow Meter," *ASTM STP 789*, Govan, Greason, and McCallister, Eds, 1983, pp. 277–292.

(17) Hollingsworth, M. "Experimental Determination of the Thickness Effect in Glass Fiber Building Insulation," *ASTM STP 718*, McElroy and Tye Eds, 1980, pp. 255–271.

(18) *1982 Annual Book of ASTM Standards*, Part 18, pp. 35–47.

(19) Shirtliffe, C. J., "Establishing Steady-State Thermal Conditions in Flat Slab Specimens," *Heat Transmission Measurement in Thermal Insulations, ASTM STP 544*, 1974, p. 13.

(20) Lang, D. L., "A Quick Thermal Conductivity Test on Insulating Materials," *ASTM Bulletin No. 216*, September 1956.

(21) Hust, J. G., and Pelanne, C. M., "Round Robins on the Apparent Thermal Conductivity of Low-Density Glass Fiber Insulations using Guarded Hot Plate and Heat Flow Meter Apparatus," *NBSIR 85-3026*, May 1985.

(22) Adams, R. D., and Hust, J. G., "A Round Robin on Apparent Thermal Conductivity of Several Loose-Fill Insulations," *Insulation Materials, Testing and Applications, ASTM STP* 1030, June 1990, p. 263.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM C549: Standard Specification for Perlite Loose Fill Insulation

CFR Section(s): 10 CFR 440 Appendix A

Standards Body: American Society for Testing and Materials

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: C 549 – 81 (Reapproved 1995)$^{\epsilon 1}$

Standard Specification for Perlite Loose Fill Insulation[1]

This standard is issued under the fixed designation C 549; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

ϵ^1 NOTE—Keywords were added editorially in May 1995.

1. Scope

1.1 This specification covers the composition and physical properties of expanded perlite loose fill insulation. The specification also includes the testing procedures by which the acceptability of the material may be determined. These testing procedures deal primarily with material performance in the temperature range associated with the thermal envelope of buildings; however, the commercially usable temperature range for this insulation is from – 459 to 1400°F (1 to 1033 K). For specialized applications, refer to the manufacturer's instructions.

1.2 The specification also covers the composition and properties of perlite that has been surface-treated to produce water repellency and dust suppression for installations where liquid moisture and dust may be a factor.

1.3 *When the installation or use of thermal insulation materials, accessories and systems, may pose safety or health problems, the manufacturer shall provide the user appropriate current information regarding any known problems associated with the recommended use of the company's products, and shall also recommend protective measures to be employed in their safe utilization. The user shall establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.* For additional precautionary statements, see Section 11.

1.4 The following precautionary caveat pertains to the test methods portion, Section 8, of this specification. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates[2]

C 168 Terminology Relating to Thermal Insulating Materials[3]

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded Hot Plate Apparatus[3]

C 390 Criteria for Sampling and Acceptance of Preformed Thermal Insulation Lots[3]

C 518 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus[3]

C 520 Test Methods for Density of Granular Loose Fill Insulations[3]

E 84 Test Method for Surface Burning Characteristics of Building Materials[4]

E 136 Test Method for Behavior of Materials in a Vertical Tube Furnace at 750°C[4]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[5]

2.2 *Federal Specification:*

HH-I-515D Insulation Thermal (Loose Fill for Pneumatic or Poured Application) Cellulosic or Wood Fiber[6]

3. Classification

3.1 Perlite loose fill insulation shall be specified by four type designations as follows:

3.1.1 *Type I*—The product that results from the expanding of natural perlite ore by grading and heating to meet the requirements of this specification.

---

[1] This specification is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.23 on Blanket and Loose Fill Insulation.

Current edition approved Oct. 30, 1981. Published February 1982. Originally published as C 549 – 64 T. Last previous edition C 549 – 73 (1979).

[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.06.

[4] *Annual Book of ASTM Standards*, Vol 04.07.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

3.1.2 *Type II*—Expanded perlite that has been surface-treated to produce water repellency and limited absorption of moisture from both liquid and vapor phase.

3.1.3 *Type III*—Expanded perlite that has been surface-treated to limit the amount of dust generated during application.

3.1.4 *Type IV*—Expanded perlite that has been surface-treated to produce water repellency and to limit the dust generated during application.

4. Ordering Information

4.1 All purchase orders should designate the type of insulation desired. The type and grade classification in this specification differ from the classification in earlier issues. Purchasers referencing this specification should include the date of issue.

5. Materials and Manufacture

5.1 Perlite is a generic term for a naturally occurring siliceous mineral which is mined, crushed, sieved and dried to produce a crude perlite ore. The crude ore, in the form of particles of varying size, weighing 60 to 75 lb/ft^3 (960 to 1200 kg/m^3), is expanded in high-temperature furnaces to densities in the range from 2.0 to 11.0 lb/ft^3 (32 to 176 kg/m^3). As a naturally occurring mineral, it is classified as an elementary building material. It is noncombustible as determined by Test Method C 136.

6. Physical Requirements

6.1 The physical requirements listed in this section are defined as Inspection Requirements (see Criteria C 390). The insulation shall conform to the following requirements:

	All Types
Bulk density, lb/ft^3(kg/m^3)	2–11 (32–176)
Grading (particle size):	
Cumulative percentage retained, maximum (by weight), Sieve No. 4 (4.75 mm), %	5

	Types I and III	Types II and IV only
Water repellency, min mL of water repelled	N.A.	175
Small-scale fire test	no sparking or ignition	

6.2 The physical requirements listed in this section are defined as Qualification Requirements (see Criteria C 390). The insulation shall conform to the following requirements:

	All Types
Thermal resistance, h·ft^2·°F/Btu (m^2·K/W)	See Table 1
Moisture absorption, weight %/14 days, max, %	1.0

	Types I and III	Types II and IV
Wickability, max g wicked in 5 min	N.A.	1.0
Combustibility (by Test Method E 136)	noncombustible	N.A.[A]
Surface burning characteristics:		
Flame spread, max	0	25
Smoke developed, max	0	50
Dust suppression:		
Weight of collected material, max, mg	N.A.	85

[A] Federal Standards for attic floor insulation require conformance to two non-ASTM tests for combustibility: Critical Radiant Flux (HH-I-515D, 4.8.7) and Smoldering Combustion (HH-I-515D, 4.8.8). Types III and IV perlite insulation, when tested for Critical Radiant Flux, showed no ignition or flame front advance at a flux of 1.07 W/cm^2. When tested for Smoldering Combustion, Types III and IV perlite insulation showed no flaming combustion and 0 % weight loss.

7. Sampling

7.1 For purposes of standard tests, sampling shall be in accordance with Criteria C 390.

8. Test Methods

8.1 *Bulk Density*—Test Methods C 520, Method A.

8.2 *Grading*—Test Method C 136, except that when a mechanical sieving device is used, the sieving time shall be 5 min and the test sample shall be 50 g of material.

8.3 *Water Repellency* (Types II and IV only):

8.3.1 *Apparatus*:

8.3.1.1 *Rigid Plastic Tube*, 50 mm inside diameter by 300 mm long with a 150-μm (100-mesh) screen covering firmly fastened or adhered to the bottom. The tube shall be marked at 400 mL from the screen-covered end.

8.3.1.2 *Rubber Stopper*, No. 15.

8.3.1.3 *Graduated Cylinder*, 250-mL.

8.3.1.4 *Beaker*, 500-mL.

8.3.2 *Sample Preparation*:

8.3.2.1 Spoon a representative sample into the test cylinder to a level slightly above the 400-mL mark.

8.3.2.2 Compact it by dropping the tube from a height of approximately 75 mm on a large rubber stopper (No. 15 recommended) for a total of ten drops. As the sample compacts to a level below the 400-mL mark, add additional material so that after the tenth drop the level of the sample is within 3 mL of the 400-mL mark.

8.3.3 *Procedure*:

8.3.3.1 With the tube supported in a vertical position and a beaker positioned under the tube, pour 250 mL of cold tap water rapidly onto the perlite. Take care while pouring, that the stream hits the middle of the surface of the bed of perlite and does not merely slide down the side of the test cylinder.

8.3.3.2 Allow the water to drain through the bed of perlite for exactly 3 min. Tilt the tube at approximately 45° to drain water collected on the screen. Tilt it only momentarily for this purpose.

8.3.3.3 Measure the collected water in the 250-mL graduate.

8.3.4 *Calculations*:

8.3.4.1 Report the amount of collected water in millilitres as "millilitres repelled."

8.3.4.2 Report results as the mean of three independent tests.

8.3.5 *Precision and Bias*—The purpose of this test is to confirm that the intended degree of surface treatment has been accomplished to achieve water repellency. The specified characteristic is a minimum (no range or maximum). Quantitative values for conforming products exceeding the limit have no commercial significance.

8.4 *Small-Scale Fire Test*:

8.4.1 *Scope*—The purpose of this quality control test is to confirm that excessive coating or organic material has not been added during processing.

8.4.2 *Significance*—This test is intended as a means to determine the resistance of the material to heat and flame under controlled conditions and should not be used to describe or appraise the fire hazard or fire risk under actual conditions.

TABLE 1 Thermal Resistance

Density, lb/ft^3(kg/m^3)	All Types Thermal Resistance for 1-in. (0.0254-m) Thickness, h·ft^2·°F/Btu (m^2·K/W)[A] Mean Temperature,° F (°C) 40 (4)	 75 (24)	 105 (41)
2.0–4.1 (32.0–65.6)	4.3–3.9 (0.78–0.69)	3.7–3.3 (0.65–0.58)	3.7–3.2 (0.65–0.56)
4.1–7.4 (65.6–118.4)	3.9–3.3 (0.69–0.58)	3.3–2.8 (0.58–0.49)	3.2–2.7 (0.56–0.47)
7.4–11.0 (118.4–176.0)	3.3–2.9 (0.58–0.51)	2.8–2.4 (0.49–0.42)	2.7–2.4 (0.47–0.42)

[A] The thermal values in this table are given at 1 in. (0.0254 m) of thickness with a gradient of 50°F (10°C). Test data that cover the thickness range from 1 to 6 in. (0.0254 to 0.1524 m) show that thermal resistance per inch of thickness is essentially linear with respect to thickness. Test values do not deviate on the low side from the value in the table by more than 5 %.

8.4.3 *Procedure*—Hold a small sample (such as 10 to 20 gr) of the perlite insulation in a suitable fixture (such as 6 by 6 by 2-in. deep wire mesh basket constructed from (150 µm 100-mesh) stainless steel woven wire cloth) and positioned in direct contact with the flame of a bunsen burner for about 20 s. Sparking or ignition indicates failure to conform to the requirements of 6.1

8.4.4 *Precision and Bias*—No statement is made about either the precision or the accuracy of the small-scale fire test for determining resistance of the perlite insulation to heat and flame since the result merely states whether there is conformance to the criteria for success specified in the procedure.

8.5 *Thermal Resistance*—Tests of thermal resistance may be made in accordance with Test Method C 177 or C 518. Testing shall be done at the design density. The thermal resistance of the various types shall not be lower than the values listed in Table 1, except that the average thermal resistance of any four specimens measured in accordance with 8.5, may fall up to 5 % below the value in the table. The thermal resistance (*R*-value) shall be determined at mean temperature of 75°F (24°C) and 40°F (4°C) at design density and in accordance with the current editions of Test Methods C 177 and C 518. Report the direction of heat flow. Thermal resistance at other mean temperatures may be determined if required.

8.6 *Moisture Absorption*—The test specimen shall be a sample of approximately 50 g. Loose fill and level the sample into a sample holder 9 by 9 by 5 in. (228 by 228 by 127 mm) deep.

8.6.1 Precondition in accordance with Test Method C 177 or C 518. Conditioning shall be with minimum air movement across the sample surface. Condition at 50 ± 2 % relative humidity and 120°F (49°C) to constant weight and record. State the density of the sample conditioned to constant weight in the report of results.

8.6.2 Increase the relative humidity to 90 ± 2 %. Condition to constant weight by check-weighing at 24-h intervals. Determine the moisture pickup as a percent of conditioned weight.

8.6.3 Record percent absorption at 14 days.

8.6.4 *Precision and Bias*—This test establishes a typical property of perlite. It is an inherent characteristic. It is only intended to indicate absorption under high humidity conditions which are known to be characteristic of its usual insulation end use.

8.7 *Wickability* (Types II and IV only):

8.7.1 *Apparatus*:

8.7.1.1 *Rigid Plastic Tube*, 50 mm inside diameter by 300 mm long with a 150-µm (100-mesh) screen covering firmly fastened or adhered to the bottom.

8.7.1.2 *Compaction Plug*, 50 mm long, fitting snugly inside the sample holder to give standard compaction to the sample.

8.7.1.3 *Tubing Rack*, capable of supporting the sample tube.

8.7.1.4 *Water Immersion Tray.*

8.7.2 *Sample Preparation*:

8.7.2.1 Spoon a representative sample into the test cylinder to a level slightly below the top of the cylinder.

8.7.2.2 Compact it with the plug until a moderate resistance pressure is felt. In no event insert the compaction plug into the sample holding tube more than 24 mm.

8.7.3 *Procedure*:

8.7.3.1 Place the sample tube screen end down, on a rack in the water immersion tray. The water level in the tray will be 50 mm above the bottom of the tube.

8.7.3.2 Sample shall be in this position and allowed to wick water for 5 min.

8.7.3.3 Remove the tube from the tray onto a tared 75-mm watch glass so that all water that is allowed to drain is caught by the watch glass and is included in the weight of water picked up by the sample.

8.7.3.4 Determine the weight of the water picked up by the sample in grams.

8.7.4 *Calculations*:

8.7.4.1 Report the amount of water picked up by the perlite sample through this wicking action as grams.

8.7.4.2 Report results as the mean of three independent tests.

8.7.5 *Precision and Bias*—The purpose of this test is to ascertain the resistance of the material to wicking action of water. The specified characteristic is a maximum (no range or minimum). Quantitative values for conforming products below the limit have no commercial significance.

8.8 *Combustibility* of Types I and II perlite insulation may be determined in accordance with Test Method E 136. Combustibility of Types III and IV insulation may be determined in accordance with tests on Critical Radiant Flux and Smoldering Combustion as specified in Federal Specification HH-I-515D.

8.9 *Surface Burning Characteristics* of perlite insulation may be determined in accordance with Test Method E 84.

8.10 *Dust Suppression* (Types III and IV only):

8.10.1 *Apparatus*:

8.10.1.1 *Glass Plate*, 16½ by 16½ by ¼ in. (420 by 420 by 6.4 mm).

8.10.1.2 *Clear Methacrylate Sheet Box*, open bottom, 15½ by 15½ by 19⅝ in. (400 by 400 by 500 mm) high with a hole in the center of the top 2$\frac{1}{16}$ in. (52.4 mm) in diameter.

8.10.1.3 *Plastic Tube*, 2 in. outside diameter by 1¾ in. inside diameter (50 by 45 mm), 17¾ in. long (450 mm), with a rubber washer such that the washer snugly fits the outside diameter of the plastic tube and has a larger outside diameter than the hole in the box.

8.10.1.4 *Beaker*, 2000-mL.

8.10.1.5 *Laboratory Funnel* with a minimum nozzle inside diameter of 1 in. (25.4 mm).

8.10.1.6 *Graduated Cylinder*, 100-mL.

8.10.1.7 *Small Brush.*

8.10.1.8 *Scale* capable of measuring perlite material to 0.001 g.

8.10.2 *Sample Preparation*—Spoon a representative sample into the graduated cylinder to the 1000-mL level.

8.10.3 *Procedure*:

8.10.3.1 Place the glass plate flat on a suitable work surface.

8.10.3.2 Place the 2000-mL beaker in the exact center of the glass plate.

8.10.3.3 Place the plastic box on the glass plate such that the box is centered on the plate and hole in top of the box is centered directly over the 2000-mL beaker.

8.10.3.4 Insert the plastic pipe into the hole in the top of the box such that it protrudes down into the box such that the clear vertical distance from the bottom of the tube to the top of the 2000-mL beaker is $1\frac{3}{16}$ in. (30 mm). The tube is supported in the vertical position by the rubber washer.

8.10.3.5 Place the laboratory funnel into the plastic tube.

8.10.3.6 Pour 1000 mL of test material into the funnel such that it empties of all material in 10 s. It is the intent to have a uniform stream of material falling into the box for 10 s.

8.10.3.7 After all visible material has settled onto the glass plate, carefully remove the plastic box and gently sweep the material that has collected on the glass plate into a pile, remove it, and determine its weight.

8.10.4 *Calculations*:

8.10.4.1 Report the amount of test material in milligrams as "milligrams collected."

8.10.4.2 Report results as the average of five or more independent tests with no single reading over 100 mg with separate samples taken from the same source.

8.10.5 *Precision and Bias*—The single-specimen, single-operator, single-day precision is a standard deviation of ±0.02 g (1S) maximum for the dust-suppressed material over a range from 0.01 to 0.08 g as defined in Practice E 177.

9. Inspection

9.1 Inspection of the materials shall be made as agreed upon by the purchaser and the manufacturer as part of the purchase contract.

10. Packaging and Marking

10.1 *Packaging*—Unless otherwise specified, the insulation shall be packed in the manufacturer's standard commercial container. The insulation shall be packed to ensure carrier acceptance and safe delivery at destination in containers complying with the rules and regulations applicable to the mode of transportation.

10.2 *Marking*—Shipping containers shall be marked with the name of the insulation, minimum weight of container, and the name of the manufacturer. For residential insulation, a chart shall also be affixed or printed on the container listing the minimum thickness, maximum net coverage area, and maximum weight per square foot at minimum *R* values of 11, 19, and 22. In addition to this chart, the following statements must be added: 1. "*R* means resistance to heat flow. The higher the *R*-value, the greater the insulating power." 2. "To get the marked *R*-value, it is essential that this insulation be installed properly. If you do it yourself, follow the instructions carefully."

11. Health and Safety Precautions

11.1 *Preinstallation*—The insulation material should be handled and stored in accordance with manufacturer's instructions. It should be kept dry and free of extraneous materials.

11.2 *Installation*:

11.2.1 The insulating material should be poured into the spaces and cavities to be insulated in a manner that minimizes free-fall and impact. This will minimize crushing and breakdown of insulation particles and unnecessary formation of dust.

11.2.2 Perlite loose fill insulation that may be installed in confined, poorly ventilated attic spaces may generate a buildup of airborne nuisance dust. Avoid creating this nuisance dust. The use of respiratory and eye protection may be necessary in some applications. Refer to manufacturer's instructions regarding recommended installation practices.

12. Keywords

12.1 dust suppression; insulation; loose fill; perlite; water repellency

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM C564: Standard Specification for Rubber Gaskets for Cast Iron Soil Pipe and Fittings

CFR Section(s): 24 CFR 3280.611(d)(5)(iv)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: C 564 – 70 (Reapproved 1982)

Endorsed by the
Cast Iron Soil Pipe Institute

Standard Specification for
RUBBER GASKETS FOR CAST IRON SOIL PIPE AND FITTINGS[1]

This standard is issued under the fixed designation C 564; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This specification has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This specification covers performed rubber gaskets used to seal joints in cast iron soil pipe and fittings.

1.2 The values stated in inch-pound units are to be regarded as the standard.

2. Materials and Manufacture

2.1 Gaskets shall be made of a properly vulcanized virgin rubber compound containing no scrap or reclaim.

2.2 If a splice occurs in the gasket, the strength of the splice shall be such that the gasket will withstand the stretch test described in 7.9 with no visible separation or peeling.

3. Physical Requirements

3.1 Sample gaskets selected as specified in Section 6 shall conform to the requirements for physical properties listed in Table 1 when tested in accordance with the methods specified in Section 7.

4. Dimensions and Permissible Variations

4.1 Gaskets shall conform to the dimensions specified by the manufacturer of the pipe in which they are to be used.

4.2 All cross-sectional dimensions shall have an RMA Class 3 tolerance as shown in the appendix, and all diametral dimensions shall have a tolerance of ± 1 percent unless otherwise mutually agreed by the pipe manufacturer and the gasket supplier.

5. Workmanship

5.1 The surface of the gasket shall be smooth and free of pitting, cracks, blisters, air marks, and any other imperfections that will affect its behavior in service. The body of the gasket shall be free of porosity and air pockets.

5.2 Neither the flash thickness nor the flash extension shall exceed $\frac{1}{32}$ in. (1 mm), at any point on the ring.

5.3 The offset, or failure of the mold to register accurately, shall not exceed $\frac{1}{64}$ in. (0.5 mm).

6. Sampling

6.1 For the stretch test specified in 7.9, sample gaskets shall be selected at random from each shipment of gaskets, in accordance with Table 2. For each of the other tests, gaskets shall be selected at random as required by the method of test specified.

7. Test Methods

7.1 *Hardness*—Test for hardness in accordance with ASTM Method D 2240, Test for Rubber Property—Durometer Hardness.[2] ASTM Method D 1415, Test for Rubber Property—International Hardness,[2] shall be used as the referee method. Make hardness measurements on specimens prepared in accordance with 7.2. However, hardness readings for guidance purposes may be taken directly on the gasket, recognizing that these may vary slightly from those taken on the dumb-bell specimens.

7.2 *Elongation and Tensile Strength*—Test for elongation and tensile strength in accordance with ASTM Method D 412, Tests for Rubber Properties in Tension[3] Cut standard

---

[1] This specification is under the jurisdiction of ASTM Committee C-24 on Building Seals and Sealants.

Current edition effective May 29, 1970. Originally issued 1965. Replaces C 564 – 68.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

[3] *Annual Book of ASTM Standards*, Vols 09.01 and 09.02.

ASTM Type C dumbbell specimens conforming to Fig. 1 of Method D 412 from sections of the gasket for this test. To obtain a uniform thickness, these sections may be buffed prior to cutting into dumbbell specimens, so as to produce a finely ground surface without cuts or burns.

7.3 *Tear Strength*—Test for tear strength in accordance with ASTM Method D 624, Test for Rubber Property—Tear Resistance,[2] using Die C.

7.4 *Compression Set*—Test for compression set in accordance with ASTM Methods D 395, Tests for Rubber Property—Compression Set,[2] using Method B. Oven age specimens 22 h at 158 ± 2 F (70 ± 1 C). Where plied specimens are necessary, the results shall comply with the requirements of Table 1.

7.5 *Heat Aging*—Test for effects of heat aging in accordance with ASTM Method D 573, Test for Rubber Deterioration in an Air Oven.[2] Prepare specimens in accordance with 7.2, and age for 96 h at 158 ± 2 F (70 ± 1 C). Hardness measurements shall be made as specified in 7.1.

7.6 *Water Absorption*—Test for weight increase due to water absorption in accordance with ASTM Method D 471, Test for Rubber Property—Effect of Liquids.[2] If a 1-in. (25.4-mm) specimen cannot be cut from the sample gasket, use the greatest width obtainable. Immerse the test specimen in distilled water at 158 ± 2 F (70 ± 1 C) for 7 days.

7.7 *Ozone Resistance*—Test for ozone resistance in accordance with ASTM Method D 1149, Test for Rubber Deterioration—Surface Ozone Cracking in a Chamber (Flat Specimens),[2] using specimens and procedure specified under Method B. The ozone concentration shall be 150 parts/100,000,000 of air by volume. Age specimens 100 h at 104 F (40 C). Use a two-power hand glass to examine the gasket for cracks.

7.8 *Oil Immersion*—Test for volume decrease due to oil absorption in accordance with Method D 471. If a 1-in (25.4-mm) specimen cannot be cut from the sample gasket, the greatest width obtainable shall be used. The test specimen shall be immersed in ASTM Oil No. 3 for 70 h at 212 ± 2 F (100 ± 1 C).

7.9 *Stretch Test for Spliced Gaskets*—Stretch gaskets until the circumference is increased 50 percent, then visually inspect for defects as described in 2.2 and 5.1. The number of gaskets to be examined and the maximum number of defective gaskets for acceptance of the lot is shown in Table 2.

8. Certification

8.1 Upon request of the purchaser, the manufacturer shall be prepared to certify that his product conforms to the requirements of this specification.

9. Marking

9.1 Mark each gasket with clearly legible letters not exceeding ¼ in. (6.35 mm) in height. These markings shall include the gasket manufacturer's name or symbol, the pipe size and class, the year of manufacture, and the ASTM specification number.

TABLE 1 Physical Requirements of Gaskets

Property	Requirements			ASTM Test Method
Hardness (nominal durometer ±5) as specified by the pipe manufacturer	50	60	70	D 2240
Elongation, min, percent	350	300	250	D 412
Tensile strength, min, psi	1500	1500	1500	D 412
(MPa)	(10)	(10)	(10)	
Tear strength, min, lbf/in.	150	150	150	D 624
(N/cm)	(268)	(268)	(268)	
Compression set, max, percent	25	25	25	D 395
Heat aging, 96 h at 158 ± 2 F (70 ± 1 C):				D 573
Hardness increase, max, durometer points	10	10	10	. . .
Loss in tensile strength, max, percent	15	15	15	. . .
Loss in elongation, max, percent	20	20	20	. . .
Water absorption:				D 471
Weight increase, max, percent	20	20	20	. . .
Ozone resistance	no cracks	no cracks	no cracks	D 1149
Oil immersion:				
Volume increase, max, percent	80	80	80	D 471

TABLE 2 Sampling Plan for Stretch Test for Visual Inspection

Number of Gaskets in Shipment	Number of Gaskets in Sample	Maximum Number of Defectives for Acceptance
Up to 800	75	4
801 to 3200	150	8
3201 to 8000	225	11
8001 to 22 000	300	14

APPENDIX

X1. RUBBER MANUFACTURERS ASSOCIATION, INC. TOLERANCES

TABLE X1 RMA Class 3 Dimensional Tolerances (Commercial Tolerances)

Size, in.	Fixed[A]	Closure[B,C]
0 to 0.499	±0.010	±0.015
0.500 to 0.999	±0.010	±0.018
1.000 to 1.999	±0.015	±0.020
2.000 to 2.999	±0.020	±0.025
3.000 to 3.999	±0.025	±0.030
4.000 to 4.999	±0.030	±0.035
5.000 to 7.999	±0.035	±0.050
Greater than 8.000	multiply by	0.0050

[A] Fixed dimensions are those which are parallel to the mold parting line or major mold sections and which are not affected by flash thickness variations. Tolerances apply individually to each fixed dimension according to its own size.

[B] Closure dimensions are those vertical to the mold parting line or parting lines of major sections and are affected by flash thickness variation.

[C] The tolerance on closure dimensions is that tolerance for the largest closure dimension. This tolerance is then applied to all other closure dimensions.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D86: Standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure

CFR Section(s): 40 CFR 1065.710

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed [redacted]

Designation: D 86 – 07

An American National Standard

Standard Test Method for
Distillation of Petroleum Products at Atmospheric Pressure[1]

This standard is issued under the fixed designation D 86; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method covers the atmospheric distillation of petroleum products using a laboratory batch distillation unit to determine quantitatively the boiling range characteristics of such products as light and middle distillates, automotive spark-ignition engine fuels, aviation gasolines, aviation turbine fuels, 1-D and 2-D regular and low sulfur diesel fuels, special petroleum spirits, naphthas, white spirits, kerosines, and Grades 1 and 2 burner fuels.

1.2 The test method is designed for the analysis of distillate fuels; it is not applicable to products containing appreciable quantities of residual material.

1.3 This test method covers both manual and automated instruments.

1.4 Unless otherwise noted, the values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 All standards are subject to revision, and parties to agreement on this test method are to apply the most recent edition of the standards indicated below, unless otherwise specified, such as in contractual agreements or regulatory rules where earlier versions of the method(s) identified may be required.

2.2 *ASTM Standards:* [2]

D 97 Test Method for Pour Point of Petroleum Products
D 323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)
D 2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)
D 5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method)
D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)
D 5842 Practice for Sampling and Handling of Fuels for Volatility Measurement
D 5949 Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)
D 5950 Test Method for Pour Point of Petroleum Products (Automatic Tilt Method)
D 5985 Test Method for Pour Point of Petroleum Products (Rotational Method)
E 1 Specification for ASTM Liquid-in-Glass Thermometers
E 77 Test Method for Inspection and Verification of Thermometers
E 1272 Specification for Laboratory Glass Graduated Cylinders
E 1405 Specification for Laboratory Glass Distillation Flasks

2.3 *Energy Institute Standards:*[3]

IP 69 Determination of Vapour Pressure—Reid Method
IP 123 Petroleum Products—Determination of Distillation Characteristics
IP 394 Determination of Air Saturated Vapour Pressure
IP Standard Methods for Analysis and Testing of Petroleum and Related Products 1996—Appendix A

---

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08.0A on Distillation.

In the IP, the equivalent test method is published under the designation IP 123. It is under the jurisdiction of the Standardization Committee.

Current edition approved Jan. 15, 2007. Published February 2007. Originally approved in 1921. Last previous edition approved in 2005 as D 86–05.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

***A Summary of Changes section appears at the end of this standard.**

TABLE 1 Preparation of Apparatus

		Group 1	Group 2	Group 3	Group 4
Flask, mL		125	125	125	125
ASTM distillation thermometer		7C (7F)	7C (7F)	7C (7F)	8C (8F)
IP distillation thermometer range		low	low	low	high
Flask support board		B	B	C	C
diameter of hole, mm		38	38	50	50
Temperature at start of test					
Flask	°C	13–18	13–18	13–18	not above ambient
	°F	55–65	55–65	55–65	
Flask support and shield		not above ambient	not above ambient	not above ambient	
Receiving cylinder and 100 mL charge					
	°C	13–18	13–18	13–18[A]	13–ambient[A]
	°F	55–65	55–65	55–65[A]	55–ambient[A]

[A] See 10.3.1.1 for exceptions.

3. Terminology

3.1 *Definitions:*

3.1.1 *charge volume, n*—the volume of the specimen, 100 mL, charged to the distillation flask at the temperature specified in Table 1.

3.1.2 *decomposition, n—of a hydrocarbon,* the pyrolysis or cracking of a molecule yielding smaller molecules with lower boiling points than the original molecule.

3.1.2.1 *Discussion*—Characteristic indications of thermal decomposition are evolution of fumes and erratic temperature readings that usually decrease after any attempt is made to adjust the heat.

3.1.3 *decomposition point, n*—the corrected thermometer reading that coincides with the first indications of thermal decomposition of the liquid in the flask.

3.1.3.1 *Discussion*—The decomposition point, as determined under the conditions of this test method, does not necessarily correspond to the decomposition temperature in other applications.

3.1.4 *dry point, n*—the corrected thermometer reading that is observed at the instant the last drop of liquid (exclusive of any drops or film of liquid on the side of the flask or on the temperature sensor), evaporates from the lowest point in the distillation flask.

3.1.4.1 *Discussion*—The end point (final boiling point), rather than the dry point, is intended for general use. The dry point can be reported in connection with special purpose naphthas, such as those used in the paint industry. Also, it is substituted for the end point (final boiling point) whenever the sample is of such a nature that the precision of the end point (final boiling point) cannot consistently meet the requirements given in the precision section.

3.1.5 *dynamic holdup, n*—the amount of material present in the neck of the flask, in the sidearm of the flask, and in the condenser tube during the distillation.

3.1.6 *emergent stem effect, n*—the offset in temperature reading caused by the use of total immersion mercury-in-glass thermometers in the partial immersion mode.

3.1.6.1 *Discussion*—In the partial immersion mode, a portion of the mercury thread, that is, the emergent portion, is at a lower temperature than the immersed portion, resulting in a shrinkage of the mercury thread and a lower temperature reading.

3.1.7 *end point (EP) or final boiling point (FBP), n*—the maximum corrected thermometer reading obtained during the test.

3.1.7.1 *Discussion*—This usually occurs after the evaporation of all liquid from the bottom of the flask. The term maximum temperature is a frequently used synonym.

3.1.8 *front end loss, n*—loss due to evaporation during transfer from receiving cylinder to distillation flask, vapor loss during the distillation, and uncondensed vapor in the flask at the end of the distillation.

3.1.9 *initial boiling point (IBP), n*—the corrected thermometer reading that is observed at the instant the first drop of condensate falls from the lower end of the condenser tube.

3.1.10 *percent evaporated, n*—the sum of the percent recovered and the percent loss.

3.1.11 *percent loss (or observed loss), n*—one hundred minus the percent total recovery.

3.1.11.1 *corrected loss, n*—percent loss corrected for barometric pressure.

3.1.12 *percent recovered, n*—the volume of condensate observed in the receiving cylinder, expressed as a percentage of the charge volume, associated with a simultaneous temperature reading.

3.1.13 *percent recovery, n*—the maximum percent recovered, as observed in accordance with 10.18.

3.1.13.1 *corrected percent recovery, n*—the percent recovery, adjusted for the difference between the observed loss and the corrected loss, as described in Eq 8.

3.1.13.2 *percent total recovery, n*—the combined percent recovery and residue in the flask, as determined in accordance with 11.1.

3.1.14 *percent residue, n*—the volume of residue in the flask, measured in accordance with 10.19, and expressed as a percentage of the charge volume.

3.1.15 *rate of change (or slope), n*—the change in temperature reading per percent evaporated or recovered, as described in 13.2.

3.1.16 *temperature lag, n*—the offset between the temperature reading obtained by a temperature sensing device and the true temperature at that time.

3.1.17 *temperature measurement device, n*—a thermometer, as described in 6.3.1, or a temperature sensor, as described in 6.3.2.

3.1.18 *temperature reading, n*—the temperature obtained by a temperature measuring device or system that is equal to the thermometer reading described in 3.1.19.

3.1.18.1 *corrected temperature reading, n*—the temperature reading, as described in 3.1.18, corrected for barometric pressure.

3.1.19 *thermometer reading (or thermometer result), n*—the temperature of the saturated vapor measured in the neck of the flask below the vapor tube, as determined by the prescribed thermometer under the conditions of the test.

3.1.19.1 *corrected thermometer reading, n*—the thermometer reading, as described in 3.1.19, corrected for barometric pressure.

4. Summary of Test Method

4.1 Based on its composition, vapor pressure, expected IBP or expected EP, or combination thereof, the sample is placed in one of four groups. Apparatus arrangement, condenser temperature, and other operational variables are defined by the group in which the sample falls.

4.2 A 100-mL specimen of the sample is distilled under prescribed conditions for the group in which the sample falls. The distillation is performed in a laboratory batch distillation unit at ambient pressure under conditions that are designed to provide approximately one theoretical plate fractionation. Systematic observations of temperature readings and volumes of condensate are made, depending on the needs of the user of the data. The volume of the residue and the losses are also recorded.

4.3 At the conclusion of the distillation, the observed vapor temperatures can be corrected for barometric pressure and the data are examined for conformance to procedural requirements, such as distillation rates. The test is repeated if any specified condition has not been met.

4.4 Test results are commonly expressed as percent evaporated or percent recovered versus corresponding temperature, either in a table or graphically, as a plot of the distillation curve.

5. Significance and Use

5.1 The basic test method of determining the boiling range of a petroleum product by performing a simple batch distillation has been in use as long as the petroleum industry has existed. It is one of the oldest test methods under the jurisdiction of ASTM Committee D02, dating from the time when it was still referred to as the Engler distillation. Since the test method has been in use for such an extended period, a tremendous number of historical data bases exist for estimating end-use sensitivity on products and processes.

5.2 The distillation (volatility) characteristics of hydrocarbons have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the behavior of the fuel during storage and use. Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapors.

5.3 The distillation characteristics are critically important for both automotive and aviation gasolines, affecting starting, warm-up, and tendency to vapor lock at high operating temperature or at high altitude, or both. The presence of high boiling point components in these and other fuels can significantly affect the degree of formation of solid combustion deposits.

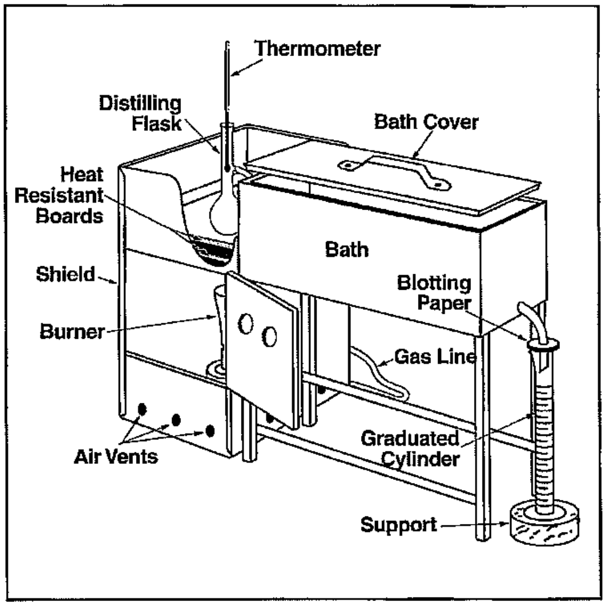


FIG. 1 Apparatus Assembly Using Gas Burner

5.4 Volatility, as it affects rate of evaporation, is an important factor in the application of many solvents, particularly those used in paints.

5.5 Distillation limits are often included in petroleum product specifications, in commercial contract agreements, process refinery/control applications, and for compliance to regulatory rules.

6. Apparatus

6.1 *Basic Components of the Apparatus*:

6.1.1 The basic components of the distillation unit are the distillation flask, the condenser and associated cooling bath, a metal shield or enclosure for the distillation flask, the heat source, the flask support, the temperature measuring device, and the receiving cylinder to collect the distillate.

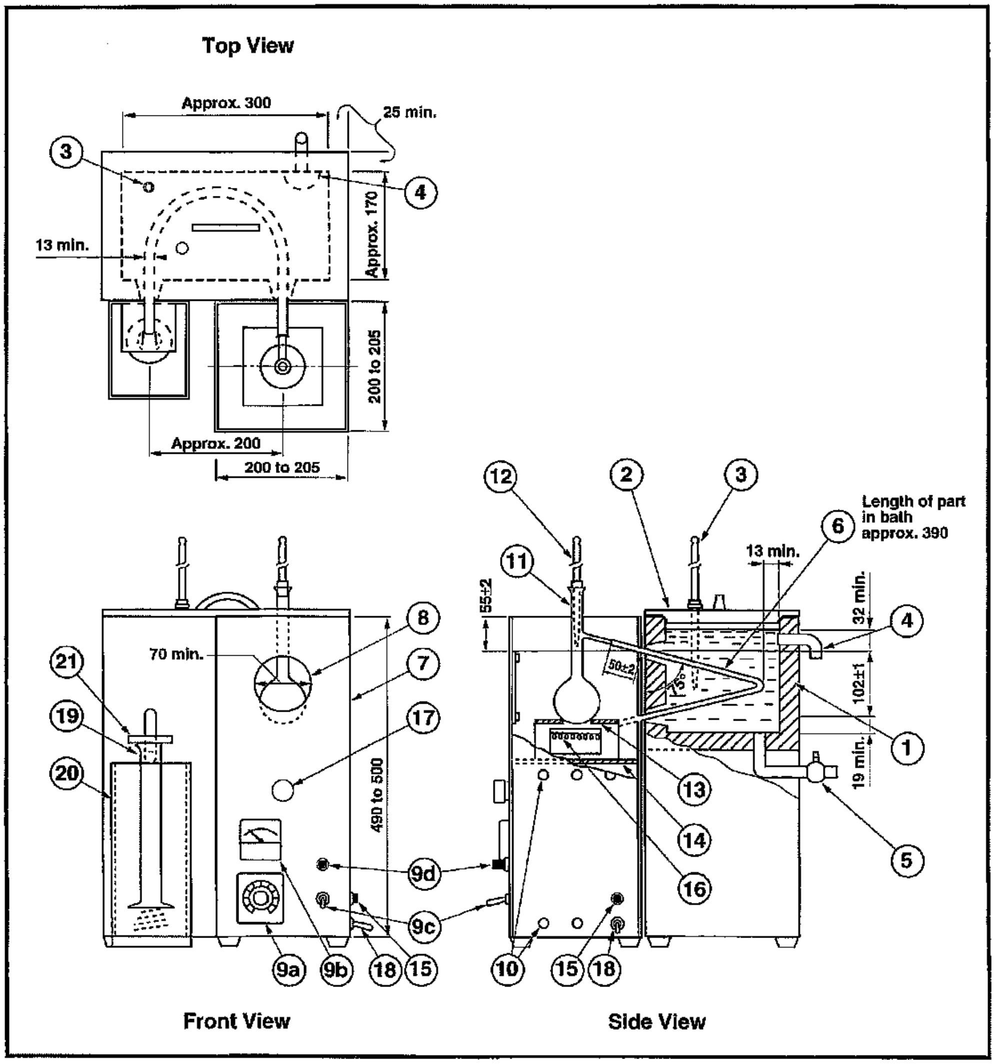
6.1.2 Figs. 1 and 2 are examples of manual distillation units.

6.1.3 In addition to the basic components described in 6.1.1, automated units also are equipped with a system to measure and automatically record the temperature and the associated recovered volume in the receiving cylinder.

6.2 A detailed description of the apparatus is given in Annex A2.

6.3 *Temperature Measuring Device*:

6.3.1 Mercury-in-glass thermometers, if used, shall be filled with an inert gas, graduated on the stem and enamel backed. They shall conform to Specification E 1 or IP Standard Methods for Analysis and Testing of Petroleum and Related Products 1996—Appendix A, or both, for thermometers ASTM



1–Condenser bath
2–Bath cover
3–Bath temperature sensor
4–Bath overflow
5–Bath drain
6–Condenser tube
7–Shield
8–Viewing window
9a–Voltage regulator
9b–Voltmeter or ammeter
9c–Power switch
9d–Power light indicator
10–Vent
11–Distillation flask
12–Temperature sensor
13–Flask support board
14–Flask support platform
15–Ground connection
16–Electric heater
17–Knob for adjusting level of support platform
18–Power source cord
19–Receiver cylinder
20–Receiver cooling bath
21–Receiver cover

FIG. 2 Apparatus Assembly Using Electric Heater

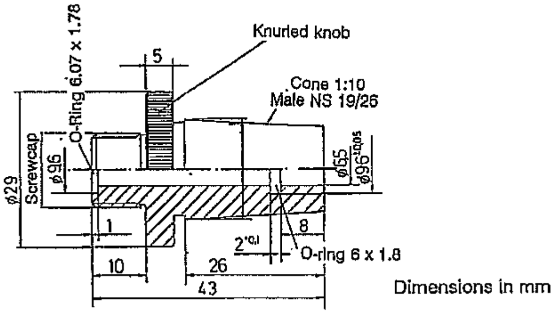


FIG. 3 PTFE Centering Device for Ground Glass Joint

7C/IP 5C and ASTM 7F for the low range thermometers, and ASTM 8C/IP 6C and ASTM 8F for the high range thermometers.

6.3.1.1 Thermometers that have been exposed for an extended period above an observed temperature of 370°C shall not be reused without a verification of the ice point or checked as prescribed in Specification E 1 and Test Method E 77.

NOTE 1—At an observed thermometer reading of 370°C, the temperature of the bulb is approaching a critical range in the glass and the thermometer may lose its calibration.

6.3.2 Temperature measurement systems other than those described in 6.3.1 are satisfactory for this test method, provided that they exhibit the same temperature lag, emergent stem effect, and accuracy as the equivalent mercury-in-glass thermometer.

6.3.2.1 The electronic circuitry or the algorithms, or both, used shall include the capability to simulate the temperature lag of a mercury-in-glass thermometer.

6.3.2.2 Alternatively, the sensor can also be placed in a casing with the tip of the sensor covered so that the assembly, because of its adjusted thermal mass and conductivity, has a temperature lag time similar to that of a mercury-in-glass thermometer.

NOTE 2—In a region where the temperature is changing rapidly during the distillation, the temperature lag of a thermometer can be as much as 3 seconds.

6.3.3 In case of dispute, the referee test method shall be carried out with the specified mercury-in-glass thermometer.

6.4 *Temperature Sensor Centering Device*:

6.4.1 The temperature sensor shall be mounted through a snug-fitting device designed for mechanically centering the sensor in the neck of the flask without vapor leakage. Examples of acceptable centering devices are shown in Figs. 3 and 4. (**Warning**—The use of a plain stopper with a hole drilled through the center is not acceptable for the purpose described in 6.4.1.)

NOTE 3—Other centering devices are also acceptable, as long as they position and hold the temperature sensing device in the proper position in the neck of the distillation column, as shown in Fig. 5 and described in 10.5.

NOTE 4—When running the test by the manual method, products with

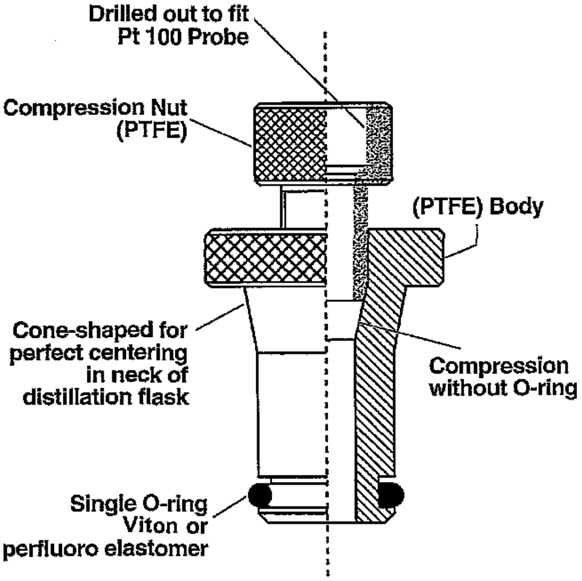


FIG. 4 Example of Centering Device Designs for Straight-Bore Neck Flasks

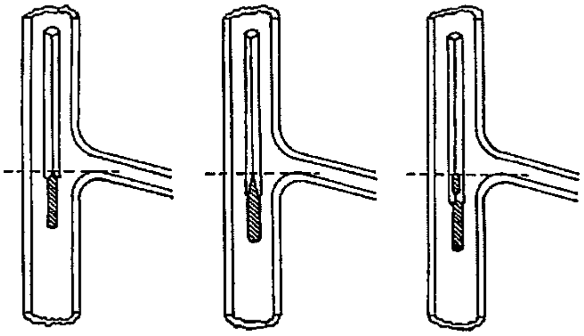


FIG. 5 Position of Thermometer in Distillation Flask

a low IBP may have one or more readings obscured by the centering device. See also 10.14.3.1.

6.5 Automated equipment manufactured in 1999 and later shall be equipped with a device to automatically shut down power to the unit and to spray an inert gas or vapor in the chamber where the distillation flask is mounted in the event of fire.

NOTE 5—Some causes of fires are breakage of the distillation flask, electrical shorts, and foaming and spilling of liquid sample through the top opening of the flask.

6.6 *Barometer*—A pressure measuring device capable of measuring local station pressure with an accuracy of 0.1 kPa (1 mm Hg) or better, at the same elevation relative to sea level as the apparatus in the laboratory. (**Warning**—Do not take readings from ordinary aneroid barometers, such as those used

TABLE 2 Group Characteristics

	Group 1	Group 2	Group 3	Group 4
Sample characteristics				
Distillate type				
Vapor pressure at				
37.8°C, kPa	≥65.5	<65.5	<65.5	<65.5
100°F, psi	≥9.5	<9.5	<9.5	<9.5
(Test Methods D 323, D 4953, D 5190, D 5191, D 5482, IP 69 or IP 394)				
Distillation, IBP °C			≤100	>100
°F			≤212	>212
EP °C	≤250	≤250	>250	>250
°F	≤482	≤482	>482	>482

at weather stations and airports, since these are precorrected to give sea level readings.)

7. Sampling, Storage, and Sample Conditioning

7.1 Determine the Group characteristics that correspond to the sample to be tested (see Table 2). Where the procedure is dependent upon the group, the section headings will be so marked.

7.2 *Sampling*:

7.2.1 Sampling shall be done in accordance with Practice D 4057 or D 4177 and as described in Table 3.

7.2.1.1 *Group 1*—Condition the sample container to below 10°C, preferably by filling the bottle with the cold liquid sample and discarding the first sample. If this is not possible because, for instance, the product to be sampled is at ambient temperature, the sample shall be drawn into a bottle prechilled to below 10°C, in such a manner that agitation is kept at a minimum. Close the bottle immediately with a tight-fitting closure. (**Warning**—Do not completely fill and tightly seal a cold bottle of sample because of the likelihood of breakage on warming.)

7.2.1.2 *Groups 2, 3, and 4*—Collect the sample at ambient temperature. After sampling, close the sample bottle immediately with a tight-fitting closure.

7.2.1.3 If the sample received by the testing laboratory has been sampled by others and it is not known whether sampling has been performed as described in 7.2, the sample shall be assumed to have been so sampled.

7.3 *Sample Storage*:

7.3.1 If testing is not to start immediately after collection, store the samples as indicated in 7.3.2, 7.3.3, and Table 3. All samples shall be stored away from direct sunlight or sources of direct heat.

7.3.2 *Group 1*—Store the sample at a temperature below 10°C.

NOTE 6—If there are no, or inadequate, facilities for storage below 10°C, the sample may also be stored at a temperature below 20°C, provided the operator ensures that the sample container is tightly closed and leak-free.

7.3.3 *Group 2*—Store the sample at a temperature below 10°C.

NOTE 7—If there are no, or inadequate, facilities for storage below 10°C, the sample may also be stored at a temperature below 20°C, provided the operator ensures that the sample container is tightly closed and leak-free.

7.3.4 *Groups 3 and 4*—Store the sample at ambient or lower temperature.

7.4 *Sample Conditioning Prior to Analysis*:

7.4.1 Samples shall be conditioned to the temperature shown in Table 3 before opening the sample container.

7.4.1.1 *Groups 1 and 2*—Samples shall be conditioned to a temperature of less than 10°C (50°F) before opening the sample container.

7.4.1.2 *Groups 3 and 4*—If the sample is not fluid at ambient temperature, it is to be heated to a temperature of 9 to 21°C above its pour point (Test Method D 97, D 5949, or D 5985) prior to analysis. If the sample has partially or completely solidified during storage, it shall be vigorously shaken after melting prior to opening the sample container to ensure homogeneity.

7.4.1.3 If the sample is not fluid at room temperature, the temperature ranges shown in Table 3 for the flask and for the sample do not apply.

7.5 *Wet Samples*:

7.5.1 Samples of materials that visibly contain water are not suitable for testing. If the sample is not dry, obtain another sample that is free from suspended water.

7.5.2 *Groups 1 and 2*—If such a sample cannot be obtained, the suspended water can be removed by maintaining the sample at 0 to 10°C, adding approximately 10 g of anhydrous sodium sulfate per 100 mL of sample, shaking the mixture for approximately 2 min, and then allowing the mixture to settle for approximately 15 min. Once the sample shows no visible signs of water, use a decanted portion of the sample, maintained between 1 and 10°C, for the analysis. Note in the report that the sample has been dried by the addition of a desiccant.

NOTE 8—Suspended water in hazy samples in Groups 1 and 2 can be removed by the addition of anhydrous sodium sulfate and separating the liquid sample from the drying agent by decanting without statistically affecting the results of the test.[4]

7.5.3 *Groups 3 and 4*—In cases in which a water-free sample is not practical, the suspended water can be removed by shaking the sample with anhydrous sodium sulfate or other suitable drying agent and separating it from the drying agent by decanting. Note in the report that the sample has been dried by the addition of a desiccant.

8. Preparation of Apparatus

8.1 Refer to Table 1 and prepare the apparatus by choosing the appropriate distillation flask, temperature measuring device, and flask support board, as directed for the indicated group. Bring the temperature of the receiving cylinder, the flask, and the condenser bath to the indicated temperature.

8.2 Make any necessary provisions so that the temperature of the condenser bath and the receiving cylinder will be maintained at the required temperatures. The receiving cylinder shall be in a bath such that either the liquid level is at least

---

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1455.

TABLE 3 Sampling, Storage, and Sample Conditioning

		Group 1	Group 2	Group 3	Group 4
Temperature of sample bottle	°C	<10			
	°F	<50			
Temperature of stored sample	°C	<10[A]	<10	ambient	ambient
	°F	<50[A]	<50	ambient	ambient
Temperature of sample after conditioning prior to analysis	°C	<10	<10	Ambient or	Ambient or
				9 to 21°C above pour point[B]	
	°F	<50	<50	Ambient or	Ambient or
				48 to 70°F above pour point[B]	
If sample is wet		resample	resample	dry in accordance with 7.5.3	
If resample is still wet[C]		dry in accordance with 7.5.2			

[A] Under certain circumstances, samples can also be stored at temperatures below 20°C (68°F). See also 7.3.2 and 7.3.3.

[B] If sample is (semi)-solid at ambient temperature, see also 10.3.1.1.

[C] If sample is known to be wet, resampling may be omitted. Dry sample in accordance with 7.5.2 and 7.5.3.

as high as the 100-mL mark or the entire receiving cylinder is surrounded by an air circulation chamber.

8.2.1 *Groups 1, 2, and 3*—Suitable media for low temperature baths include, but are not limited to, chopped ice and water, refrigerated brine, and refrigerated ethylene glycol.

8.2.2 *Group 4*—Suitable media for ambient and higher bath temperatures include, but are not limited to, cold water, hot water, and heated ethylene glycol.

8.3 Remove any residual liquid in the condenser tube by swabbing with a piece of soft, lint-free cloth attached to a cord or wire.

9. Calibration and Standardization

9.1 *Temperature Measurement System*—Temperature measurement systems using other than the specified mercury-in-glass thermometers shall exhibit the same temperature lag, emergent stem effect, and accuracy as the equivalent mercury-in-glass thermometer. Confirmation of the calibration of these temperature measuring systems shall be made at intervals of not more than six months, and after the system has been replaced or repaired.

9.1.1 The accuracy and the calibration of the electronic circuitry or computer algorithms, or both, shall be verified by the use of a standard precision resistance bench. When performing this verification, no algorithms shall be used to correct the temperature for lag and the emergent stem effect (see manufacturer's instructions).

9.1.2 Verification of the calibration of temperature measuring devices shall be conducted by distilling toluene in accordance with Group 1 of this test method and comparing the 50 % recovered temperature with that shown in Table 4.[5]

9.1.2.1 If the temperature reading is not within the values shown in Table 4 for the respective apparatus being used (see Note 10 and Table 4), the temperature measurement system shall be considered defective and shall not be used for the test.

NOTE 9—Toluene is used as a verification fluid for calibration; it will yield almost no information on how well an electronic measurement system simulates the temperature lag of a liquid-in-glass thermometer.

9.1.2.2 Reagent grade toluene and hexadecane (cetane), conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[6] shall be used. However, other grades may also be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

NOTE 10—At 101.3 kPa, toluene is shown in reference manuals as boiling at 110.6°C when measured using a partial immersion thermometer. Because this test method uses thermometers calibrated for total immersion, the results typically will be lower and, depending on the thermometer and the situation, may be different for each thermometer. At 101.3 kPa, hexadecane is shown in reference manuals as boiling at 287.0°C when measured using a partial immersion thermometer. Because this test method uses thermometers calibrated for total immersion, the results typically will be lower, and, depending on the thermometer and the situation, may be different for each thermometer.

9.1.3 A procedure to determine the magnitude of the temperature lag is described in Annex A3.

9.1.4 A procedure to emulate the emergent stem effect is described in Appendix X4.

9.1.5 To verify the calibration of the temperature measurement system at elevated temperatures, use hexadecane. The temperature measurement system shall indicate, at 50% recovered, a temperature comparable to that shown in Table 4 for the respective apparatus under Group 4 distillation conditions.

NOTE 11—Because of the high melting point of hexadecane, Group 4 verification distillations will have to be carried out with condenser temperatures >20°C.

9.2 *Automated Method*:

9.2.1 *Level Follower*—For an automated distillation apparatus, the level follower/recording mechanism of the apparatus shall have a resolution of 0.1 mL or better with a maximum error of 0.3 mL between the 5 and 100 mL points. The calibration of the assembly shall be verified in accordance with manufacturer's instructions at intervals of not more than three months and after the system has been replaced or repaired.

NOTE 12—The typical calibration procedure involves verifying the output with the receiver containing 5 and 100 mL of material respectively.

9.2.2 *Barometric Pressure*—At intervals of not more than six months, and after the system has been replaced or repaired,

---

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1580.

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 4 True and Min and Max D 86 50 % Recovered Boiling Points (°C)[A]

		Manual		Automated	
		Distillation conditions min D 86 50 % boiling point	Distillation conditions max D 86 50 % boiling point	Distillation conditions min D 86 50 % boiling point	Distillation conditions max D 86 50 % boiling point
Toluene	ASTM/IP true boiling point	Group 1, 2, and 3	Group 1, 2, and 3	Group 1, 2, and 3	Group 1, 2, and 3
	110.6	105.9	111.8	108.5	109.7
Hexadecane	ASTM/IP true boiling point	Group 4	Group 4	Group 4	Group 4
	287.0	272.2	283.1	277.0	280.0

[A] The manual and automated temperatures show in this table are the values for the 95 % tolerance interval for the 99 % population coverage. The proposed tolerance is approximately 3 × sigma. Information on the values in this table can be found in RR:D02–1580.

the barometric reading of the instrument shall be verified against a barometer, as described in 6.6.

10. Procedure

10.1 Record the prevailing barometric pressure.

10.2 *Groups 1 and 2*—Fit a low range thermometer provided with a snug-fitting cork or stopper of silicone rubber, or equivalent polymeric material, tightly into the neck of the sample container and bring the temperature of the sample to the temperature indicated in Table 3.

10.3 *Groups 1, 2, 3, and 4*—Check that the temperature of the sample is as shown in Table 3. Pour the specimen precisely to the 100-mL mark of the receiving cylinder, and transfer the contents of the receiving cylinder as completely as practical into the distillation flask, ensuring that none of the liquid flows into the vapor tube.

NOTE 13—It is important that the difference between the temperature of the specimen and the temperature of the bath around the receiving cylinder is as small as practically possible. A difference of 5°C can make a difference of 0.7 mL.

10.3.1 *Groups 3 and 4*—If the sample is not fluid at ambient temperature, it is to be heated to a temperature between 9 and 21°C above its pour point (Test Methods D 97, D 5949, D 5950, or D 5985) prior to analysis. If the sample has partially or completely solidified in the intervening period, it shall be vigorously shaken after melting, and prior to sampling, to ensure homogeneity.

10.3.1.1 If the sample is not fluid at ambient temperatures, disregard the temperature range shown in Table 1 for the receiving cylinder and sample. Prior to analysis, heat the receiving cylinder to approximately the same temperature as the sample. Pour the heated specimen precisely to the 100-mL mark of the receiving cylinder, and transfer the contents of the receiving cylinder as completely as practical into the distillation flask, ensuring that none of the liquid flows into the vapor tube.

NOTE 14—Any material that evaporates during the transfer will contribute to the loss; any material that remains in the receiving cylinder will contribute to the observed recovery volume at the time of the IBP.

10.4 If the sample can be expected to demonstrate irregular boiling behavior, that is, bumping, add a few boiling chips to the specimen. The addition of a few boiling chips is acceptable for any distillation.

10.5 Fit the temperature sensor through a snug-fitting device, as described in 6.4, to mechanically center the sensor in the neck of the flask. In the case of a thermometer, the bulb is centered in the neck and the lower end of the capillary is level with the highest point on the bottom of the inner wall of the vapor tube (see Fig. 5). In the case of a thermocouple or resistance thermometer, follow the manufacturer's instructions as to placement (see Fig. 6).

NOTE 15—If vacuum grease is used on the mating surface of the centering device, use the minimum amount of grease that is practical.

10.6 Fit the flask vapor tube, provided with a snug-fitting cork or rubber stopper of silicone, or equivalent polymeric material, tightly into the condenser tube. Adjust the flask in a vertical position so that the vapor tube extends into the condenser tube for a distance from 25 to 50 mm. Raise and adjust the flask support board to fit it snugly against the bottom of the flask.

10.7 Place the receiving cylinder that was used to measure the specimen, without drying the inside of the cylinder, into its temperature-controlled bath under the lower end of the condenser tube. The end of the condenser tube shall be centered in the receiving cylinder and shall extend therein for a distance of at least 25 mm, but not below the 100-mL mark.

10.8 *Initial Boiling Point*:

10.8.1 *Manual Method*—To reduce evaporation loss of the distillate, cover the receiving cylinder with a piece of blotting paper, or similar material, that has been cut to fit the condenser tube snugly. If a receiver deflector is being used, start the distillation with the tip of the deflector just touching the wall of the receiving cylinder. If a receiver deflector is not used, keep the drip tip of the condenser away from the wall of the receiving cylinder. Note the start time. Observe and record the IBP to the nearest 0.5°C (1.0°F). If a receiver deflector is not being used, immediately move the receiving cylinder so that the tip of the condenser touches its inner wall.

10.8.2 *Automated Method*—To reduce evaporation loss of the distillate, use the device provided by the instrument manufacturer for this purpose. Apply heat to the distillation flask and contents with the tip of the receiver deflector just touching the wall of the receiving cylinder. Note the start time. Record the IBP to the nearest 0.1°C (0.2°F).

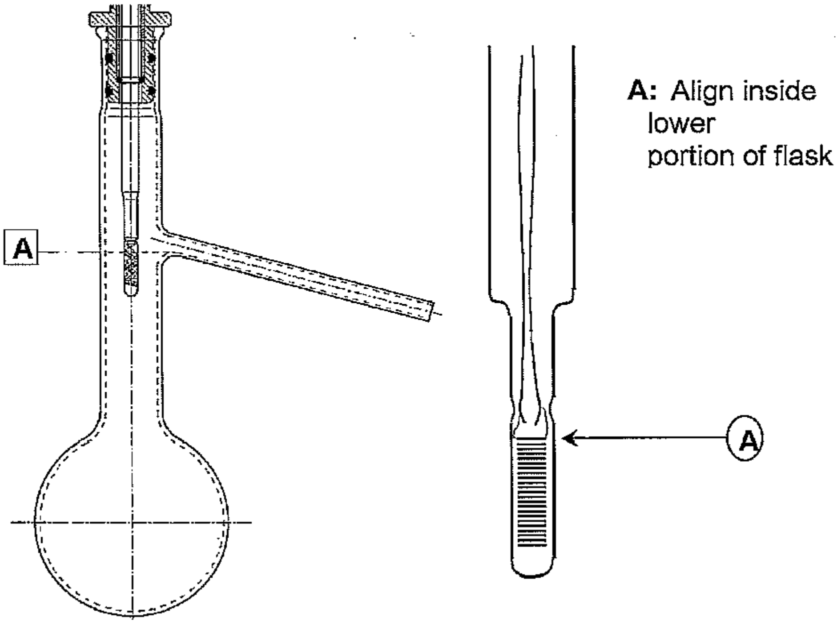


FIG. 6 Example of Recommended Placement of Pt-100 Probe Relative to Distillation Flask Sidearm for Automated D 86 Distillation Instrument

10.9 Regulate the heating so that the time interval between the first application of heat and the IBP is as specified in Table 5.

10.10 Regulate the heating so that the time from IBP to 5 or 10 % recovered is as indicated in Table 5.

10.11 Continue to regulate the heating so that the uniform average rate of condensation from 5 or 10 % recovered to 5 mL residue in the flask is 4 to 5 mL per min. (**Warning**—Due to the configuration of the boiling flask and the conditions of the test, the vapor and liquid around the temperature sensor are not in thermodynamic equilibrium. The distillation rate will consequently have an effect on the measured vapor temperature. The distillation rate shall, therefore, be kept as constant as possible throughout the test.)

NOTE 16—When testing gasoline samples, it is not uncommon to see the condensate suddenly form non-miscible liquid phases and bead up on the temperature measuring device and in the neck of the boiling flask at a vapor temperature of around 160°C. This may be accompanied by a sharp (about 3°C) dip in the vapor temperature and a drop in the recovery rate. The phenomenon, which may be due to the presence of trace water in the sample, may last for 10 to 30 s before the temperature recovers and the condensate starts flowing smoothly again. This point is sometimes colloquially referred to as the Hesitation Point.

10.12 Repeat any distillation that did not meet the requirements described in 10.9, 10.10, and 10.11.

10.13 If a decomposition point, as described in 3.1.3, is observed, discontinue the heating and proceed as directed in 10.17.

10.14 In the interval between the IBP and the end of the distillation, observe and record data necessary for the calculation and reporting of the results of the test as required by the specification involved, or as previously established for the sample under test. These observed data can include temperature readings at prescribed percentages recovered or percentages recovered at prescribed temperature readings, or both.

10.14.1 *Manual Method*—Record all volumes in the graduated cylinder to the nearest 0.5 mL, and all temperature readings to the nearest 0.5°C (1.0°F).

10.14.2 *Automated Method*—Record all volumes in the receiving cylinder to the nearest 0.1 mL, and all temperature readings to the nearest 0.1°C (0.2°F).

10.14.3 *Group 1, 2, 3, and 4*—In cases in which no specific data requirements have been indicated, record the IBP and the EP (FBP) or the dry point, or both, and temperature readings at 5, 15, 85, and 95 % recovered, and at each 10 % multiple of volume recovered from 10 to 90, inclusive.

10.14.3.1 *Group 4*—When a high range thermometer is used in testing aviation turbine fuels and similar products, pertinent thermometer readings can be obscured by the centering device. If these readings are required, perform a second distillation in accordance with Group 3. In such cases, reading from a low range thermometer can be reported in place of the obscured high range thermometer readings, and the test report shall so indicate. If, by agreement, the obscured readings are waived, the test report shall so indicate.

10.14.4 When it is required to report the temperature reading at a prescribed percent evaporated or recovered for a sample that has a rapidly changing slope of the distillation curve in the region of the prescribed percent evaporated or recovered reading, record temperature readings at every 1 % recovered. The slope is considered rapidly changing if the

TABLE 5 Conditions During Test Procedure

		Group 1	Group 2	Group 3	Group 4
Temperature of cooling bath[A]	°C	0–1	0–5	0–5	0–60
	°F	32–34	32–40	32–40	32–140
Temperature of bath around	°C	13–18	13–18	13–18	±3
receiving cylinder	°F	55–65	55–65	55–65	±5 of charge temperature
Time from first application of heat to initial boiling point, min		5–10	5–10	5–10	5–15
Time from initial boiling point to 5 % recovered, s		60–100	60–100		
to 10 % recovered, min					
Uniform average rate of condensation from 5 % recovered to 5 mL in flask, mL/min		4–5	4–5	4–5	4–5
Time recorded from 5 mL residue to end point, min		5 max	5 max	5 max	5 max

[A] the proper condenser bath temperature will depend upon the wax content of the sample and of its distillation fractions. The test is generally performed using one single condenser temperature. Wax formation in the condenser can be deduced from (*a*) the presence of wax particles in the distillate coming off the drip tip, (*b*) a higher distillation loss than what would be expected based on the initial boiling point of the specimen, (*c*) an erratic recovery rate and (*d*) the presence of wax particles during the removal of residual liquid by swabbing with a lint-free cloth (see 8.3). The minimum temperature that permits satisfactory operation shall be used. In general, a bath temperature in the 0 to 4°C range is suitable for kerosine, Grade No. 1 fuel oil and Grade No. 1-D diesel fuel oil. In some cases involving Grade No. 2 fuel oil, Grade No. 2-D diesel fuel oil, gas oils and similar distillates, it may be necessary to hold the condenser bath temperature in the 38 to 60°C range.

change in slope (C) of the data points described in 10.14.2 in that particular area is greater than 0.6 (change of slope (F) is greater than 1.0) as calculated by Eq 1 (Eq 2).

$$\text{Change of Slope } (C) = (C_2 - C_1)/(V_2 - V_1) - (C_3 - C_2)/(V_3 - V_2) \tag{1}$$

$$\text{Change of Slope } (F) = (F_2 - F_1)/(V_2 - V_1) - (F_3 - F_2)/(V_3 - V_2) \tag{2}$$

where:

C_1 = temperature at the volume % recorded one reading prior to the volume % in question, °C,
C_2 = temperature at the volume % recorded in question, °C,
C_3 = temperature at the volume % recorded following the volume % in question, °C,
F_1 = temperature at the volume % recorded one reading prior to the volume % in question, °F,
F_2 = temperature at the volume % recorded in question, °F,
F_3 = temperature at the volume % recorded following the volume % in question, °F,
V_1 = volume % recorded one reading prior to the volume % in question,
V_2 = volume % recorded at the volume % in question, and
V_3 = volume % recorded following the volume % in question.

10.15 When the residual liquid in the flask is approximately 5 mL, make a final adjustment of the heat. The time from the 5 mL of liquid residue in the flask to the EP (FBP) shall be within the limits prescribed in Table 5. If this condition is not satisfied, repeat the test with appropriate modification of the final heat adjustment.

NOTE 17—Since it is difficult to determine when there is 5 mL of boiling liquid left in the flask, this time is determined by observing the amount of liquid recovered in the receiving cylinder. The dynamic holdup has been determined to be approximately 1.5 mL at this point. If there are no front end losses, the amount of 5 mL in the flask can be assumed to correspond with an amount of 93.5 mL in the receiving cylinder. This amount has to be adjusted for the estimated amount of front end loss.

10.15.1 If the actual front end loss differs more than 2 mL from the estimated value, the test shall be rerun.

10.16 Observe and record the EP (FBP) or the dry point, or both, as required, and discontinue the heating.

10.17 Allow the distillate to drain into the receiving cylinder, after heating has been discontinued.

10.17.1 *Manual Method*—While the condenser tube continues to drain into the graduated cylinder, observe and note the volume of condensate to the nearest 0.5 mL at 2 min intervals until two successive observations agree. Measure the volume in the receiving cylinder accurately, and record it to the nearest 0.5 mL.

10.17.2 *Automated Method*—The apparatus shall continually monitor the recovered volume until this volume changes by no more than 0.1 mL in 2 min. Record the volume in the receiving cylinder accurately to the nearest 0.1 mL.

10.18 Record the volume in the receiving cylinder as percent recovery. If the distillation was previously discontinued under the conditions of a decomposition point, deduct the percent recovered from 100, report this difference as the sum of percent residue and percent loss, and omit the procedure given in 10.19.

10.19 After the flask has cooled and no more vapor is observed, disconnect the flask from the condenser, pour its contents into a 5-mL graduated cylinder, and with the flask suspended over the cylinder, allow the flask to drain until no appreciable increase in the volume of liquid in the cylinder is observed. Measure the volume in the graduated cylinder to the nearest 0.1 mL, and record as percent residue.

10.19.1 If the 5-mL graduated cylinder does not have graduations below 1 mL and the volume of liquid is less than 1 mL, prefill the cylinder with 1 mL of a heavy oil to allow a better estimate of the volume of the material recovered.

10.19.1.1 If a residue greater than expected is obtained, and the distillation was not purposely terminated before the EP, check whether adequate heat was applied towards the end of the distillation and whether conditions during the test conformed to those specified in Table 5. If not, repeat test.

NOTE 18—The distillation residues of this test method for gasoline, kerosine, and distillate diesel are *typically* 0.9–1.3, 0.9–1.3, and 1.0–1.4 volume %, respectively.

NOTE 19—The test method is not designed for the analysis of distillate fuels containing appreciable quantities of residual material (see 1.2).

10.19.2 *Groups 1, 2, 3, and 4*—Record the volume in the 5-mL graduated cylinder, to the nearest 0.1 mL, as percent residue.

10.20 If the intent of the distillation is to determine the percent evaporated or percent recovered at a predetermined corrected temperature reading, modify the procedure to conform to the instructions described in Annex A4.

10.21 Examine the condenser tube and the side arm of the flask for waxy or solid deposits. If found, repeat the test after making adjustments described in Footnote A of Table 5.

11. Calculations

11.1 The percent total recovery is the sum of the percent recovery (see 10.18) and the percent residue (see 10.19). Deduct the percent total recovery from 100 to obtain the percent loss.

11.2 Do not correct the barometric pressure for meniscus depression, and do not adjust the pressure to what it would be at sea level.

NOTE 20—The observed barometric reading does not have to be corrected to a standard temperature and to standard gravity. Even without performing these corrections, the corrected temperature readings for the same sample between laboratories at two different locations in the world will, in general, differ less than 0.1°C at 100°C. Almost all data obtained earlier have been reported at barometric pressures that have not been corrected to standard temperature and to standard gravity.

11.3 Correct temperature readings to 101.3 kPa (760 mm Hg) pressure. Obtain the correction to be applied to each temperature reading by means of the Sydney Young equation as given in Eq 3, Eq 4, or Eq 5, as appropriate, or by the use of Table 6. For Celsius temperatures:

$$C_c = 0.0009\,(101.3 - P_k)\,(273 + t_c) \qquad (3)$$

$$C_c = 0.00012\,(760 - P)\,(273 + t_c) \qquad (4)$$

For Fahrenheit temperatures:

$$C_f = 0.00012\,(760 - P)\,(460 + t_f) \qquad (5)$$

where:

t_c = the observed temperature reading in °C,
t_f = the observed temperature reading in °F,
C_c and C_f = corrections to be added algebraically to the observed temperature readings,
P_k = barometric pressure, prevailing at the time and location of the test, kPa, and
P = barometric pressure, prevailing at the time and location of the test, mm Hg.

After applying the corrections and rounding each result to the nearest 0.5°C (1.0°F) or 0.1°C (0.2°F), as appropriate to the apparatus being used, use the corrected temperature readings in all further calculations and reporting.

TABLE 6 Approximate Thermometer Reading Correction

Temperature Range		Correction[A] per 1.3 kPa (10 mm Hg) Difference in Pressure	
°C	°F	°C	°F
10–30	50–86	0.35	0.63
30–50	86–122	0.38	0.68
50–70	122–158	0.40	0.72
70–90	158–194	0.42	0.76
90–110	194–230	0.45	0.81
110–130	230–266	0.47	0.85
130–150	266–302	0.50	0.89
150–170	302–338	0.52	0.94
170–190	338–374	0.54	0.98
190–210	374–410	0.57	1.02
210–230	410–446	0.59	1.07
230–250	446–482	0.62	1.11
250–270	482–518	0.64	1.15
270–290	518–554	0.66	1.20
290–310	554–590	0.69	1.24
310–330	590–626	0.71	1.28
330–350	626–662	0.74	1.33
350–370	662–698	0.76	1.37
370–390	698–734	0.78	1.41
390–410	734–770	0.81	1.46

[A] Values to be added when barometric pressure is below 101.3 kPa (760 mm Hg) and to be subtracted when barometric pressure is above 101.3 kPa.

NOTE 21—Temperature readings are not corrected to 101.3 kPa (760 mm Hg) when product definitions, specifications, or agreements between the parties involved indicate, specifically, that such correction is not required or that correction shall be made to some other base pressure.

11.4 Correct the actual loss to 101.3 kPa (760 mm Hg) pressure when temperature readings are corrected to 101.3 kPa pressure. The corrected loss, L_c, is calculated from Eq 6 or Eq 7, as appropriate, or can be read from the tables presented as Fig. X3.1 or Fig. X3.2.

$$L_c = 0.5 + (L - 0.5)/\{1 + (101.3 - P_k)/8.00\} \qquad (6)$$

$$L_c = 0.5 + (L - 0.5)/\{1 + (760 - P)/60.0\} \qquad (7)$$

where:

L = observed loss,
L_c = corrected loss,
P_k = pressure, kPa, and
P = pressure, mm Hg.

NOTE 22—Eq 6 and 7 above have been derived from the data in Table 7 and Eqs 5 and 6 in Test Method D 86 – 95 and earlier versions. It is probable that Eq 6 and 7 shown were the original empirical equations from which the table and equations in the Test Method D 86 – 95 and earlier versions were derived.

11.4.1 Calculate the corresponding corrected percent recovery in accordance with the following equation:

$$R_c = R + (L - L_c) \qquad (8)$$

where:

L = percent loss or observed loss,
L_c = corrected loss,
R = percent recovery, and
R_c = corrected percent recovery.

TABLE 7 Data Points for Determining Slope, S_C or S_F

Slope at %	IBP	5	10	20	30	40	50	60	70	80	90	95	EP
T_L at %	0	0	0	10	20	30	40	50	60	70	80	90	95
T_U at %	5	10	20	30	40	50	60	70	80	90	90	95	V_{EP}
V_U- V_L	5	10	20	20	20	20	20	20	20	20	10	5	V_{EP}–95

11.5 To obtain the percent evaporated at a prescribed temperature reading, add the percent loss to each of the observed percent recovered at the prescribed temperature readings, and report these results as the respective percent evaporated, that is:

$$P_e = P_r + L \quad (9)$$

where:

L = observed loss,
P_e = percent evaporated, and
P_r = percent recovered.

11.6 To obtain temperature readings at prescribed percent evaporated, and if no recorded temperature data is available within 0.1 volume % of the prescribed percent evaporated, use either of the two following procedures, and indicate on the report whether the arithmetical procedure or the graphical procedure has been used.

11.6.1 *Arithmetical Procedure*—Deduct the observed loss from each prescribed percent evaporated to obtain the corresponding percent recovered. Calculate each required temperature reading as follows:

$$T = T_L + (T_H - T_L)(R - R_L)/(R_H - R_L) \quad (10)$$

where:

R = percent recovered corresponding to the prescribed percent evaporated,
R_H = percent recovered adjacent to, and higher than R,
R_L = percent recovered adjacent to, and lower than R,
T = temperature reading at the prescribed percent evaporated,
T_H = temperature reading recorded at R_H, and
T_L = temperature reading recorded at R_L.

Values obtained by the arithmetical procedure are affected by the extent to which the distillation graphs are nonlinear. Intervals between successive data points can, at any stage of the test, be no wider than the intervals indicated in 10.18. In no case shall a calculation be made that involves extrapolation.

11.6.2 *Graphical Procedure*—Using graph paper with uniform subdivisions, plot each temperature reading corrected for barometric pressure, if required (see 11.3), against its corresponding percent recovered. Plot the IBP at 0 % recovered. Draw a smooth curve connecting the points. For each prescribed percent evaporated, deduct the distillation loss to obtain the corresponding percent recovered and take from the graph the temperature reading that this percent recovered indicates. Values obtained by graphical interpolation procedures are affected by the care with which the plot is made.

NOTE 23—See Appendix X1 for numerical examples illustrating the arithmetical procedure.

11.6.3 In most automated instruments, temperature-volume data are collected at 0.1 volume % intervals or less and stored in memory. To report a temperature reading at a prescribed percent evaporated, neither of the procedures described in 11.6.1 and 11.6.2 have to be used. Obtain the desired temperature directly from the database as the temperature closest to and within 0.1 volume % of the prescribed percent evaporated.

12. Report

12.1 Report the following information (see Appendix X5 for examples of reports):

12.2 Report the barometric pressure to the nearest 0.1 kPa (1 mm Hg).

12.3 Report all volumetric readings in percentages.

12.3.1 *Manual Method*—Report volumetric readings to the nearest 0.5, and all temperature readings to the nearest 0.5°C (1.0°F).

12.3.2 *Automated Method*—Report volumetric readings to the nearest 0.1, and all temperature readings to the nearest 0.1°C (0.2°F) or less.

12.4 After barometric corrections of the temperature readings have been made, the following data require no further calculation prior to reporting: IBP, dry point, EP (FBP), decomposition point, and all pairs of corresponding values involving percent recovered and temperature readings.

12.4.1 The report shall state if the temperature readings have not been corrected for barometric pressure.

12.5 When the temperature readings have not been corrected to 101.3 kPa (760 mm Hg) pressure, report the percent residue and percent loss as *observed* in accordance with 10.19 and 11.1, respectively.

12.6 Do not use the corrected loss in the calculation of percent evaporated.

12.7 It is advisable to base the report on relationships between temperature readings and percent evaporated when the sample is a gasoline, or any other product classified under Group 1, or in which the percent loss is greater than 2.0. Otherwise, the report can be based on relationships between temperature readings and percent evaporated or percent recovered. Every report must indicate clearly which basis has been used.

12.7.1 In the manual method, if results are given in percent evaporated versus temperature readings, report if the arithmetical or the graphical procedure was used (see 11.6).

12.8 Report if a drying agent, as described in 7.5.2 or 7.5.3, was used.

12.9 Fig. X1.1 is an example of a tabular report. It shows the percent recovered versus the corresponding temperature reading and versus the corrected temperature reading. It also shows the percent loss, the corrected loss, and the percent evaporated versus the corrected temperature reading.

TABLE 8 Repeatability and Reproducibility for Group 1

Evaporated Point, %	Manual Repeatability[A]		Manual Reproducibility[A]		Automated Repeatability[A]		Automated Reproducibility[A]	
	°C	°F	°C	°F	°C	°F	°C	°F
IBP	3.3	6	5.6	10	3.9	7	7.2	13
5	$1.9+0.86S_C$	$3.4+0.86S_F$	$3.1+1.74S_C$	$5.6+1.74S_F$	$2.1+0.67S_C$	$3.8+0.67S_F$	$4.4+2.0S_C$	$7.9+2.0S_F$
10	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.7+0.67S_C$	$3.0+0.67S_F$	$3.3+2.0S_C$	$6.0+2.0S_F$
20	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$3.3+2.0S_C$	$6.0+2.0S_F$
30–70	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$2.6+2.0S_C$	$4.7+2.0S_F$
80	$1.2+0.86S_C$	$2.2+0.86S_F$	$2.0+1.74S_C$	$3.6+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$1.7+2.0S_C$	$3.0+2.0S_F$
90	$1.2+0.86S_C$	$2.2+0.86S_F$	$0.8+1.74S_C$	$1.4+1.74S_F$	$1.1+0.67S_C$	$2.0+0.67S_F$	$0.7+2.0S_C$	$1.2+2.0S_F$
95	$1.2+0.86S_C$	$2.2+0.86S_F$	$1.1+1.74S_C$	$1.9+1.74S_F$	$2.5+0.67S_C$	$4.5+0.67S_F$	$2.6+2.0S_C$	$4.7+2.0S_F$
FBP	3.9	7	7.2	13	4.4	8	8.9	16

[A] S_C or S_F is the average slope (or rate of change) calculated in accordance with 13.2.

13. Precision and Bias

13.1 *Precision*:

13.1.1 The precision of this test method has been determined by the statistical examination of interlaboratory test results obtained by 26 laboratories on 14 gasolines, by 4 laboratories on 8 samples of kerosine by the manual procedure, 3 laboratories on 6 samples of kerosine by the automated procedure, and 5 laboratories on 10 samples of diesel fuel by both the manual and automated procedures. Table A1.1 lists which tables and figures are to be used for the different fuel groups, distillation methods, and temperature scales.

13.1.2 The following terms are used in this section: (*1*) *r* = repeatability and (*2*) *R* = reproducibility. The value of any of these terms will depend upon whether the calculations were carried out in °C or °F.

13.2 *Slope or Rate of Change of Temperature*:

13.2.1 To determine the precision of a result, it is generally necessary to determine the slope or rate of change of the temperature at that particular point. This variable, denoted as S_C or S_F, is equal to the change in temperature, either in °C or in °F, respectively, per percent recovered or evaporated.

13.2.2 For Group 1 in the manual method and for all groups in the automated method, the precision of the IBP and EP does not require any slope calculation.

13.2.3 With the exception stated in 13.2.2 and in 13.2.4, the slope at any point during the distillation is calculated from the following equations, using the values shown in Table 7:

$$S_C \text{ (or } S_F) = (T_U - T_L) / (V_U - V_L) \quad (11)$$

where:

S_C = is the slope, °C/volume %,
S_F = is the slope, °F/volume %,
T_U = is the upper temperature, °C (or °F),
T_L = is the lower temperature, °C (or °F),
V_U = is the volume % recovered or evaporated corresponding to T_U,
V_L = is the volume % recovered or evaporated corresponding to T_L, and
V_{EP} = is the volume % recovered or evaporated corresponding to the end point.

13.2.4 In the event that the distillation end point occurs prior to the 95 % point, the slope at the end point is calculated as follows:

$$S_C \text{ (or } S_F) = (T_{EP} - T_{HR}) / (V_{EP} - V_{HR}) \quad (12)$$

where:

T_{EP} or T_{HR} is the temperature, in °C or °F at the percent volume recovered indicated by the subscript,

V_{EP} or V_{HR} is the volume % recovered.

13.2.4.1 The subscripts in Eq 12 refer to:

EP = end point
HR = highest reading, either 80 % of 90 %, prior to the end point.

13.2.5 For points between 10 to 85 % recovered which are not shown in Table 7, the slope is calculated as follows:

$$S_C \text{ (or } S_F) = 0.05\,(T_{(V+10)} - T_{(V-10)}) \quad (13)$$

13.2.6 For samples in Group 1, the precision data reported are based on slope values calculated from percent evaporated data.

13.2.7 For samples in Group 2, 3, and 4, the precision data reported (Table 8) are based on slope values calculated from percent recovered data.

13.2.8 When results are reported as volume % recovered, slope values for the calculation of precision are to be determined from percent recovered data; when results are reported as volume % evaporated slope values are to be determined from % evaporated data.

13.3 *Manual Method*:

13.3.1 *Repeatability*:

13.3.1.1 *GROUP 1*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values calculated from Table 9 in only one case in twenty.

13.3.1.2 *GROUPS 2, 3, and 4*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values calculated from the values in Table 9 in only one case in twenty.

13.3.2 *Reproducibility*:

TABLE 9 Repeatability and Reproducibility for Groups 2, 3 and 4 (Manual Method)

	Repeatability[A]		Reproducibility[A]	
	°C	°F	°C	°F
IBP	$1.0+0.35S_C$	$1.9+0.35S_F$	$2.8+0.93S_C$	$5.0+0.93S_F$
5—95 %	$1.0+0.41S_C$	$1.8+0.41S_F$	$1.8+1.33S_C$	$3.3+1.33S_F$
FBP	$0.7+0.36S_C$	$1.3+0.36S_F$	$3.1+0.42S_C$	$5.7+0.42S_F$
% volume at temperature reading	$0.7+0.92/S_C$	$0.7+1.66/S_F$	$1.5+1.78/S_C$	$1.53+3.20/S_F$

[A] Calculate S_C or S_F from 13.2.

13.3.2.1 *GROUP 1*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the normal and correct operation of this method, exceed the values calculated from Table 9 in only one case in twenty.[7]

13.3.2.2 *GROUPS 2, 3, and 4*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the normal and correct operation of this test method, exceed the values calculated from the data in Table 9 in only one case in twenty.[8]

13.4 *Automated Method*:

13.4.1 *Repeatability*:

13.4.1.1 *GROUP 1*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values calculated from Table 8 in only one case in twenty.

13.4.1.2 *GROUPS 2, 3, and 4*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values calculated from Table 10 in only one case in twenty.

13.4.2 *Reproducibility*:

13.4.2.1 *GROUP 1*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the normal and correct operation of this test method, exceed the values calculated from Table 8 in only one case in twenty.[7]

13.4.2.2 *GROUPS 2, 3, and 4*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the normal and correct operation of this test method, exceed the values calculated from Table 10 in only one case in twenty.

13.5 *Bias*:

13.5.1 *Bias*—Due to the use of total immersion thermometers, or temperature sensing systems designed to emulate them, the distillation temperatures in this test method are somewhat lower than the true temperatures. The amount of bias depends on the product being distilled and the thermometer used.

13.5.2 *Relative Bias*—There exists a bias between the empirical results of distillation properties obtained by this test method and the true boiling point distillation curve obtained by Test Method D 2892. The magnitude of this bias, and how it relates to test precision, has not been rigorously studied.

13.5.3 *Relative Bias*—An interlaboratory study[5] conducted in 2003 using manual and automated apparatus has concluded that there is no statistical evidence to suggest that there is a bias between manual and automated results.

14. Keywords

14.1 batch distillation; distillates; distillation; laboratory distillation; petroleum products

---

[7] Precision data obtained from RR study on both manual and automated D 86 units by North American and IP Laboratories.

[8] Table 9 has been derived from the nomographs in Figs. 6 and 7 in ASTM D 86–97.

TABLE 10 Repeatability and Reproducibility for Groups 2, 3 and 4 (Automated)

Collected, %	Repeatability[A]		Reproducibility[A]	
	°C	°F	°C	°F
IBP	3.5	6.3	8.5	15.3
2 %	3.5	6.3	$2.6 + 1.92S_C$	$4.7 + 1.92S_F$
5 %	$1.1 + 1.08S_C$	$2.0 + 1.08S_F$	$2.0 + 2.53S_C$	$3.6 + 2.53S_F$
10 %	$1.2 + 1.42S_C$	$2.2 + 1.42S_F$	$3.0 + 2.64S_C$	$5.4 + 2.64S_F$
20–70 %	$1.2 + 1.42S_C$	$2.2 + 1.42S_F$	$2.9 + 3.97S_C$	$5.2 + 3.97S_F$
80 %	$1.2 + 1.42S_C$	$2.2 + 1.42S_F$	$3.0 + 2.64S_C$	$5.4 + 2.64S_F$
90–95 %	$1.1 + 1.08S_C$	$2.0 + 1.08S_F$	$2.0 + 2.53S_C$	$3.6 + 2.53S_F$
FBP	3.5	6.3	10.5	18.9

[A] S_C or S_F is the average slope (or rate of change) calculated in accordance with 13.5.

ANNEXES

(Mandatory Information)

A1. REPEATABILITY AND REPRODUCIBILITY DEFINITION AIDS

A1.1 Table A1.1 is an aid for determining which repeatability and reproducibility table or section, is to be used.

TABLE A1.1 Summary of Aids for Definition of Repeatability and Reproducibility

Group	Method	Temperature Scale	Table or Section to Use	
			Repeatability	Reproducibility
1	Manual	°C	Table 8	Table 8
		°F	Table 8	Table 8
1	Automated	°C	Table 8	Table 8
		°F	Table 8	Table 8
2,3,4	Manual	°C	Table 9	Table 9
		°F	Table 9	Table 9
2,3,4	Automated	°C	Table 10	Table 10
		°F	Table 10	Table 10

A2. DETAILED DESCRIPTION OF APPARATUS

A2.1 *Distillation Flasks*—Flasks shall be of heat resistant glass, constructed to the dimensions and tolerances shown in Fig. A2.1 and shall otherwise comply with the requirements of Specification E 1405. Flask A (100 mL) may also be constructed with a ground glass joint, in which case the diameter of the neck shall be the same as the 125-mL flask.

NOTE A2.1—For tests specifying dry point, specially selected flasks with bottoms and walls of uniform thickness are desirable.

A2.2 *Condenser and Condenser Bath*—Typical types of condenser and condenser baths are illustrated in Figs. 1 and 2.

A2.2.1 The condenser shall be made of seamless noncorrosive metal tubing, 560 ± 5 mm in length, with an outside diameter of 14 mm and a wall thickness of 0.8 to 0.9 mm.

NOTE A2.2—Brass or stainless steel has been found to be a suitable material for this purpose.

A2.2.2 The condenser shall be set so that 393 ± 3 mm of the tube is in contact with the cooling medium, with 50 ± 3 mm outside the cooling bath at the upper end, and with 114 ± 3 mm outside at the lower end. The portion of the tube projecting at the upper end shall be set at an angle of 75 ± 3° with the vertical. The portion of the tube inside the condenser bath shall be either straight or bent in any suitable continuous smooth curve. The average gradient shall be 15 ± 1° with respect to the horizontal, with no 10-cm section having a gradient outside of the 15 ± 3° range. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and the lower end shall be cut off at an acute angle. Provisions shall be made to enable the flow of the distillate to run down the side of the receiving cylinder. This can be accomplished by using a drip-deflector, which is attached to the outlet of the tube. Alternatively, the lower portion of the condenser tube can be curved slightly backward to ensure contact with the wall of the receiving cylinder at a point 25 to 32 mm below the top of the receiving cylinder. Fig. A2.3 is a drawing of an acceptable configuration of the lower end of the condenser tube.

A2.2.3 The volume and the design of the bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single condenser bath may be used for several condenser tubes.

A2.3 *Metal Shield or Enclosure for Flask.* (Manual units only).

A2.3.1 *Shield for Gas Burner (see* Fig. 1*)*—The purpose of this shield is to provide protection for the operator and yet allow easy access to the burner and to the distillation flask during operation. A typical shield would be 480-mm high, 280-mm long and 200-mm wide, made of sheet metal of 0.8-mm thickness (22 gauge). The shield shall be provided with at least one window to observe the dry point at the end of the distillation.

A2.3.2 *Shield for Electric Heater (see* Fig. 2*)*—A typical shield would be 440-mm high, 200-mm long, and 200-mm wide, made of sheet metal of approximately 0.8-mm thickness (22 gauge) and with a window in the front side. The shield shall be provided with at least one window to observe the dry point at the end of the distillation.

A2.4 *Heat Source:*

A2.4.1 *Gas Burner* (see Fig. 1), capable of bringing over the first drop from a cold start within the time specified and of continuing the distillation at the specified rate. A sensitive manual control valve and gas pressure regulator to give complete control of heating shall be provided.

A2.4.2 *Electric Heater* (see Fig. 2), of low heat retention.

NOTE A2.3—Heaters, adjustable from 0 to 1000 W, have been found to be suitable for this purpose.

A2.5 *Flask Support:*

A2.5.1 *Type 1*—Use a Type 1 flask support with a gas burner (see Fig. 1). This support consists of either a ring support of the ordinary laboratory type, 100 mm or larger in diameter, supported on a stand inside the shield, or a platform adjustable from the outside of the shield. On this ring or platform is mounted a hard board made of ceramic or other heat-resistant material, 3 to 6 mm in thickness, with a central opening 76 to 100 mm in diameter, and outside line dimensions slightly smaller than the inside boundaries of the shield.

A2.5.2 *Type 2*—Use a Type 2 flask support assembly with electric heating (see Fig. 2 as one example). The assembly consists of an adjustable system onto which the electric heater is mounted with provision for placement of a flask support board (see A2.6) above the electric heater. The whole assembly is adjustable from the outside of the shield.

A2.6 *Flask Support Board*—The flask support board shall be constructed of ceramic or other heat-resistant material, 3 to 6 mm in thickness. Flask support boards are classified as A, B, or C, based on the size of the centrally located opening, the dimension of which is shown in Table 1. The flask support board shall be of sufficient dimension to ensure that thermal heat to the flask only comes from the central opening and that extraneous heat to the flask other than through the central opening is minimized. (**Warning**—Asbestos-containing materials shall not be used in the construction of the flask support board.)

A2.7 The flask support board can be moved slightly in different directions on the horizontal plane to position the distillation flask so that direct heat is applied to the flask only through the opening in this board. Usually, the position of the flask is set by adjusting the length of the side-arm inserted into the condenser.

A2.8 Provision shall be made for moving the flask support assembly vertically so that the flask support board is in direct contact with the bottom of the distillation flask during the distillation. The assembly is moved down to allow for easy mounting and removal of the distillation flask from the unit.

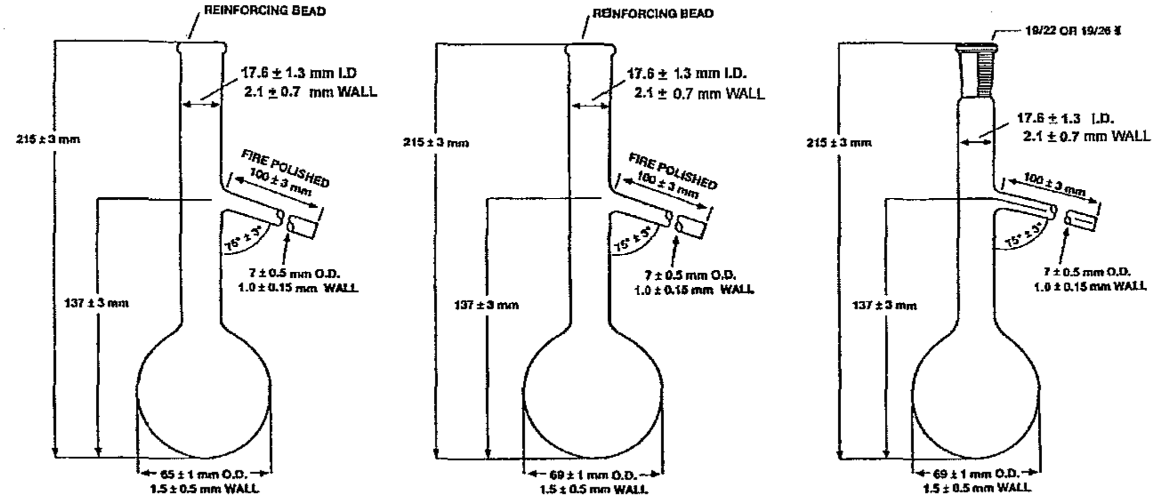
A2.9 *Receiving Cylinders*—The receiving cylinder shall have a capacity to measure and collect 100 mL. The shape of the base shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of 13° from the horizontal.

A2.9.1 *Manual Method*—The cylinder shall be graduated at intervals of 1 mL and have a graduation at the 100-mL mark. Construction details and tolerances for the graduated cylinder are shown in Fig. A2.4.

A2.9.2 *Automated Method*—The cylinder shall conform to the physical specifications described in Fig. A2.4, except that graduations below the 100-mL mark are permitted, as long as they do not interfere with the operation of the level follower. Receiving cylinders for use in automated units may also have a metal base.

A2.9.3 If required, the receiving cylinder shall be immersed during the distillation to above the 100-mL graduation line in a cooling liquid contained in a cooling bath, such as a tall-form beaker of clear glass or transparent plastic. Alternatively, the receiving cylinder may be placed in a thermostated bath air circulation chamber.

A2.10 *Residue Cylinder*—The graduated cylinder shall have a capacity of 5 or 10 mL, with graduations into 0.1 mL subdivisions, beginning at 0.1 mL. The top of the cylinder may be flared, the other properties shall conform to Specification E 1272.



Flask A, 100 mL　　Flask B, 125 mL　　Flask B, 125 mL

FIG. A2.1 Flask A, 100 mL, Flask B, 125 mL, and Flask B with Ground Glass Joint, 125 mL

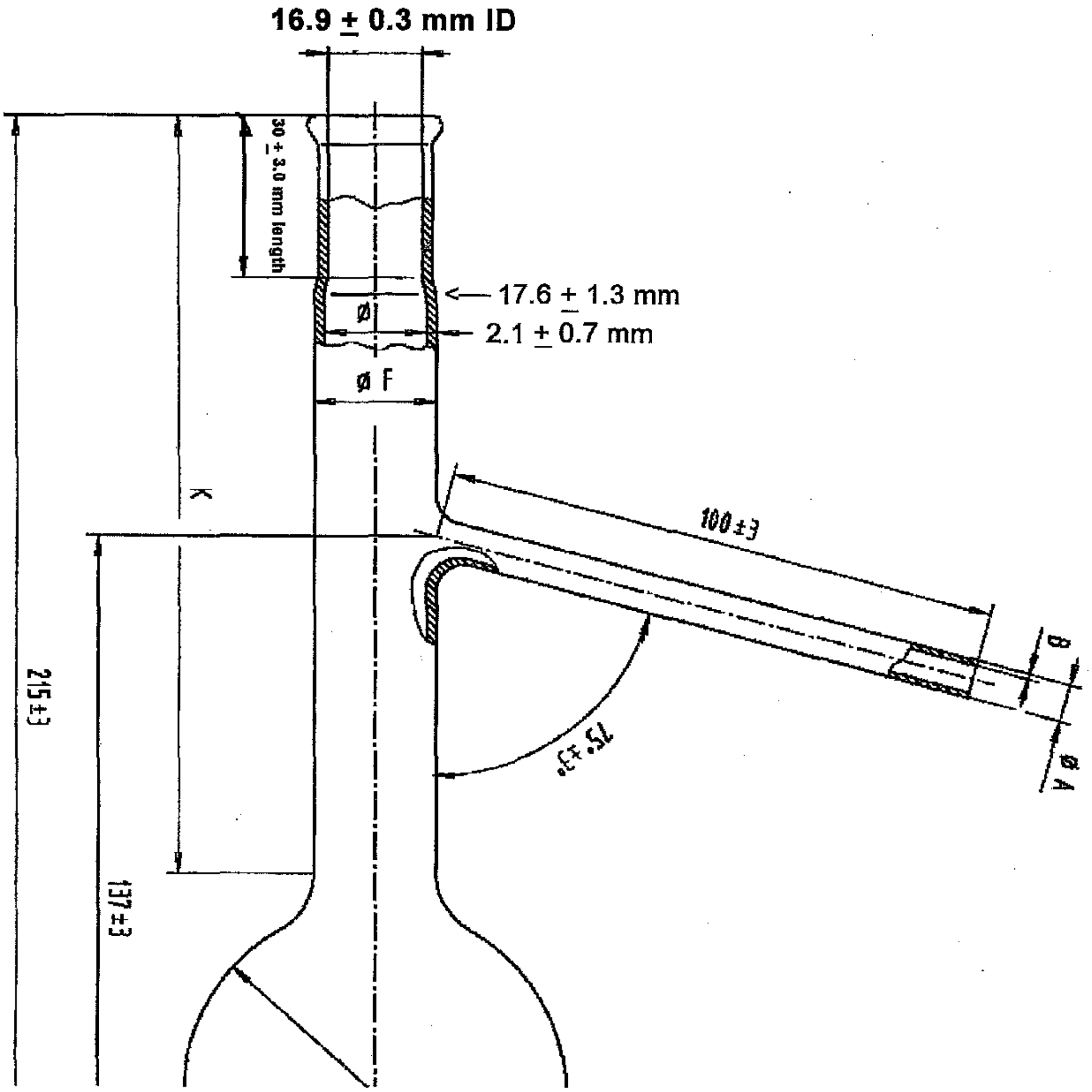
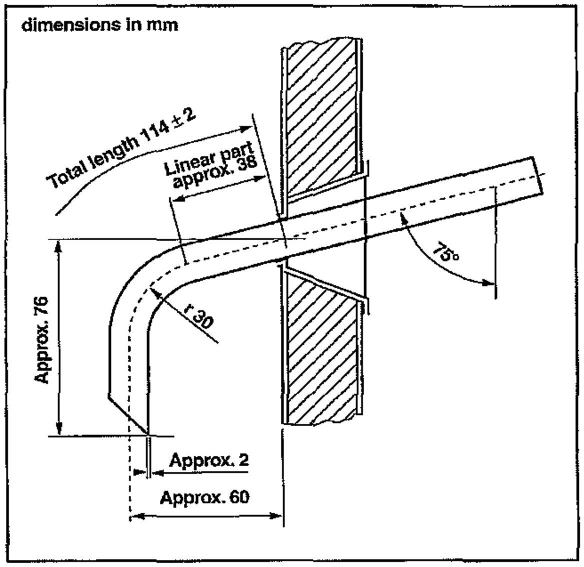
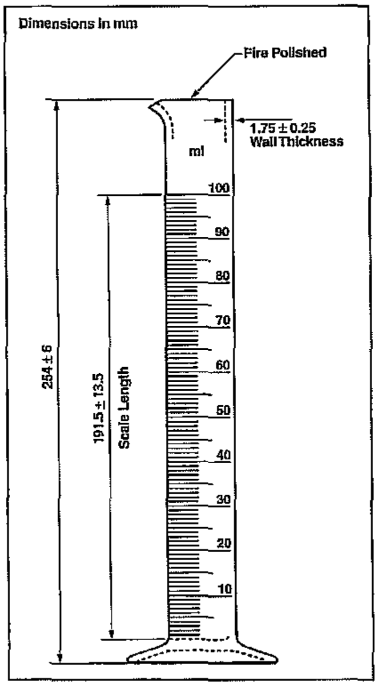


FIG. A2.2 Detail of Upper Neck Section



Lower End of Condenser Tube

FIG. A2.3 Lower End of Condenser Tube



NOTE—1 to 100 mL in 1 mL graduations; tolerance ± 1.0 mL.

FIG. A2.4 100 mL Graduated Cylinder

A3. DETERMINATION OF THE DIFFERENCE IN LAG TIME BETWEEN AN ELECTRONIC TEMPERATURE MEASUREMENT SYSTEM AND A MERCURY-IN-GLASS THERMOMETER

A3.1 The response time of an electronic temperature measuring device is inherently more rapid than that of a mercury-in-glass thermometer. The temperature measuring device assembly in general use, consisting of the sensor and its casing, or an electronic system and its associated software, or both, is so designed that the temperature measuring system will simulate the temperature lag of the mercury-in-glass thermometer.

A3.2 To determine the difference in lag time between such a temperature measuring system and a mercury-in-glass thermometer, analyze a sample such as gasoline, kerosine, jet fuel, or light diesel fuel with the electronic temperature measurement system in place and in accordance with the procedures described in this test method. In most cases this is the standard distillation step performed with an automated unit.

A3.2.1 Do not use a single pure compound, a very narrow boiling range product, or a synthetic blend of less than six compounds for this test.

A3.2.2 Best results are obtained with a sample that is typical of the sample load of the laboratory. Alternatively, use a full-range mixture with a 5 to 95 % boiling range of at least 100°C.

A3.3 Replace the electronic temperature measuring device with a low range or a high range mercury-in-glass thermometer, depending on the boiling range of the sample.

A3.4 Repeat the distillation with this thermometer, and manually record the temperature at the various percent recovered as described in 10.14.

A3.5 Calculate the values for the repeatability for the observed slope ($\Delta T/\Delta V$) for the different readings in the test.

A3.6 Compare the test data obtained using these two temperature measuring devices. The difference at any point shall be equal to, or less than, the repeatability of the method at that point. If this difference is larger, replace the electronic temperature measuring device or adjust the electronics involved, or both.

A4. PROCEDURE TO DETERMINE THE PERCENT EVAPORATED OR PERCENT RECOVERED AT A PRESCRIBED TEMPERATURE READING

A4.1 Many specifications require specific percentages evaporated or recovered at prescribed temperature readings, either as maxima, minima, or ranges. The procedures to determine these values are frequently designated by the terms Exxx or Rxxx, where xxx is the desired temperature.

NOTE A4.1—Regulatory standards on the certification of reformulated gasoline under the complex model procedure require the determination of E 200 and E 300, defined as the percent evaporated fuel at 93.3°C (200°F) and 148.9°C (300°F), respectively. E 158, the percent evaporated at a distillation temperature of 70°C (158°F), is also used in describing fuel volatility characteristics. Other typical temperatures are R 200 for kerosines and R 250 and R 350 for gas oils, where R 200, R 250, and R 350 are the percent recovered fuel at 200°C, 250°C, and 350°C, respectively.

A4.2 Determine the barometric pressure, and calculate the correction to the desired temperature reading using Eq 3, Eq 4, or Eq 5 for t = xxx°C (or t_f = xxx°F).

A4.2.1 *Manual Method*—Determine this correction to 0.5°C (1°F).

A4.2.2 *Automated Method*—Determine this correction to 0.1°C (0.2°F).

A4.3 Determine the expected temperature reading to yield xxx°C (or xxx°F) after the barometric correction. To obtain the expected value, add the absolute value of the calculated correction to the desired temperature if the barometric pressure is above 101.3 kPa. If the barometric pressure is below 101.3 kPa, subtract the absolute value of the calculated correction from the desired temperature.

A4.4 Perform the distillation, as described in Section 10, while taking into account A4.5 and A4.6.

A4.5 *Manual Distillation:*

A4.5.1 In the region between about 10°C below and 10°C above the desired expected temperature reading determined in A4.3 record the temperature reading in intervals of 1 volume %.

A4.5.2 If the intent of the distillation is to solely determine the value of Exxx or Rxxx, discontinue the distillation after at least another 2 mL of distillate have been collected. Otherwise, continue the distillation, as described in Section 10, and determine the observed loss, as described in 11.1.

A4.5.2.1 If the intent of the distillation is to determine the value of Exxx and the distillation was terminated after about 2 mL of distillate was collected beyond the desired temperature, allow the distillate to drain into the receiving graduate. Allow the contents of the flask to cool to below approximately 40°C and then drain its contents into the receiving graduate. Note the volume of product in the receiving graduate to the nearest 0.5 mL at 2 min intervals until two successive observations agree.

A4.5.2.2 The amount recovered in the receiving graduate is the percent recovery. Determine the amount of observed loss by subtracting the percent recovery from 100.0.

A4.6 *Automated Distillation:*

A4.6.1 In the region between about 10°C below and 10°C above the desired expected temperature reading determined in A4.3, collect temperature-volume data at 0.1 volume % intervals or less.

A4.6.2 Continue the distillation, as described in Section 10, and determine the percent loss, as described in 11.1.

A4.7 *Calculations:*

A4.7.1 *Manual Method*—If a volume % recovered reading is not available at the exact temperature calculated in A4.3, determine the percent recovered by interpolation between the two adjacent readings. Either the linear, as described in 11.6.1, or the graphical procedure, as described in 11.6.2, is permitted. The percent recovered is equal to Rxxx.

A4.7.2 *Automated Method*—Report the observed volume to 0.1 volume % corresponding to the temperature closest to the expected temperature reading. This is the percent recovered, or Rxxx.

A4.7.3 *Manual and Automated Methods*—To determine the value of Exxx, add the observed loss to the percent recovered, Rxxx, as determined in A4.7.1 or A4.7.2 and as described in Eq 9.

A4.7.3.1 As prescribed in 12.6, do not use the corrected loss.

A4.8 *Precision:*

A4.8.1 The statistical determination of the precision of the volume % evaporated or recovered at a prescribed temperature has not been directly measured in an interlaboratory program. It can be shown that the precision of the volume % evaporated or recovered at a prescribed temperature is equivalent to the precision of the temperature measurement at that point divided by the rate of change of temperature versus volume % evaporated or recovered. The estimation of precision becomes less precise at high slope values.

A4.8.2 Calculate the slope or rate of change in temperature reading, S_C(or S_F), as described in 13.2 and Eq 11 and using temperature values bracketing the desired temperature.

A4.8.3 Calculate the repeatability, *r*, or the reproducibility, *R*, from the slope, S_C (or S_F), and the data in Table 8, Table 9, or Table 10.

A4.8.4 Determine the repeatability or reproducibility, or both, of the volume % evaporated or recovered at a prescribed temperature from the following formulas:

$$^{r}\text{volume \%} = r/S_C\,(S_F) \tag{A4.1}$$

$$^{R}\text{volume \%} = R/S_C\,(S_F) \tag{A4.2}$$

where:

^{r}volume % = repeatability of the volume % evaporated or recovered,

^{R}volume % = reproducibility of the volume % evaporated or recovered,

r = repeatability of the temperature at the prescribed temperature at the observed percent distilled,

R = reproducibility of the temperature at the prescribed temperature at the observed percent distilled, and

$S_C(S_F)$ = rate of change in temperature reading in °C (°F) per the volume % evaporated or recovered.

A4.8.5 Examples on how to calculate the repeatability and the reproducibility are shown in Appendix X2.

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES ILLUSTRATING CALCULATIONS FOR REPORTING OF DATA

X1.1 The observed distillation data used for the calculation of the examples below are shown in the first three columns of Fig. X1.1.

X1.1.1 Temperature readings corrected to 101.3 kPa (760 mm Hg) pressure (see 11.3) are as follows:

$$\text{correction } (°C) = 0.0009\,(101.3 - 98.6)\,(273 + t_c) \tag{X1.1}$$

$$\text{correction } (°F) = 0.00012\,(760 - 740)\,(460 + t_f) \tag{X1.2}$$

X1.1.2 Loss correction to 101.3 kPa (see 11.4) are as follows. The data for the examples are taken from Fig. X1.1.

$$\text{corrected loss} = (0.5 + (4.7 - 0.5)/\{1 + (101.3 - 98.6)/8.0\} = 3.6 \tag{X1.3}$$

X1.1.3 Recovery correction to 101.3 kPa (see 11.4.1) are as follows:

$$\text{corrected recovery} = 94.2 + (4.7 - 3.6) = 95.3 \tag{X1.4}$$

X1.2 *Temperature Readings at Prescribed Percent Evaporated:*

X1.2.1 Temperature reading at 10 % evaporated (4.7 % observed loss = 5.3 % recovered) (see 11.6.1) are as follows:

$$T_{10E}\,(°C) = 33.7 + [(40.3 - 33.7)\,(5.3 - 5)/(10 - 5)] = 34.1°C \tag{X1.5}$$

$$T_{10E}\,(°F) = 92.7 + [(104.5 - 92.7)\,(5.3 - 5)/(10 - 5)] = 93.1°F \tag{X1.6}$$

X1.2.2 Temperature reading at 50 % evaporated (45.3 % recovered) (see 11.6.1) are as follows:

$$T_{50E}(°C) = 93.9 + [(108.9 - 93.9)\,(45.3 - 40)/(50 - 40)] = 101.9°C \tag{X1.7}$$

$$T_{50E}\,(°F) = 201 + [(228 - 201)\,(45.3 - 40)/(50 - 40)] = 215.3°F \tag{X1.8}$$

X1.2.3 Temperature reading at 90 % evaporated (85.3 % recovered) (see 11.6.1) are as follows:

$$T_{90E}\,(°C) = 181.6 + [(201.6 - 181.6)\,(85.3 - 85)/(90 - 85)\,] = 182.8°C \tag{X1.9}$$

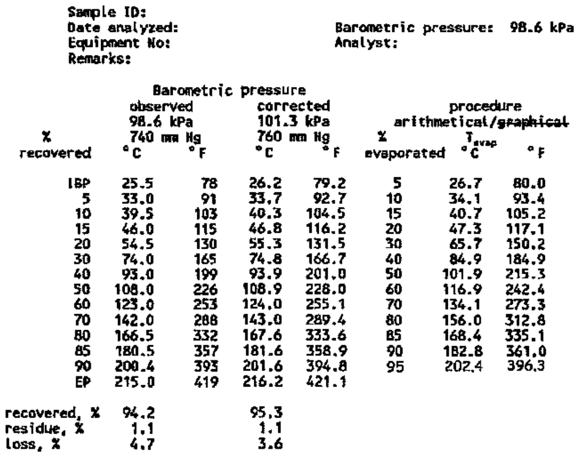
$$T_{90E}\,(°F) = 358.9 + [(394.8 - 358.9)(85.3 - 85)/(90 - 85)] = 361.0°F \tag{X1.10}$$

X1.2.4 Temperature reading at 90 % evaporated (85.3 % recovered) not corrected to 101.3 kPa pressure (see 11.6.1) are as follows:

$$T_{90E}\,(°C) = 180.5 + [(200.4 - 180.5)(85.3 - 85)/(90 - 85)] = 181.7°C \tag{X1.11}$$

$$T_{90E}\,(°F) = 357 + [(392 - 357)(85.3 - 85)/(90 - 85)] = 359.1°F \tag{X1.12}$$

NOTE X1.1—Results calculated from °C data may not correspond exactly to results calculated from °F data because of errors in rounding.



Sample ID:
Date analyzed:
Equipment No:
Remarks:

Barometric pressure: 98.6 kPa
Analyst:

	Barometric pressure				procedure		
	observed 98.6 kPa 740 mm Hg		corrected 101.3 kPa 760 mm Hg		arithmetical/~~graphical~~		
% recovered	°C	°F	°C	°F	% evaporated	T_{evap} °C	°F
IBP	25.5	78	26.2	79.2	5	26.7	80.0
5	33.0	91	33.7	92.7	10	34.1	93.4
10	39.5	103	40.3	104.5	15	40.7	105.2
15	46.0	115	46.8	116.2	20	47.3	117.1
20	54.5	130	55.3	131.5	30	65.7	150.2
30	74.0	165	74.8	166.7	40	84.9	184.9
40	93.0	199	93.9	201.0	50	101.9	215.3
50	108.0	226	108.9	228.0	60	116.9	242.4
60	123.0	253	124.0	255.1	70	134.1	273.3
70	142.0	288	143.0	289.4	80	156.0	312.8
80	166.5	332	167.6	333.6	85	168.4	335.1
85	180.5	357	181.6	358.9	90	182.8	361.0
90	200.4	393	201.6	394.8	95	202.4	396.3
EP	215.0	419	216.2	421.1			
recovered, %	94.2		95.3				
residue, %	1.1		1.1				
loss, %	4.7		3.6				

FIG. X1.1 Example of Test Report

X2. EXAMPLES OF CALCULATION OF REPEATABILITY AND REPRODUCIBILITY OF VOLUME % (RECOVERED OR EVAPORATED) AT A PRESCRIBED TEMPERATURE READING

X2.1 Some specifications require the reporting of the volume % evaporated or recovered at a prescribed temperature. Table X2.1 shows the distillation data of a Group 1 sample as obtained by an automated unit.

X2.2 *Example Calculation:*

X2.2.1 For a Group 1 sample exhibiting distillation characteristics as per Table X2.1, as determined by an automated unit, the reproducibility of the volume evaporated,^{R}volume %, at 93.3°C (200°F) is determined as follows:

X2.2.1.1 Determine first the slope at the desired temperature:

$$S_C\,\% = 0.1\,(T_{(20)} - T_{(10)}) \qquad (X2.1)$$

$$= 0.1\,(94 - 83)$$

$$= 1.1$$

$$S_F\,\% = 0.1\,(T_{(20)} - T_{(10)})$$

$$= 0.1\,(201 - 182)$$

$$= 1.9$$

X2.2.2 From Table 9, determine the value of R, the reproducibility at the observed percentage distilled. In this case, the observed percentage distilled is 18 % and

$$R = 3.3 + 2.0\,(S_C) \qquad (X2.2)$$

$$= 3.3 + 2.0 \times 1.1$$

$$= 5.5$$

$$R = 6.0 + 2.0\,(S_F)$$

$$= 6.0 + 2.0 \times 1.9$$

$$= 9.8$$

X2.2.3 From the calculated value of R, determine the value of volume, as described in A4.8.4.

$$R\text{ volume }\% = R/(S_C) \qquad (X2.3)$$

$$= 5.5/1.1$$

$$= 5.0$$

$$R\text{ volume }\% = R/(S_F)$$

$$= 9.8/1.9$$

$$= 5.1$$

TABLE X2.1 Distillation Data from a Group 1 Sample Automated Distillation

Distillation Point Recovered, mL	Temperature° C	Temperature °F	Volume (mL) Recovered at 93.3°C (200°F)
			18.0
10	84	183	
20	94	202	
30	103	217	
40	112	233	
Distillation Point Evaporated, mL	**Temperature° C**	**Temperature° F**	**Volume (mL0 Evaporated at 93.3°C (200°F)**
			18.4
10	83	182	
20	94	201	
30	103	217	
40	111	232	

X3. TABLES OF CORRECTED LOSS FROM MEASURED LOSS AND BAROMETRIC PRESSURE

X3.1 The table presented as Fig. X3.1 can be used to determine the corrected loss from the measured loss and the barometric pressure in kPa.

X3.2 The table presented as Fig. X3.2 can be used to determine the corrected loss from the measured loss and the barometric pressure in mm Hg.

Barometric Pressure, kPa

from	76.1	80.9	84.5	87.9	89.6	91.5	93.1	94.1	95.5	96.4	97.2	97.9	98.4	98.9	99.5	100.0	100.4	100.8	101.2	101.5	102.0	102.4	102.8	103.2
through	80.8	84.4	87.2	89.5	91.4	93.0	94.0	95.4	96.3	97.1	97.8	98.3	98.8	99.4	99.9	100.3	100.7	101.1	101.4	101.9	102.3	102.7	103.1	103.5
Observed Loss	/-- Corrected Loss ------>																							
Units																								
0	0.37	0.35	0.33	0.31	0.29	0.27	0.25	0.23	0.20	0.18	0.16	0.14	0.13	0.11	0.09	0.06	0.04	0.02	−0.00	−0.02	−0.06	−0.09	−0.13	−0.17
1	0.63	0.65	0.67	0.69	0.71	0.73	0.75	0.78	0.80	0.82	0.84	0.86	0.87	0.89	0.92	0.94	0.96	0.98	1.00	1.03	1.06	1.09	1.13	1.17
2	0.89	0.95	1.01	1.08	1.14	1.20	1.26	1.33	1.40	1.46	1.52	1.57	1.62	1.68	1.75	1.81	1.87	1.94	2.00	2.08	2.17	2.27	2.38	2.51
3	1.15	1.25	1.36	1.46	1.57	1.67	1.77	1.88	1.99	2.09	2.19	2.28	2.37	2.47	2.58	2.69	2.79	2.90	3.00	3.13	3.29	3.45	3.63	3.84
4	1.41	1.56	1.70	1.84	1.99	2.14	2.28	2.43	2.59	2.73	2.87	3.00	3.12	3.26	3.41	3.56	3.70	3.85	4.00	4.18	4.40	4.63	4.89	5.18
5	1.68	1.86	2.04	2.23	2.42	2.61	2.79	2.98	3.19	3.37	3.55	3.71	3.87	4.05	4.25	4.44	4.62	4.81	5.00	5.23	5.51	5.81	6.14	6.52
6	1.94	2.16	2.39	2.61	2.84	3.08	3.30	3.53	3.78	4.01	4.23	4.42	4.62	4.84	5.08	5.31	5.53	5.77	6.00	6.28	6.63	6.99	7.40	7.86
7	2.20	2.46	2.73	3.00	3.27	3.55	3.80	4.08	4.36	4.65	4.90	5.14	5.37	5.63	5.91	6.18	6.44	6.73	7.00	7.33	7.74	8.17	8.65	9.20
8	2.46	2.76	3.07	3.38	3.70	4.02	4.31	4.63	4.98	5.28	5.58	5.85	6.12	6.41	6.74	7.06	7.36	7.69	8.00	8.38	8.86	9.35	9.90	10.53
9	2.72	3.07	3.41	3.76	4.12	4.49	4.82	5.18	5.57	5.92	6.26	6.56	6.87	7.20	7.57	7.93	8.27	8.65	9.00	9.43	9.97	10.53	11.16	11.87
10	2.98	3.37	3.76	4.15	4.55	4.96	5.33	5.73	6.17	6.56	6.94	7.28	7.62	7.99	8.41	8.81	9.19	9.60	10.00	10.48	11.08	11.71	12.41	13.21
11	3.24	3.67	4.10	4.53	4.97	5.43	5.84	6.28	6.77	7.20	7.61	7.99	8.37	8.78	9.24	9.68	10.10	10.56	11.00	11.53	12.20	12.89	13.67	14.55
12	3.50	3.97	4.44	4.92	5.40	5.90	6.35	6.83	7.36	7.84	8.29	8.71	9.12	9.57	10.07	10.56	11.02	11.52	12.00	12.59	13.31	14.07	14.92	15.89
13	3.76	4.27	4.78	5.30	5.83	6.36	6.86	7.39	7.96	8.47	8.97	9.42	9.86	10.36	10.90	11.43	11.93	12.48	13.00	13.64	14.43	15.25	16.17	17.22
14	4.03	4.58	5.13	5.69	6.25	6.83	7.36	7.94	8.56	9.11	9.64	10.13	10.61	11.15	11.74	12.31	12.85	13.44	14.00	14.69	15.54	16.43	17.43	18.56
15	4.29	4.88	5.47	6.07	6.68	7.30	7.87	8.49	9.15	9.75	10.32	10.85	11.36	11.93	12.57	13.18	13.76	14.40	15.00	15.74	16.66	17.61	18.68	19.90
16	4.55	5.16	5.81	6.45	7.10	7.77	8.38	9.04	9.75	10.39	11.00	11.56	12.11	12.72	13.40	14.06	14.68	15.36	16.00	16.79	17.77	18.79	19.94	21.24
17	4.81	5.48	6.16	6.84	7.53	8.24	8.89	9.59	10.35	11.03	11.68	12.27	12.86	13.51	14.23	14.93	15.59	16.31	17.00	17.84	18.86	19.97	21.19	22.58
18	5.07	5.78	6.50	7.22	7.96	8.71	9.40	10.14	10.94	11.66	12.35	12.99	13.61	14.30	15.07	15.80	16.50	17.27	18.00	18.89	20.00	21.15	22.44	23.91
19	5.33	6.08	6.84	7.61	8.38	9.18	9.91	10.69	11.54	12.30	13.03	13.70	14.36	15.09	15.90	16.68	17.42	18.23	19.00	19.94	21.11	22.39	23.70	25.25
20	5.59	6.39	7.18	7.99	8.81	9.65	10.41	11.24	12.14	12.94	13.71	14.41	15.11	15.88	16.73	17.65	18.33	19.19	20.00	20.99	22.23	23.51	24.95	26.59
Tenths																								
0.0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.1	0.03	0.03	0.03	0.04	0.04	0.05	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.08	0.08	0.09	0.09	0.10	0.10	0.11	0.11	0.12	0.13	0.13
0.2	0.05	0.06	0.07	0.08	0.09	0.09	0.10	0.11	0.12	0.13	0.14	0.14	0.15	0.16	0.17	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.27
0.3	0.08	0.09	0.10	0.12	0.13	0.14	0.15	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.26	0.27	0.29	0.30	0.32	0.33	0.35	0.38	0.40
0.4	0.10	0.12	0.14	0.15	0.17	0.19	0.20	0.22	0.24	0.26	0.27	0.29	0.30	0.32	0.33	0.35	0.37	0.38	0.40	0.42	0.45	0.47	0.50	0.54
0.5	0.13	0.15	0.17	0.19	0.21	0.23	0.25	0.28	0.30	0.32	0.34	0.36	0.37	0.39	0.42	0.44	0.46	0.48	0.50	0.53	0.56	0.59	0.63	0.67
0.6	0.16	0.18	0.21	0.23	0.26	0.28	0.31	0.33	0.36	0.38	0.41	0.43	0.45	0.47	0.50	0.52	0.55	0.58	0.60	0.63	0.67	0.71	0.75	0.80
0.7	0.18	0.21	0.24	0.27	0.30	0.33	0.36	0.39	0.42	0.45	0.47	0.50	0.52	0.55	0.58	0.61	0.64	0.67	0.70	0.74	0.78	0.83	0.88	0.94
0.8	0.21	0.24	0.27	0.31	0.34	0.38	0.41	0.44	0.48	0.51	0.54	0.57	0.60	0.63	0.67	0.70	0.73	0.77	0.80	0.84	0.89	0.94	1.00	1.07
0.9	0.24	0.27	0.31	0.35	0.38	0.42	0.46	0.50	0.54	0.57	0.61	0.64	0.67	0.71	0.75	0.79	0.82	0.86	0.90	0.95	1.00	1.06	1.13	1.20

FIG. X3.1 Corrected Loss from Observed Loss and Barometric Pressure kPa

Barometric Pressure, mm Hg.

from	571	607	634	655	672	686	698	706	716	723	729	734	738	742	746	750	753	756	759	762	765	768	771	774
through	606	633	654	671	685	697	705	715	722	728	733	737	741	745	749	752	755	758	761	764	767	770	773	776
Observed Loss	/-- Corrected Loss ----->																							
Units																								
0	0.37	0.35	0.33	0.31	0.29	0.27	0.25	0.23	0.20	0.18	0.16	0.14	0.13	0.11	0.09	0.07	0.05	0.02	−0.00	−0.03	−0.06	−0.09	−0.13	−0.17
1	0.63	0.65	0.67	0.69	0.71	0.73	0.75	0.77	0.80	0.82	0.84	0.86	0.87	0.89	0.91	0.93	0.95	0.98	1.00	1.03	1.06	1.09	1.13	1.17
2	0.89	0.95	1.01	1.07	1.14	1.20	1.26	1.32	1.39	1.45	1.51	1.57	1.62	1.68	1.74	1.80	1.86	1.93	2.00	2.08	2.17	2.27	2.38	2.50
3	1.15	1.25	1.36	1.46	1.56	1.67	1.77	1.87	1.99	2.09	2.19	2.28	2.36	2.46	2.57	2.67	2.77	2.88	3.00	3.13	3.28	3.44	3.63	3.83
4	1.41	1.55	1.70	1.84	1.99	2.14	2.27	2.42	2.58	2.72	2.86	2.99	3.11	3.25	3.40	3.54	3.68	3.83	4.00	4.19	4.39	4.62	4.88	5.17
5	1.67	1.86	2.04	2.22	2.41	2.61	2.78	2.97	3.18	3.36	3.54	3.70	3.86	4.03	4.23	4.41	4.59	4.79	5.00	5.24	5.50	5.80	6.13	6.50
6	1.93	2.16	2.38	2.61	2.84	3.07	3.29	3.52	3.77	3.99	4.21	4.41	4.60	4.82	5.05	5.28	5.50	5.74	6.00	6.29	6.61	6.97	7.38	7.84
7	2.19	2.46	2.72	2.99	3.26	3.54	3.79	4.07	4.36	4.63	4.88	5.12	5.35	5.60	5.88	6.15	6.41	6.69	7.00	7.34	7.72	8.15	8.63	9.17
8	2.46	2.76	3.07	3.37	3.69	4.01	4.30	4.62	4.96	5.27	5.56	5.83	6.09	6.38	6.71	7.02	7.32	7.64	8.00	8.40	8.84	9.33	9.88	10.50
9	2.72	3.06	3.41	3.76	4.11	4.46	4.81	5.17	5.55	5.90	6.23	6.54	6.84	7.17	7.54	7.89	8.23	8.60	9.00	9.45	9.95	10.50	11.13	11.84
10	2.98	3.36	3.75	4.14	4.54	4.94	5.31	5.71	6.15	6.54	6.91	7.25	7.58	7.95	8.37	8.76	9.14	9.55	10.00	10.50	11.06	11.68	12.38	13.17
11	3.24	3.66	4.09	4.52	4.96	5.41	5.82	6.26	6.74	7.17	7.58	7.96	8.33	8.74	9.19	9.63	10.05	10.50	11.00	11.56	12.17	12.86	13.63	14.51
12	3.50	3.96	4.43	4.91	5.39	5.88	6.33	6.81	7.34	7.81	8.26	8.67	9.07	9.52	10.02	10.50	10.96	11.46	12.00	12.61	13.28	14.03	14.86	15.84
13	3.76	4.27	4.78	5.29	5.81	6.35	6.83	7.36	7.93	8.44	8.93	9.38	9.82	10.31	10.85	11.37	11.87	12.41	13.00	13.66	14.39	15.21	16.13	17.17
14	4.02	4.57	5.12	5.67	6.24	6.82	7.34	7.91	8.53	9.08	9.61	10.09	10.57	11.09	11.68	12.24	12.78	13.36	14.00	14.71	15.51	16.39	17.38	18.51
15	4.28	4.87	5.46	6.06	6.66	7.28	7.85	8.46	9.12	9.71	10.28	10.80	11.31	11.88	12.51	13.11	13.68	14.31	15.00	15.77	16.62	17.57	18.63	19.84
16	4.54	5.17	5.80	6.44	7.09	7.75	8.35	9.01	9.72	10.35	10.95	11.51	12.06	12.66	13.33	13.98	14.59	15.27	16.00	16.82	17.73	18.74	19.88	21.18
17	4.80	5.47	6.14	6.82	7.51	8.22	8.86	9.56	10.31	10.98	11.63	12.22	12.80	13.45	14.16	14.85	15.50	16.22	17.00	17.87	18.84	19.92	21.13	22.51
18	5.06	5.77	6.49	7.21	7.94	8.69	9.37	10.11	10.91	11.62	12.30	12.93	13.55	14.23	14.99	15.72	16.41	17.17	18.00	18.93	19.95	21.10	22.38	23.84
19	5.32	6.07	6.83	7.59	8.36	9.15	9.88	10.65	11.50	12.25	12.98	13.64	14.29	15.02	15.82	16.59	17.32	18.12	19.01	19.98	21.06	22.27	23.64	25.18
20	5.58	6.37	7.17	7.97	8.79	9.62	10.38	11.20	12.09	12.89	13.65	14.35	15.04	15.80	16.64	17.46	18.23	19.08	20.01	21.03	22.17	23.45	24.89	26.51
Tenths																								
0.0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.1	0.03	0.03	0.03	0.04	0.04	0.05	0.05	0.05	0.06	0.06	0.07	0.07	0.07	0.08	0.08	0.09	0.09	0.10	0.10	0.11	0.11	0.12	0.13	0.13
0.2	0.05	0.06	0.07	0.08	0.08	0.09	0.10	0.11	0.12	0.13	0.13	0.14	0.15	0.16	0.17	0.17	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.27
0.3	0.08	0.09	0.10	0.11	0.13	0.14	0.15	0.16	0.18	0.19	0.20	0.21	0.22	0.24	0.25	0.26	0.27	0.29	0.30	0.32	0.33	0.35	0.38	0.40
0.4	0.10	0.12	0.14	0.15	0.17	0.19	0.20	0.22	0.24	0.25	0.27	0.28	0.30	0.31	0.33	0.35	0.36	0.38	0.40	0.42	0.44	0.47	0.50	0.53
0.5	0.13	0.15	0.17	0.19	0.21	0.23	0.25	0.27	0.30	0.32	0.34	0.36	0.37	0.39	0.41	0.43	0.45	0.48	0.50	0.53	0.56	0.59	0.63	0.67
0.6	0.16	0.18	0.21	0.23	0.25	0.28	0.30	0.33	0.36	0.38	0.40	0.43	0.45	0.47	0.50	0.52	0.55	0.57	0.60	0.63	0.67	0.71	0.75	0.80
0.7	0.18	0.21	0.24	0.27	0.30	0.33	0.35	0.38	0.42	0.44	0.47	0.50	0.52	0.55	0.58	0.61	0.64	0.67	0.70	0.74	0.78	0.82	0.88	0.93
0.8	0.21	0.24	0.27	0.31	0.34	0.37	0.41	0.44	0.48	0.51	0.54	0.57	0.60	0.63	0.66	0.70	0.73	0.76	0.80	0.84	0.89	0.94	1.00	1.07
0.9	0.23	0.27	0.31	0.34	0.38	0.42	0.46	0.49	0.54	0.57	0.61	0.64	0.67	0.71	0.75	0.78	0.82	0.86	0.90	0.95	1.00	1.06	1.13	1.20

FIG. X3.2 Corrected Loss from Observed Loss and Barometric Pressure mm Hg

X4. PROCEDURE TO EMULATE THE EMERGENT STEM ERROR OF A MERCURY-IN-GLASS THERMOMETER

X4.1 When an electronic or other sensor without an emergent stem error is used, the output of this sensor or the associated data system should emulate the output of a mercury-in-glass thermometer. Based on information supplied by four manufacturers of automated Test Method D 86 equipment, the averaged equations shown in X4.2 and X4.3 have been reported to be in use.

X4.1.1 The equations shown in X4.2 have limited applicability and are shown for information purposes only. In addition to the correction for the emergent stem, the electronic sensor and associated data system will also have to emulate the lag in response time observed for mercury-in-glass thermometers.

X4.2 When a low range thermometer would have been used, no stem correction is to be applied below 20°C. Above this temperature, the correction is calculated using the following formula:

$$ASTM\ 7C\ T_{elr} = T_t - 0.000162 \times (T_t - 20°C)^2 \qquad (X4.1)$$

X4.3 When a high range thermometer would have been used, no stem correction is to be applied below 35°C. Above this temperature the correction is calculated using the following formula:

$$ASTM\ 8C\ T_{ehr} = T_t - 0.000131 \times (T_t - 35°C)^2 \qquad (X4.2)$$

where:

T_{elr} = emulated temperature in °C for low range thermometers,

T_{ehr} = emulated temperature in °C for high range thermometers, and

T_t = true temperature in °C.

X5. EXPLANATORY REPORT FORMS

X5.1 Fig. X5.1 and Fig. X5.2 show report forms.

"Percent Recovered" Report Form

Date:	
Time	
Operator:	

Ambient temperature (°C)	
Atmospheric pressure (kPa)	
Condenser temperature (°C)	
Temperature of the bath around receiving cylinder (°C)	

	Percent Recovered	Corrected Temperature Reading (°C)	Time or mL / min
	IBP		
	5		
	10		
	15		
	20		
	25		
	30		
	35		
	40		
	45		
	50		
	55		
	60		
	65		
	70		
	75		
	80		
	85		
	90		
5 ml residue			
	95		
FBP			

Percent Recovery		
Percent Residue		
Percent Total Recovery		
Percent Loss		Corrected Loss
Corrected Percent Recovery		Corrected Total Recovery

Comments:

Ambient temperature at the start of the test

Ambient barometric pressure at the start of the test

Volume of condensate observed in the receiving cylinder at any point in the distillation, expressed as a percentage of the charge volume, in connection with simultaneous temperature reading

Temperature measuring device readings which are corrected to 101,3 kPa barometric pressure

Group 1, 2 & 3:	5 to 10 minutes
Group 4:	5 to 15 minutes

Group 1 & 2:	60 to 100 seconds

4 to 5 ml / min uniform avrage rate from 5% recovered to 5 ml in flask

Volume of condensate observed in the receiving cylinder when the 5ml conditions are reached

Volume of condensate observed in the receiving cylinder when the final boiling point is observed

Maximum percent recovered

Volume of residue in the flask expressed as a percentage of the charge volume

Combined Percent Recovery and Percent Residue in the flask

Time from 5 ml in flask to FBP =< 5 minutes

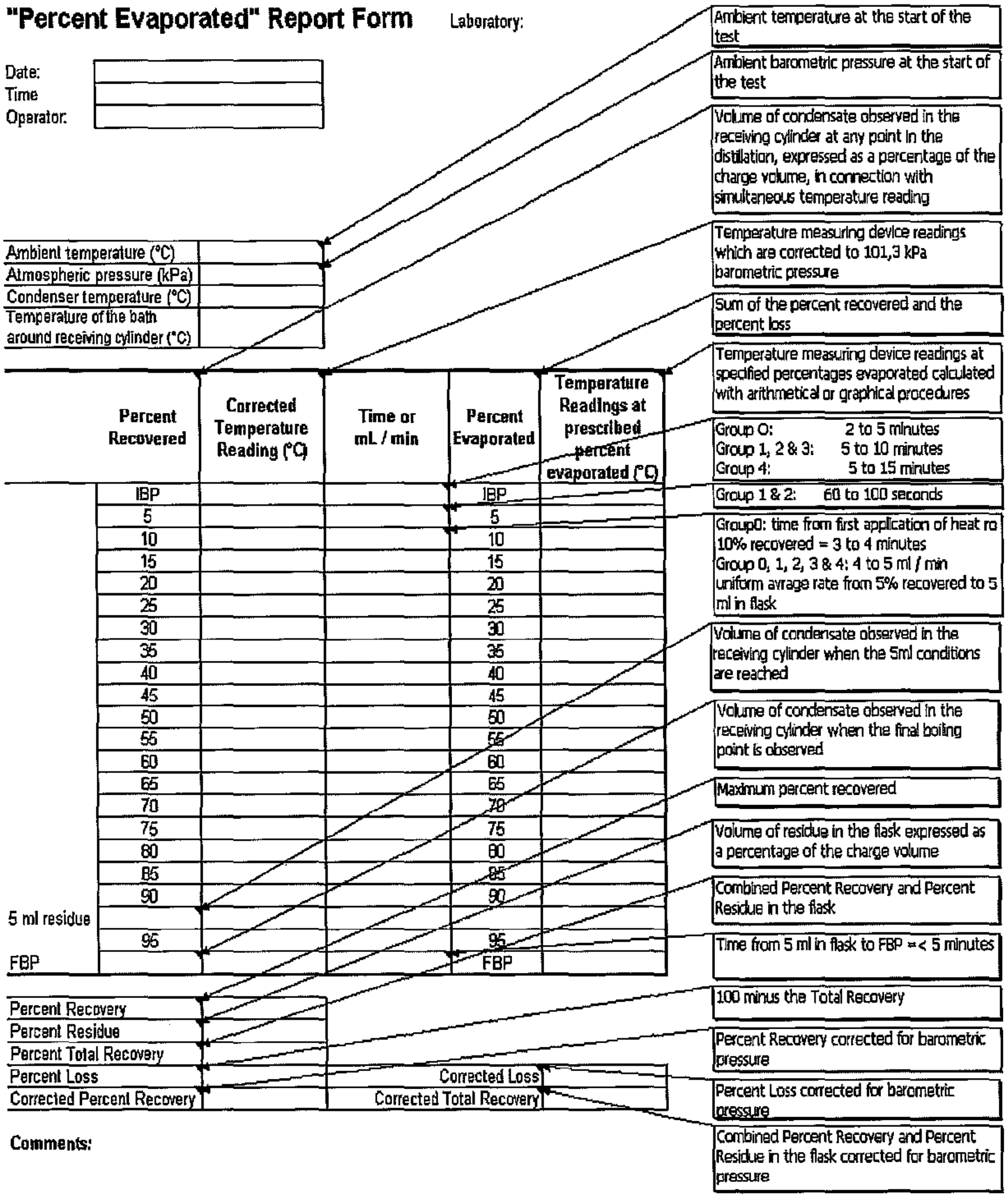
100 minus the Total Recovery

Percent Recovery corrected for barometric pressure

Percent Loss corrected for barometric pressure

Combined Percent Recovery and Percent Residue in the flask corrected for barometric pressure

FIG. X5.1 Percent Recovered Report Form



"Percent Evaporated" Report Form

Laboratory:

Date:	
Time	
Operator:	

Ambient temperature (°C)	
Atmospheric pressure (kPa)	
Condenser temperature (°C)	
Temperature of the bath around receiving cylinder (°C)	

	Percent Recovered	Corrected Temperature Reading (°C)	Time or mL / min	Percent Evaporated	Temperature Readings at prescribed percent evaporated (°C)
	IBP			IBP	
	5			5	
	10			10	
	15			15	
	20			20	
	25			25	
	30			30	
	35			35	
	40			40	
	45			45	
	50			50	
	55			55	
	60			60	
	65			65	
	70			70	
	75			75	
	80			80	
	85			85	
	90			90	
5 ml residue					
	95			95	
FBP				FBP	

Percent Recovery			
Percent Residue			
Percent Total Recovery			
Percent Loss		Corrected Loss	
Corrected Percent Recovery		Corrected Total Recovery	

Comments:

Ambient temperature at the start of the test

Ambient barometric pressure at the start of the test

Volume of condensate observed in the receiving cylinder at any point in the distillation, expressed as a percentage of the charge volume, in connection with simultaneous temperature reading

Temperature measuring device readings which are corrected to 101,3 kPa barometric pressure

Sum of the percent recovered and the percent loss

Temperature measuring device readings at specified percentages evaporated calculated with arithmetical or graphical procedures

Group 0:	2 to 5 minutes
Group 1, 2 & 3:	5 to 10 minutes
Group 4:	5 to 15 minutes

Group 1 & 2: 60 to 100 seconds

Group0: time from first application of heat ro 10% recovered = 3 to 4 minutes
Group 0, 1, 2, 3 & 4: 4 to 5 ml / min uniform avrage rate from 5% recovered to 5 ml in flask

Volume of condensate observed in the receiving cylinder when the 5ml conditions are reached

Volume of condensate observed in the receiving cylinder when the final boiling point is observed

Maximum percent recovered

Volume of residue in the flask expressed as a percentage of the charge volume

Combined Percent Recovery and Percent Residue in the flask

Time from 5 ml in flask to FBP =< 5 minutes

100 minus the Total Recovery

Percent Recovery corrected for barometric pressure

Percent Loss corrected for barometric pressure

Combined Percent Recovery and Percent Residue in the flask corrected for barometric pressure

FIG. X5.2 Percent Evaporated Report Form

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D 86–05) that may impact the use of this standard. (Approved Jan. 15, 2007.)

(*1*) Deleted "natural gasolines" from 1.1.
(2) Deleted "Group 0" from the entire standard.
(*3*) Added Fig. 6.

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue, (D 86–04b), that may impact the use of this standard. (Approved July 1, 2005.)

(*1*) Replaced Table 4 with new values.
(2) Revised 9.1.2-9.1.2.2, 9.1.5, and Notes 9-11.
(*3*) Added 13.5.3 and footnote reference to the research report.
(*4*) Added Appendix X5, and cross-reference in Section 12.1.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D129: Standard Test Method for Sulfur in Petroleum Products (General Bomb Method)

CFR Section(s): 40 CFR 60.106(j)(2)

Standards Body: American Society for Testing and Materials

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 129 – 95

Designation: 61/99

An American National Standard
British Standard 4454

Standard Test Method for Sulfur in Petroleum Products (General Bomb Method)[1]

This standard is issued under the fixed designation D 129; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This test method has been adopted for use by government agencies to replace Method 5202 of Federal Test Method No. 791b

1. Scope

1.1 This test method covers the determination of sulfur in petroleum products, including lubricating oils containing additives, additive concentrates, and lubricating greases that cannot be burned completely in a wick lamp. The test method is applicable to any petroleum product sufficiently low in volatility that it can be weighed accurately in an open sample boat and containing at least 0.1 % sulfur.

NOTE 1—This test method is not applicable to samples containing elements that give residues, other than barium sulfate, which are insoluble in dilute hydrochloric acid and would interfere in the precipitation step. These interfering elements include iron, aluminum, calcium, silicon, and lead which are sometimes present in greases, lube oil additives, or additive oils. Other acid insoluble materials that interfere are silica, molybdenum disulfide, asbestos, mica, etc. The test method is not applicable to used oils containing wear metals, and lead or silicates from contamination. Samples that are excluded can be analyzed by Test Method D 1552.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See 3.2 for specific precautionary directions incorporated in the test method.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)[3]

E 144 Practice for Safe Use of Oxygen Combustion Bombs[4]

3. Summary of Test Method

3.1 The sample is oxidized by combustion in a bomb containing oxygen under pressure. The sulfur, as sulfate in the bomb washings, is determined gravimetrically as barium sulfate.

3.2 **Warning—** *Strict adherence to all of the provisions prescribed hereafter ensures against explosive rupture of the bomb, or a blow-out, provided the bomb is of proper design and construction and in good mechanical condition. It is desirable, however, that the bomb be enclosed in a shield of steel plate at least 13 mm thick, or equivalent protection be provided against unforseeable contingencies.*

4. Apparatus and Materials

4.1 *Bomb,*[5,6] having a capacity of not less than 300 mL, so constructed that it will not leak during the test and that quantitative recovery of the liquids from the bomb may be achieved readily. The inner surface of the bomb may be made of stainless steel or any other material that will not be affected by the combustion process or products. Materials used in the bomb assembly, such as the head gasket and lead-wire insulation, shall be resistant to heat and chemical action, and shall not undergo any reaction that will affect the sulfur content of the liquid in the bomb.

4.2 *Sample Cup*, platinum, 24 mm in outside diameter at the bottom, 27 mm in outside diameter at the top, 12 mm in height outside, and weighing 10 to 11 g.

4.3 *Firing Wire*, platinum, No. 26 B & S gage, 0.41 mm (16 thou), 27 SWG, or equivalent.

NOTE 2—**Caution:** The switch in the ignition circuit shall be of a type which remains open, except when held in closed position by the operator.

4.4 *Ignition Circuit*, capable of supplying sufficient current to ignite the cotton wicking or nylon thread without melting the wire. The current shall be drawn from a step-down transformer or from a suitable battery.

4.5 *Cotton Wicking or Nylon Sewing Thread*, white.

---

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

Current edition approved Aug. 15, 1995. Published October 1995. Originally published as D 129 – 22. Last previous edition D 129 – 91.

This test method was adopted as a joint ASTM-IP standard in 1964.

In the IP, this test method is under the jurisdiction of the Standardization Committee.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Criteria for judging the acceptability of new and used oxygen combustion bombs are described in Practice E 144.

[6] A bomb conforming to the test specifications in IP Standard IP 12 is suitable.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall mean water as defined by Type II or III of Specification D 1193.

5.3 *Barium Chloride Solution* (85 g/litre)—Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in distilled water and dilute to 1 liter.

5.4 *Bromine Water (saturated).*

5.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.6 *Oxygen*, free of combustible material and sulfur compounds, available at a pressure of 41 kgf/cm^2 (40 atm).

5.7 *Sodium Carbonate Solution* (50 g/litre)—Dissolve 135 g of sodium carbonate decahydrate ($Na_2CO_3 \cdot 10H_2O$) or its equivalent weight in distilled water and dilute to 1 litre.

5.8 *White Oil, USP, or Liquid Paraffin*, BP, *or equivalent.*

6. Procedure

6.1 *Preparation of Bomb and Sample*— Cut a piece of firing wire 100 mm in length. Coil the middle section (about 20 mm) and attach the free ends to the terminals. Arrange the coil so that it will be above and to one side of the sample cup. Insert between two loops of the coil a wisp of cotton or nylon thread of such length that one end will extend into the sample cup. Place about 5 mL of Na_2CO_3 solution in the bomb (Note 3) and rotate the bomb in such a manner that the interior surface is moistened by the solution. Introduce into the sample cup the quantities of sample and white oil (Note 5 and Note 6) specified in the following table, weighing the sample to the nearest 0.2 mg (when white oil is used, stir the mixture with a short length of quartz rod and allow the rod to remain in the sample cup during the combustion).

NOTE 3—After repeated use of the bomb for sulfur determinations, a film may be noticed on the inner surface. This dullness can be removed by periodic polishing of the bomb. A satisfactory method for doing this is to rotate the bomb in a lathe at about 300 rpm and polish the inside surface with emery polishing papers Grit No. 2/0, or equivalent paper,[8] coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide[9] and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before the bomb is used it shall be washed with soap and water to remove oil or paste left from the polishing operation.

NOTE 4—**Caution:** Do not use more than 1.0 g total of sample and white oil or other low sulfur combustible material or more than 0.8 g if the IP 12 bomb is used.

Sulfur Content percent	Weight of Sample, g	Weight of White Oil, g
5 or under	0.6 to 0.8	0.0
Over 5	0.3 to 0.4	0.3 to 0.4

NOTE 5—Use of sample weights containing over 20 mg of chlorine may cause corrosion of the bomb. To avoid this, it is recommended that for samples containing over 2 % chlorine, the sample weight be based on the chlorine content as given in the following table:

Chlorine Content percent	Weight of Sample, g	Weight of White Oil, g
2 to 5	0.4	0.4
Over 5 to 10	0.2	0.6
Over 10 to 20	0.1	0.7
Over 20 to 50	0.05	0.7

NOTE 6—If the sample is not readily miscible with white oil, some other low sulfur combustible diluent may be substituted. However, the combined weight of sample and nonvolatile diluent shall not exceed 1.0 g or more than 0.8 g if the IP 12 bomb is used.

6.2 *Addition of Oxygen*—Place the sample cup in position and arrange the cotton wisp or nylon thread so that the end dips into the sample. Assemble the bomb and tighten the cover securely. (**Caution—**See Note 7.) Admit oxygen slowly (to avoid blowing the oil from the cup) until a pressure is reached as indicated in the following table:

Capacity of Bomb, ml	Minimum Gage Pressure,[A] kgf/cm^2(atm)	Maximum Gage Pressure,[A] kgf/cm^2(atm)
300 to 350	39 (38)	41 (40)
350 to 400	36 (35)	38 (37)
400 to 450	31 (30)	33 (32)
450 to 500	28 (27)	30 (29)

[A] The minimum pressures are specified to provide sufficient oxygen for complete combustion and the *maximum pressures represent a safety requirement.*

NOTE 7—**Caution:** Do not add oxygen or ignite the sample if the bomb has been jarred, dropped, or tilted.

6.3 *Combustion*—Immerse the bomb in a cold distilled-water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. (**Caution**—See Note 8.) Remove the bomb from the bath after immersion for at least 10 min. Release the pressure at a slow, uniform rate such that the operation requires not less than 1 min. Open the bomb and examine the contents. If traces of unburned oil or sooty deposits are found, discard the determination and thoroughly clean the bomb before again putting it in use (Note 3).

NOTE 8—**Caution:** Do not go near the bomb until at least 20 s after firing.

6.4 *Collection of Sulfur Solution*— Rinse the interior of the bomb, the oil cup, and the inner surface of the bomb cover with a fine jet of water, and collect the washings in a 600-mL beaker having a mark to indicate 75 mL. Remove any precipitate in the bomb by means of a rubber policeman. Wash the base of the terminals until the washings are neutral to the indicator methyl red. Add 10 mL of saturated bromine water to the washings in the beaker. (The volume of the washings is normally in excess of 300 mL.) Place the sample cup in a 50-mL beaker. Add 5 mL of saturated bromine water, 2 mL of HCl, and enough water just to cover the cup. Heat the contents

---

[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[8] Emery Polishing Paper Grit No. 2/0can be purchased from Norton Co., Troy, N. Y.

[9] Chromic oxide may be purchased from J. T. Baker & Co., Phillipsburg, N. J.

of the beaker to just below its boiling point for 3 or 4 min and add to the beaker containing the bomb washings. Wash the sample cup and the 50-mL beaker thoroughly with water. Remove any precipitate in the cup by means of a rubber policeman. Add the washings from the cup and the 50-mL beaker, and the precipitate, if any, to the bomb washings in the 600-mL beaker. Do not filter any of the washings, since filtering would remove any sulfur present as insoluble material.

6.5 *Determination of Sulfur*—Evaporate the combined washings to 200 mL on a hot plate or other source of heat. Adjust the heat to maintain slow boiling of the solution and add 10 mL of the $BaCl_2$ solution, either in a fine stream or dropwise. Stir the solution during the addition and for 2 min thereafter. Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume approximately 75 mL as indicated by a mark on the beaker. Remove the beaker from the hot plate (or other source of heat) and allow it to cool for 1 hr before filtering. Filter the supernatant liquid through an ashless, quantitative filter paper (Note 9). Wash the precipitate with water, first by decantation and then on the filter, until free from chloride. Transfer the paper and precipitate to a weighed crucible and dry (Note 10) at a low heat until the moisture has evaporated. Char the paper completely without igniting it, and finally ignite at a bright red heat until the residue is white in color. After ignition is complete, allow the crucible to cool at room temperature, and weigh.

NOTE 9—A weighed porcelain filter crucible (Selas type) of 5 to 9-μm porosity may be used in place of the filter paper. In this case the precipitate is washed free of chloride and then dried to constant weight at 500 ± 25°C.

NOTE 10—A satisfactory means of drying, charring, and igniting the paper and precipitate is to place the crucible containing the wet filter paper in a cold electric muffle furnace and to turn on the current. Drying, charring, and ignition usually will occur at the desired rate.

6.6 *Blank*—Make a blank determination whenever new reagents, white oil, or other low-sulfur combustible material are used. When running a blank on white oil, use 0.3 to 0.4 g and follow the normal procedure.

7. Calculation

7.1 Calculate the sulfur content of the sample as follows:

$$\text{Sulfur, weight percent} = (P - B)13.73/W \qquad (1)$$

where:

P = grams of $BaSO_4$ obtained from sample,
B = grams of $BaSO_4$ obtained from blank, and
W = grams of sample used.

8. Report

8.1 Report the results of the test to the nearest 0.01 %.

9. Precision and Bias [10]

9.1 The precision of this test is not known to have been obtained in accordance with currently accepted guidelines (for example in Committee D-2 Research Report, "Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants")[11].

9.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

9.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Sulfur, weight percent	Repeatability	Reproducibility
0.1 to 0.5	0.04	0.05
0.5 to 1.0	0.06	0.09
1.0 to 1.5	0.08	0.15
1.5 to 2.0	0.12	0.25
2.0 to 5.0	0.18	0.27

NOTE 11—The precision shown in the above table does not apply to samples containing over 2 % chlorine because an added restriction on the amount of sample which can be ignited is imposed.

NOTE 12—This test method has been cooperatively tested only in the range of 0.1 to 5.0 % sulfur.

NOTE 13—The following information on the precision of this method has been developed by the Institute of Petroleum (London):

(*a*) Results of duplicate tests should not differ by more than the following amounts:

Repeatability	Reproducibility
$0.016\,x + 0.06$	$0.037\,x + 0.13$

where x is the mean of duplicate test results.

(*b*) These precision values were obtained in 1960 by statistical examination of interlaboratory test results.[12] No limits have been established for additive concentrates.

9.2 *Bias*—Results obtained in one laboratory by Test Method D 129 on NIST Standard Reference Material Nos. 1620A, 1621C, and 1662B were found to be 0.05 mass % higher than the accepted reference values.

10. Keywords

10.1 bomb; sulfur

---

[10] Supporting data is available from ASTM Headquarters. Request RR:D02-1278.

[11] *Annual Book of ASTM Standards*, Vol 05.03.

[12] IP Standards for Petroleum and Its Products, Part I, Appendix E.

ASTM Logo Removed **D 129**

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D396: Standard Specification for Fuel Oils

CFR Section(s): 40 CFR 60.41b

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 396 – 98

An American National Standard

Standard Specification for Fuel Oils[1]

This standard is issued under the fixed designation D 396; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification (Note 1) covers grades of fuel oil intended for use in various types of fuel-oil-burning equipment under various climatic and operating conditions. These grades are described as follows:

1.1.1 Grades 1 and 2 are distillate fuels for use in domestic and small industrial burners. Grade 1 is particularly adapted to vaporizing type burners or where storage conditions require low pour point fuel.

1.1.2 Grades 4 (Light) and 4 are heavy distillate fuels or distillate/residual fuel blends used in commercial/industrial burners equipped for this viscosity range.

1.1.3 Grades 5 (Light), 5 (Heavy), and 6 are residual fuels of increasing viscosity and boiling range, used in industrial burners. Preheating is usually required for handling and proper atomization.

NOTE 1—For information on the significance of the terminology and test methods used in this specification, see Appendix X1.

NOTE 2—A more detailed description of the grades of fuel oils is given in X1.3.

1.2 This specification is for the use of purchasing agencies in formulating specifications to be included in contracts for purchases of fuel oils and for the guidance of consumers of fuel oils in the selection of the grades most suitable for their needs.

1.3 Nothing in this specification shall preclude observance of federal, state, or local regulations which can be more restrictive.

1.4 All values are stated in SI units and are regarded as standard.

NOTE 3—The generation and dissipation of static electricity can create problems in the handling of distillate burner fuel oils. For more information on the subject, see Guide D 4865.

2. Referenced Documents

2.1 *ASTM Standards:*

D 56 Test Method for Flash Point by Tag Closed Tester[2]
D 86 Test Method for Distillation of Petroleum Products[2]
D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester[2]
D 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation[2]
D 97 Test Method for Pour Point of Petroleum Oils[2]
D 129 Test Method for Sulfur in Petroleum Products (General Bomb Method)[2]
D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test[2]
D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[2]
D 473 Test Method for Sediment in Crude Oils and Fuel Oils by the Extraction Method[2]
D 482 Test Method for Ash from Petroleum Products[2]
D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products[2]
D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)[2]
D 1298 Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method[2]
D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)[2]
D 2622 Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry[3]
D 2709 Test Method for Water and Sediment in Distillate Fuels by Centrifuge[3]
D 3245 Test Method for Pumpability of Industrial Fuel Oils[3]
D 3828 Test Methods for Flash Point by Small Scale Closed Tester[3]
D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter[3]
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]
D 4294 Test Method for Sulfur in Petroleum Products by Energy-Dispersive X-Ray Fluorescence Spectrometry[3]
D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems[4]
D 5949 Test Method for Pour Point of Petroleum Products

---

[1] This specification is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E on Burner, Diesel, Non-aviation Gas Turbine, and Marine Fuels .

Current edition approved Apr. 10, 1998. Published September 1998. Originally published as D 396 – 34 T. Last previous edition D 396 – 96.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] *Annual Book of ASTM Standards*, Vol 05.03.

(Automatic Pressure Pulsing Method)[4]

D 5950 Test Method for Pour Point of Petroleum Products (Automatic Tilt Method)[4]

D 5985 Test Method for Pour Point of Petroleum Products (Rotational Method)[4]

2.2 *Other Documents:*[5]

26 CFR Part 48 Diesel Fuel Excise Tax; Dye Color and Concentration

40 Part 80 Regulation of Fuel and Fuel Additives

3. General Requirements

3.1 The grades of fuel oil specified herein shall be homogeneous hydrocarbon oils, free from inorganic acid, and free from excessive amounts of solid or fibrous foreign matter.

3.2 All grades containing residual components shall remain uniform in normal storage and not separate by gravity into light and heavy oil components outside the viscosity limits for the grade.

4. Detailed Requirements

4.1 The various grades of fuel oil shall conform to the limiting requirements shown in Table 1. A representative sample shall be taken for testing in accordance with Practice D 4057.

4.2 Modifications of limiting requirements to meet special operating conditions agreed upon between the purchaser, the seller, and the supplier shall fall within limits specified for each grade, except as stated in supplementary footnotes for Table 1.

5. Test Methods

5.1 The requirements enumerated in this specification shall be determined in accordance with the following ASTM test methods,[6] except as may be required under 5.1.1.

5.1.1 *Flash Point*—Test Method D 93, except where other methods are prescribed by law. For all grades, Test Method D 3828 may be used as an alternate with the same limits. For Grades No. 1 and No. 2, Test Method D 56 may be used as an alternate with the same limits, provided the flash point is below

---

[5] Available from Superintendent of Documents, U. S. Government Printing Office, Washington, DC 20402.

[6] For information on the precision of the ASTM test methods for fuel oils refer to "An Evaluation of Methods for Determination of Sulfur in Fuel Oils" by A. R. Crawford, Esso Mathematics & Systems Inc. and G. V. Dyroff, Esso Research and Engineering Co., 1969. This document is available from the Publications Section, API Library American Petroleum Institute, 1220 L St., N.W., Washington, DC 20005.

TABLE 1 Detailed Requirements for Fuel Oils[A]

Property	ASTM Test Method[B]	No. 1[C]	No. 2[C]	Grade No. 4 (Light)[C]	No. 4	No. 5 (Light)	No. 5 (Heavy)	No. 6
Flash Point °C, min	D 93	38	38	38	55	55	55	60
Water and sediment, % vol, max	D 2709	0.05	0.05	...	...	...	...	...
	D 95 + D 473	...	...	(0.50)[D]	(0.50)[D]	(1.00)[D]	(1.00)[D]	(2.00)[D]
Distillation temperature °C	D 86							
10 % volume recovered, max		215	...	...	...	...	...	...
90 % volume recovered, min		...	282	...	...	...	...	...
max		288	338	...	...	...	...	...
Kinematic viscosity at 40°C, mm²/s	D 445							
min		1.3	1.9	1.9	>5.5	...	...	...
max		2.1	3.4	5.5	24.0[E]			
Kinematic viscosity at 100°C, mm²/s	D 445							
min		...	...	...	...	5.0	9.0	15.0
max		...	...	...	...	8.9[E]	14.9[E]	50.0[E]
Ramsbottom carbon residue on 10 % distillation residue % mass, max	D 524	0.15	0.35	...	...	...	...	...
Ash, % mass, max	D 482	...	...	0.05	0.10	0.15	0.15	...
Sulfur, % mass max[F]	D 129	0.50	0.50	...	...	...	...	...
Copper strip corrosion rating, max, 3 h at 50°C	D 130	No. 3	No. 3	...	...	...	...	...
Density at 15°C, kg/m³	D 1298							
min		...	...	>876[G]	...	...	...	...
max		850	876	...	...	...	...	...
Pour Point °C, max[H]	D 97	−18	−6	−6	−6	...	...	[I]

[A] It is the intent of these classifications that failure to meet any requirement of a given grade does not automatically place an oil in the next lower grade unless in fact it meets all requirements of the lower grade. However, to meet special operating conditions modifications of individual limiting requirements may be agreed upon among the purchaser, seller and manufacturer.

[B] The test methods indicated are the approved referee methods. Other acceptable methods are indicated in Section 2 and 5.1.

[C] Under United States regulations, Grades No. 1, No. 2, and No.4 (Light) are required by 40 CFR Part 80 to contain a sufficient amount of the dye Solvent Red 164 so its presence is visually apparent. At or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lbs per thousand barrels of the solid dye standard SolventRed 26.

[D] The amount of water by distillation by Test Method D 95 plus the sediment by extraction by Test Method D 473 shall not exceed the value shown in the table. For Grade No. 6 fuel oil, the amount of sediment by extraction shall not exceed 0.50 mass %, and a deduction in quantity shall be made for all water and sediment in excess of 1.0 mass %.

[E] Where low sulfur fuel oil is required, fuel oil falling in the viscosity range of a lower numbered grade down to and including No. 4 can be supplied by agreement between the purchaser and supplier. The viscosity range of the initial shipment shall be identified and advance notice shall be required when changing from one viscosity range to another. This notice shall be in sufficient time to permit the user to make the necessary adjustments.

[F] Other sulfur limits may apply in selected areas in the United States and in other countries.

[G] This limit assures a minimum heating value and also prevents misrepresentation and misapplication of this product as Grade No. 2.

[H] Lower or higher pour points can be specified whenever required by conditions of storage or use. When a pour point less than − 18°C is specified, the minimum viscosity at 40°C for grade No. 2 shall be 1.7 mm²/s and the minimum 90 % recovered temperature shall be waived.

[I] Where low sulfur fuel oil is required, Grade No. 6 fuel oil will be classified as Low Pour (+ 15°C max) or High Pour (no max). Low Pour fuel oil should be used unless tanks and lines are heated.

93°C and the viscosity is below 5.5 mm^2/s at 40°C. This test method will give slightly lower values. In cases of dispute, Test Method D 93 shall be used as the referee method.

5.1.2 *Pour Point*—Test Method D 97. For all grades, the automatic Test Methods D 5949, D 5950, and D 5985 can be used as alternates with the same limits. In case of dispute, Test Method 97 shall be used as the referee method. Alternative test methods that indicate flow point properties can be used for low sulfur residual fuels by agreement between purchaser and supplier.

5.1.3 *Water and Sediment*—The water and sediment in Grade Nos. 1 and 2 shall be determined in accordance with Test Method D 2709 and in Grade Nos. 4, 5, and 6 by Test Method D 95 and Test Method D 473. A density of 1.0 kg/L shall be used for the Test Method D 95 water.

5.1.4 *Carbon Residue*—Test Method D 524.

5.1.5 *Ash*—Test Method D 482.

5.1.6 *Distillation*—Distillation of Grade No. 1 and No. 2 oils shall be determined in accordance with Test Method D 86.

5.1.7 *Viscosity*—Viscosity shall be determined in accordance with Test Method D 445.

5.1.8 *Density*—Practice D 1298. Test Method D 4052 can be used as an alternate with the same limits. In case of dispute, Practice D 1298 shall be used as the referee method.

5.1.9 *Corrosion*—Test Method D 130, 3 h test at 50°C.

5.1.10 *Sulfur*—Test Method D 129. Test Methods D 1552, D 2622, and D 4294 can also be used for all grades. In addition, Test Method D 1266 can be used for Grade 1, but only with samples having sulfur contents of 0.4 mass per cent and less (down to 0.01 %). In case of dispute, Test Method D 129 is the referee test method for this specification.

6. Keywords

6.1 burner fuels; fuel oils; furnace oils; petroleum and petroleum products; specifications

APPENDIX

(Nonmandatory Information)

X1. SIGNIFICANCE OF ASTM SPECIFICATION FOR FUEL OILS

X1.1 Scope

X1.1.1 This specification divides fuel oils into grades based upon the types of burners for which they are suitable. It places limiting values on several of the properties of the oils in each grade. The properties selected for limitation are those that are believed to be of the greatest significance in determining the performance characteristics of the oils in the types of burners in which they are most commonly used.

X1.2 Classes

X1.2.1 Because of the methods employed in their production, fuel oils fall into two broad classifications: distillates and residuals. The distillates consist of overhead or distilled fractions. The residuals are bottoms remaining from the distillation, or blends of these bottoms with distillates. In this specification, Grades No. 1 and No. 2 are distillates and the grades from No. 4 to No. 6 are usually residual, although some heavy distillates can be sold as Grade No. 4.

X1.3 Grades

X1.3.1 *Grade No. 1* is a light distillate intended for use in burners of the vaporizing type in which the oil is converted to a vapor by contact with a heated surface or by radiation. High volatility is necessary to ensure that evaporation proceeds with a minimum of residue.

X1.3.2 *Grade No. 2* is a heavier distillate than grade No. 1. It is intended for use in atomizing type burners which spray the oil into a combustion chamber where the tiny droplets burn while in suspension. This grade of oil is used in most domestic burners and in many medium capacity commercial-industrial burners where its ease of handling and ready availability sometimes justify its higher cost over the residual fuels.

X1.3.3 *Grade No. 4 (Light)* is a heavy distillate fuel or distillate/residual fuel blend meeting the specification viscosity range. It is intended for use both in pressure-atomizing commercial-industrial burners not requiring higher cost distillates and in burners equipped to atomize oils of higher viscosity. Its permissible viscosity range allows it to be pumped and atomized at relatively low-storage temperatures.

X1.3.4 *Grade No. 4* is usually a heavy distillate/residual fuel blend but can be a heavy distillate fuel meeting the specification viscosity range. It is intended for use in burners equipped with devices that atomize oils of higher viscosity than domestic burners can handle. Its permissible viscosity range allows it to be pumped and atomized at relatively low storage temperatures. Thus, in all but extremely cold weather it requires no preheating for handling.

X1.3.5 *Grade No. 5 (Light)* is residual fuel of intermediate viscosity for burners capable of handling fuel more viscous than grade No. 4 without preheating. Preheating may be necessary in some types of equipment for burning and in colder climates for handling.

X1.3.6 *Grade No. 5 (Heavy)* is a residual fuel more viscous than Grade No. 5 (Light) and is intended for use in similar service. Preheating may be necessary in some types of equipment for burning and in colder climates for handling.

X1.3.7 *Grade No. 6*, sometimes referred to as Bunker C, is a high-viscosity oil used mostly in commercial and industrial heating. It requires preheating in the storage tank to permit pumping, and additional preheating at the burner to permit atomizing. The extra equipment and maintenance required to handle this fuel usually preclude its use in small installations.

X1.3.8 Residual fuel oil supplied to meet regulations requiring low sulfur content can differ from the grade previously supplied. It may be lower in viscosity (and fall into a different grade number). If it must be fluid at a given temperature, Test Method D 97 need not accurately reflect the pour point which can be expected after a period of storage. It is suggested that the purchaser and supplier discuss the proper handling and operating techniques for a given low-sulfur residual fuel oil in the installation where it is to be used.

X1.4 Significance of Test Methods

X1.4.1 The significance of the properties of fuel oil on which limitations are placed by the specification is as follows:

X1.4.1.1 *Flash Point*—The flash point of a fuel oil is an indication of the maximum temperature at which it can be stored and handled without serious fire hazard. The minimum permissible flash point is usually regulated by federal, state, or municipal laws and is based on accepted practice in handling and use.

X1.4.1.2 *Pour Point*—The pour point is an indication of the lowest temperature at which a fuel oil can be stored and still be capable of flowing under very low forces. The pour point is prescribed in accordance with the conditions of storage and use. Higher pour point fuels are permissible where heated storage and adequate piping facilities are provided. An increase in pour point can occur when residual fuel oils are subjected to cyclic temperature variations that can occur in the course of storage or when the fuel is preheated and returned to storage tanks. To predict these properties, Test Method D 3245 may be required.

X1.4.1.3 *Water and Sediment*—Appreciable amounts of water and sediment in a fuel oil tend to cause fouling of facilities for handling it, and to give trouble in burner mechanisms. Sediment may accumulate in storage tanks and on filter screens or burner parts, resulting in obstruction to flow of oil from the tank to the burner. Water in distillate fuels can cause corrosion of tanks and equipment and it can cause emulsions in residual fuels.

X1.4.1.4 *Carbon Residue*—The carbon residue of a fuel is a measure of the carbonaceous material left after all the volatile components are vaporized in the absence of air. It is a rough approximation of the tendency of a fuel to form deposits in vaporizing burners, such as pot-type and sleeve-type burners, where the fuel is vaporized in an air-deficient atmosphere.

X1.4.1.4.1 To obtain measurable values of carbon residue in the lighter distillate fuel oils, it is necessary to distill the oil to remove 90 % of it in accordance with Section 9 of Test Method D 524, and then determine the carbon residue concentrated in the remaining 10 % bottoms.

X1.4.1.5 *Ash*—The amount of ash is the quantity of non-combustible material in an oil. Excessive amounts can indicate the presence of materials that cause high wear of burner pumps and valves, and contribute to deposits on boiler heating surfaces.

X1.4.1.6 *Distillation*—The distillation test shows the volatility of a fuel and the ease with which it can be vaporized. The test is of greater significance for oils that are to be burned in vaporizing type burners than for the atomizing type. For example, the maximum 10 % and 90 % distilled temperatures are specified for grade No. 1 fuel. The limiting 10 % value assures easy starting in vaporizing type burners and the 90 % limit excludes heavier fractions that would be difficult to vaporize.

X1.4.1.6.1 The limits specified for grade No. 2 heating oil define a product that is acceptable for burners of the atomizing type in household heating installations. Distillation limits are not specified for fuel oils of grades Nos. 4, 5, and 6.

X1.4.1.7 *Viscosity Limits for Grades Nos. 1 and 2*—The viscosity of an oil is a measure of its resistance to flow. In fuel oil it is highly significant since it indicates both the relative ease with which the oil will flow or can be pumped, and the ease of atomization.

X1.4.1.7.1 Viscosity limits for No. 1 and No. 2 grades are specified to help maintain uniform fuel flow in appliances with gravity flow, and to provide satisfactory atomization and constant flow rate through the small nozzles of household burners. For the heavier grades of industrial and bunker fuel oils, viscosity is of major importance, so that adequate preheating facilities can be provided to permit them to be pumped to the burner and to provide good atomization. However, it is equally important that the maximum viscosity under the existing conditions be such that the oil can be pumped satisfactorily from the storage tank to the preheater.

X1.4.1.8 *Density*—Density alone is of little significance as an indication of the burning characteristics of fuel oil. However, when used in conjunction with other properties, it is of value in mass-volume relationships and in calculating the specific energy (heating value) of an oil.

X1.4.1.9 *Corrosion*—The corrosion test serves to indicate the presence or absence of materials that could corrode copper, brass, and bronze components of the fuel system. This property is specified only for Nos. 1 and 2 distillate fuel oils.

X1.4.1.10 Limited sulfur content of fuel oil can be required for special uses in connection with heat treatment, nonferrous metal, glass, and ceramic furnaces or to meet federal, state, or local legislation or regulations.

X1.4.1.11 *Nitrogen*—Nitrogen oxide emission regulations have been imposed on certain combustion facilities as a function of fuel nitrogen content. For purposes of these regulations, distillate fuels, low nitrogen residual fuels, and high nitrogen residual fuels have been defined by their nitrogen content. Installations are required to meet different emission standards according to the classification of the fuel being used. When regulations require such a distinction to be made, fuel nitrogen specifications can be needed in the contractual agreement between the purchaser and the supplier.

ASTM Logo Removed

D 396

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM D512: Standard Test Methods for Chloride Ion In Water

CFR Section(s): 40 CFR 136.3(a)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 512 – 89 (Reapproved 1999)

Standard Test Methods for Chloride Ion In Water[1]

This standard is issued under the fixed designation D 512; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 These test methods cover the determination of chloride ion in water, wastewater (Test Method C only), and brines. The following three test methods are included:

	Sections
Test Method A (Mercurimetric Titration)	7 to14
Test Method B (Silver Nitrate Titration)	15 to 21
Test Method C (Ion-Selective Electrode Method)	22 to 29

1.2 Test Methods A, B, and C were validated under Practice D2777 – 77, and only Test Method B conforms also to Practice D 2777 – 86. Refer to Sections 14, 21, and 29 for further information.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 26.1.1.

1.4 A former colorimetric test method was discontinued. Refer to Appendix X1 for historical information.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1066 Practice for Sampling Steam[2]

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[2]

D 3370 Practices for Sampling Water from Closed Conduits[2]

D 4127 Terminology Used with Ion-Selective Electrodes[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminologies D 1129 and D 4127.

4. Significance and Use

4.1 Chloride ion is under regulation in water, and must, therefore, be measured accurately. It is highly detrimental to high-pressure boiler systems and to stainless steel, so monitoring is essential for prevention of damage. Chloride analysis is widely used as a tool for estimating the cycles of concentration, such as in cooling tower applications. Processing waters and pickling solutions used in the food processing industries also require dependable methods of analysis for chloride.

5. Purity of Reagents

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*— Unless otherwise indicated, all references to water shall be understood to mean Type II reagent water conforming to Specification D 1193.

6. Sampling

6.1 Collect the sample in accordance with Practice D 1066 and Practices D 3370, as applicable.

TEST METHOD A—MERCURIMETRIC TITRATION [4]

7. Scope

7.1 This test method can be used to determine chloride ion in water, provided interferences are absent (see Section 9).

7.2 Though not specified in the research report, the precision statement is presumed to have been obtained using Type II reagent water. It is the responsibility of the analyst to assure the validity of this test method for untested matrices.

7.3 This test method was validated for the concentration

---

[1] These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved Oct. 2, 1989. Published December 1989. Originally published as D 512 – 38. Last previous edition D 512 – 88.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[4] For information of interest in connection with this test method, and supporting data, refer to Clark, F. E., "Determination of Chloride in Water," *Analytical Chemistry*, Vol 22, April 1950, pp. 553–555, and Vol 22, November 1950, p. 1458.

***A Summary of Changes section appears at the end of this standard.**

range 8.0 to 250 mg/L Cl^-.

8. Summary of Test Method

8.1 Dilute mercuric nitrate solution is added to an acidified sample in the presence of mixed diphenylcarbazone-bromophenol blue indicator. The end point of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.

9. Interferences

9.1 The anions and cations generally found in water offer no interference. Zinc, lead, nickel, and ferrous and chromous ions affect solution and end-point colors, but do not reduce the accuracy of the titration when present in concentrations up to 100 mg/L. Copper is tolerable up to 50 mg/L. Titration in the presence of chromate ion requires indicator with extra background color (alphazurine) and prior reduction for concentrations above 100 mg/L. Ferric ion above 10 mg/L must be reduced before titration, and sulfite ion must be oxidized. Bromide and fluoride will be partially titrated with the chloride. Quaternary ammonium salts also interfere if present in significant amounts (1 to 2 mg/L). Deep color may also interfere.

10. Apparatus

10.1 *Microburet*, 1 or 5-mL, with 0.01-mL graduation intervals.

11. Reagents and Materials

11.1 *Hydrogen Peroxide* (30 % H_2O_2).

11.2 *Hydroquinone Solution* (10 g/L)—Dissolve 1 g of purified hydroquinone in water and dilute to 100 mL.

11.3 *Mercuric Nitrate Solution, Standard* (0.025*N*)—Dissolve 4.2830 g of mercuric nitrate ($Hg(NO_3)_2 \cdot H_2O$) in 50 mL of water acidified with 0.5 mL of concentrated nitric acid (HNO_3, sp gr 1.42). Dilute the acidified $Hg(NO_3)_2$ solution with water to 1 L. Filter if necessary, and standardize against the standard sodium chloride (NaCl) solution, using the procedure described in Section 12 (see Note 1).

NOTE 1—*Sharpness of End Point*—The end point, while sharp, can be improved somewhat for certain types of water by adding several drops of a 0.05-g/L solution of xylene cyanole FF or alphazurine blue-green dye (color index 714) to the titration sample.

11.4 *Mixed Indicator Solution* [5]—Dissolve 0.5 g of crystalline diphenylcarbazone and 0.05 g of bromophenol blue powder in 75 mL of ethyl alcohol (95 %), and dilute to 100 mL with the alcohol (Note 2). Store in a brown bottle and discard after 6 months (Note 3).

NOTE 2—Methanol, isopropanol, or ethanol denatured with either methanol or isopropanol (Formula 3A) may be used if pure ethyl alcohol is not available. Other denatured ethanol formulae are not suitable.

NOTE 3—Liquid indicator generally deteriorates to the point that it yields no end-point color after 12 to 18 months of storage. High temperature (above 37.8°C (100°F)) and exposure to bright light may shorten storage life. A dry powder mixture of the two indicator ingredients is stable for much longer periods. Both the powder mixture (capsule form) and the liquid indicator are available commercially.

11.5 *Nitric Acid* (*3 + 997*)—Mix 3 volumes of concentrated nitric acid (HNO_3, sp gr 1.42) with 997 volumes of water.

11.6 *pH Indicating Paper*, long-range type, covering a pH range 1 to 11.

11.7 *Sodium Chloride Solution, Standard* (0.025*N*)—Dry several grams of sodium chloride (NaCl) for 1 h at 600°C. Dissolve 1.4613 g of the dry salt in water, and dilute to 1 L at 25°C in a volumetric flask.

11.8 *Sodium Hydroxide Solution* (10 g/L)—Dissolve 10 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

12. Procedure

12.1 Use a volume of sample such that it will contain not more than 20 mg of chloride ion, diluting the sample with water to approximately 50-mL volume if necessary. Determine an indicator blank on 50 mL of chloride-free water, applying the same procedure followed for the sample.

12.2 Add 5 to 10 drops of mixed indicator solution, and shake or swirl the flask. If a blue-violet or red color develops, add HNO_3 (3 + 997) dropwise until the color changes to yellow. Add 1 mL of excess acid. If a yellow or orange color forms immediately on addition of the mixed indicator, add NaOH solution (10 g/L) dropwise until the color changes to blue-violet; then add HNO_3 (3 + 997) dropwise until the color changes to yellow and further add 1 mL excess of acid (Note 4).

NOTE 4—The prescribed acidification provides a satisfactory pH range from 3.0 to 3.5. Acidified samples on which electrometric pH measurements have been made shall not be used for chloride determinations, because the use of the calomel reference electrode may introduce error due to chloride contamination. For precise pH adjustment of samples having a low-chloride concentration, instrumental measurements may be made on one sample aliquot to determine treatment needed for another to be used for the chloride test.

12.3 Titrate the solution and a blank with 0.025 *N* $Hg(NO_3)_2$ solution until a blue-violet color, as viewed by transmitted light, persists throughout the solution (Note 5). Record the millilitres of $Hg(NO_3)_2$ solution added in each case.

NOTE 5—The use of indicator modifications and the presence of heavy metal ions can change solution colors without affecting accuracy of the determination. For example, solutions containing alphazurine may be bright blue when neutral, grayish purple when basic, blue-green when acidic, and blue-violet at the chloride end point. Solutions containing about 100 mg/L nickel ion and normal mixed indicator are purple when neutral, green when acid, and gray at the chloride end point. When applying this test method to samples that contain colored ions or that require modified indicator, it is recommended that the operator be familiar with the specific color changes involved by experimenting with solutions prepared as standards for comparison of color effects.

12.4 If chromate ion is present in the absence of iron and in concentration less than 100 mg/L, use the alphazurine modified mixed indicator (Note 1) and acidify the sample as described in 12.2 but to pH 3 as indicated by pH indicating paper. Titrate the solution as described in 12.3, but to an olive-purple end point.

12.5 If chromate ion is present in the absence of iron and in concentration greater than 100 mg/L, add 2 mL of fresh

---

[5] This diphenylcarbazone 1-bromophenol blue indicator is covered by U.S. Patent No. 2,784,064.

hydroquinone solution and proceed as described in 12.2 and 12.3.

12.6 If ferric ion is present in the absence or presence of chromate ions, use a sample of such volume as to contain no more than 2.5 mg of ferric ion or of ferric ion plus chromate ion. Add 2 mL of fresh hydroquinone solution, and proceed as described in 12.2 and 12.3.

12.7 If sulfite ion is present, add 0.5 mL of H_2O_2 to 50 mL of the sample in the Erlenmeyer flask and mix for 1 min. Then proceed as described in 12.2 and 12.3.

13. Calculation

13.1 Calculate the chloride ion concentration, in milligrams per litre, in the original sample as follows:

$$\text{Chloride, mg/L} = [(V_1 - V_2) \times N \times 35\,453]/S$$

where:

V_1 = standard $Hg(NO_3)_2$ solution required for titration of the sample, mL,

V_2 = standard $Hg(NO_3)_2$ solution required for titration of the blank, mL,

N = normality of the $Hg(NO_3)_2$ solution, and

S = sample used in 12.1, mL.

14. Precision and Bias [6]

14.1 *Precision Statement*—The precision of this test method may be expressed as follows:

$$S_T = 0.023X + 0.43$$

$$S_O = 0.002X + 0.46$$

where:

S_T = overall precision, mg/L,

S_O = single-operator precision, mg/L, and

X = concentration of chloride ion determined.

14.2 *Bias Statement*— Recoveries of known amounts of chloride were as follows:

Amount Added, mg/L	Amount Found, mg/L	± % Bias	Statistically Significant (95 % Confidence Level)
250	248	−0.80	no
80.0	79.3	−0.88	no
8.00	7.51	−6.13	yes

14.3 The information presented in 14.1 and 14.2 is derived from round-robin testing in which five laboratories, including seven operators, participated. Though not clearly specified in the test report, the matrix is presumed to be Type II reagent water. Of seven data sets ranked as described in Practice D 2777, none was rejected, nor were any data points determined to be "outliers." Three sample levels were run on at least three days. The method of "least squares" was used to determine the precision statement, with correlation of 0.7394 for S_O and 0.9993 for S_T.

14.4 It is the responsibility of the analyst to assure the validity of this test method for untested matrices.

14.5 Precision and bias for this test method conforms to Practice D 2777 – 77, which was in place at the time of collaborative testing. Under the allowances made in 1.5 of Practice D 2777 – 86, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D–19 test methods.

TEST METHOD B—SILVER NITRATE TITRATION

15. Scope

15.1 This test method[7] is intended primarily for water where the chloride content is 5 mg/L or more, and where interferences such as color or high concentrations of heavy metal ions render Test Method A impracticable.

15.2 Though not specified in the research report, the precision and bias statement is presumed to have been obtained using Type II reagent water. It is the responsibility of the analyst to assure the validity of this test method for untested matrices.

15.3 This test method was validated for the concentration range 8.0 to 250 mg/L Cl^-.

16. Summary of Test Method

16.1 Water adjusted to approximately pH 8.3 is titrated with silver nitrate solution in the presence of potassium chromate indicator. The end point is indicated by persistence of the brick-red silver chromate color.

17. Interferences

17.1 Bromide, iodide, and sulfide are titrated along with the chloride. Orthophosphate and polyphosphate interfere if present in concentrations greater than 250 and 25 mg/L, respectively. Sulfite and objectionable color or turbidity must be eliminated. Compounds which precipitate at pH 8.3 (certain hydroxides) may cause error by occlusion.

18. Reagents

18.1 *Hydrogen Peroxide* (30 %) (H_2O_2).

18.2 *Phenolphthalein Indicator Solution* (10 g/L)—Prepare as directed in Methods E 200.

18.3 *Potassium Chromate Indicator Solution*—Dissolve 50 g of potassium chromate (K_2CrO_4) in 100 mL of water, and add silver nitrate ($AgNO_3$) until a slight red precipitate is produced. Allow the solution to stand, protected from light, for at least 24 h after the addition of $AgNO_3$. Then filter the solution to remove the precipitate, and dilute to 1 L with water.

18.4 *Standard Solution, Silver Nitrate (0.025 N)*—Crush approximately 5 g of silver nitrate ($AgNO_3$) crystals and dry to constant weight at 40°C. Dissolve 4.2473 g of the crushed, dried crystals in water and dilute to 1 L. Standardize against the standard NaCl solution, using the procedure given in Section 19.

18.5 *Standard Solution, Sodium Chloride (*0.025 *N)*— Prepare as described in 11.7.

18.6 *Sodium Hydroxide Solution* (10 g/L)—Prepare as described in 11.8.

18.7 *Sulfuric Acid* (1 + 19)—Carefully add 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) to 19 volumes of water, while mixing.

---

[6] Supporting data are available from ASTM Headquarters. Request RR: D19-1076.

[7] Mohr, F., *ANN.*, 97, 335 (1856).

19. Procedure

19.1 Pour 50 mL, or less, of the sample, containing not more than 20 nor less than 0.25 mg of chloride ion, into a white porcelain container (see Note 6). If sulfite ions are present, add 0.5 mL of hydrogen peroxide (H_2O_2) to the sample, mix, and let stand for 1 min. Dilute to approximately 50 mL with water, if necessary. Adjust the pH to the phenolphthalein end point (pH 8.3), using H_2SO_4, (1 + 19) or NaOH solution (10 g/L).

NOTE 6—An 80 mL white porcelain dish, a 1-in stirrer bar, and a magnetic stirrer have been found useful for this purpose.

19.2 Add approximately 1.0 mL of K_2CrO_4 indicator solution and mix. Add standard $AgNO_3$ solution dropwise from a 25-mL buret until the brick-red (or pink) color persists throughout the sample when illuminated with a yellow light or viewed with yellow goggles.

19.3 Repeat the procedure described in 19.1 and 19.2, using exactly one half as much original sample, diluted to 50 mL with water.

19.4 If the volume of titrant used in 19.3 is one-half of that used in titrating the aliquot in 19.1, proceed to the calculation section. If not, significant interferences are present and compensation must be made; alternatively, another method must be used.

20. Calculation

20.1 Calculate the chloride ion concentration in the original sample, in milligrams per litre as follows:

$$\text{Chloride, mg/L} = [(V_1 - V_2) \times N \times 70\ 906]/S$$

where:

V_1 = standard solution $AgNO_3$ added in titrating the sample prepared in 19.1, mL,
V_2 = standard solution, $AgNO_3$ added in titrating the sample prepared in 19.3, mL,
N = normality of standard $AgNO_3$ solution, and
S = original sample in the 50-mL test specimen prepared in 19.1, mL.

21. Precision and Bias [6]

21.1 *Precision*—The precision of this test method may be expressed as follows:

$$S_T = 0.013X + 0.70$$
$$S_O = 0.007X + 0.53$$

where:

S_T = overall precision, mg/L,
S_O = single-operator precision, mg/L, and
X = concentration of chloride ion determined.

21.2 *Bias*—Recoveries of known amounts of chloride were as follows:

Amount Added, mg/L	Amount Found, mg/L	± % Bias	Statistically Significant (95 % Confidence Level)
250	248	−0.80	yes
80.00	79.1	−1.13	yes
8.00	7.77	−2.88	yes

21.3 The information in 21.2 is derived from round-robin testing in which six laboratories, including ten operators, participated. Though not clearly specified in the test report, the matrix is presumed to be Type II reagent water. Of ten data sets ranked as described in Practice D 2777, none was rejected, but one data point was determined to be an "outlier." Three sample levels were run on at least three days.

The method of least squares was used to determine the precision statement, with correlation of 0.9959 for S_O and 0.9940 for S_T.

21.4 It is the responsibility of the analyst to assure the validity of this test method for untested matrices.

TEST METHOD C—ION-SELECTIVE ELECTRODE

22. Scope

22.1 This test method is applicable to the measurement of chloride ion in natural waters, drinking water, and waste waters.

22.2 Samples containing 2 to 1000 mg/L of chloride may be analyzed by this test method. The concentration range may be extended by dilution of an appropriate aliquot before the addition of the ionic strength adjustor.

22.3 The precision and bias statements were obtained using reagent water and a water matrix of choice that included natural and waste waters. It is the responsibility of the analyst to determine the acceptability of this test method for the matrix being analyzed.

23. Summary of Test Method

23.1 Chloride ion is measured potentiometrically using a chloride ion-selective electrode in conjunction with a double junction, sleeve-type reference electrode. Potentials are read using a pH meter having an expanded millivolt scale, or a selective-ion meter having a direct concentration scale for chloride.

23.2 The electrodes are calibrated in known chloride solutions, and the concentrations of unknowns are determined in solutions with the same background. Samples and standards should be at the same temperature.

23.3 Standards and samples are diluted with an ionic strength adjustor that also minimizes possible interferences such as ammonia, bromide, iodide, cyanide, or sulfide.

24. Interferences

24.1 There is no interference from up to 500 mg/L of sulfide, 1000 mg/L of bromide or iodide, a hundredfold excess of cyanide over chloride, or from 1000 mg/L of ammonia.

25. Apparatus

25.1 *pH Meter*, with expanded millivolt scale. This test method can be adapted for use with a *selective-ion meter.*

25.2 *Chloride Ion-Selective Electrode*, having a light-desensitized silver chloride (AgCl) membrane. Not all chloride ion-selective electrodes are suitable for this test method, since the ionic strength adjustor is incompatible with some membranes. In particular, silver chloride/silver sulfide membranes are inappropriate, since the sulfide can be oxidized by the ionic strength adjustor.

25.3 *Sleeve-Type Double Junction Reference Electrode*, using solution prepared in 26.4 as the outer sleeve filling solution.

NOTE 7—Other types of double junction reference electrodes may be suitable, but supporting data included in this test method reflect only the recommended type.

25.4 *Mixer*, magnetic, with a TFE-fluorocarbon-coated stirring bar.

26. Reagents

26.1 *Chloride Ionic Strength Adjustor (CISA)*—Dissolve 15.1 g of sodium bromate in 800 mL of water. Add 75 mL of concentrated nitric acid (HNO_3, sp. gr 1.42) (Note 8). Stir well. Dilute with water to 1 L. Store CISA in a polyethylene or glass container.

NOTE 8—For low-level chloride measurement (less than 5 mg/L) the nitric acid used must not contain more than 0.005 % chloride and the sodium bromate must not contain more than 0.003 % chloride. A test for reagent purity is included in this test method.

26.1.1 **Caution**—Sodium bromate is a strong oxidant and should be handled appropriately. Also note that preparation and dilutions of CISA should be made in a well-ventilated area, preferably a hood.

26.2 *Chloride Solution, Stock* (1000 mg/L)—Dissolve 1.648 g of sodium chloride (dried for 1 h at 600°C), in water in a volumetric flask and dilute to 1 L.

26.3 *Chloride Solutions, Standard* (100, 10, and 1 mg/L)—Using volumetric pipets, transfer 100, 10, and 1.0 mL of the chloride stock solution into separate 1-L volumetric flasks and dilute each to 1 L with water.

26.4 *Double-Junction Reference Electrode Outer Filling Solution*—Dilute 1 volume of CISA (26.1) with 1 volume of water.

27. Calibration

27.1 Mix equal volumes of the 1000 mg/L of chloride standard solution, and CISA reagent. Do the same for each of the other three standards.

27.2 Mix equal volumes of water and CISA reagent.

27.3 Place the electrodes in the solution from 27.2, stir well, wait 3 to 5 min, and record the millivolt reading. This solution contains no added chloride, and the potential reading will not be very stable.

27.4 Rinse the electrodes thoroughly, place them in the 1 mg Cl^-/L-CISA mixture and stir well. Wait 1 to 2 min and record the result.

27.4.1 If the difference in readings between 27.3 and 27.4 is less than 15 mV, there is chloride contamination of the reagent that will affect low-level readings, and purer reagents must be obtained.

27.5 Rinse the electrodes, place them in the 10 mg Cl -/LCISA mixture and stir well. Wait 1 min and record the results.

27.6 Repeat 27.5 with 100 and 1000 mg Cl^-/L-CISA mixtures.

27.7 Prepare a calibration curve by plotting, on semilogarithmic graph paper, the potential observed (on the linear scale) versus the concentration of each of the standards used (on the logarithmic scale). Note that volume corrections are incorporated into the calibration, so that samples analyzed in accordance with Section 28 can be read directly.

28. Procedure

28.1 Mix the sample with an equal volume of CISA reagent, and stir thoroughly for 1 to 2 min.

28.2 Insert the electrodes, wait 1 to 2 min, and record the reading.

28.3 Read chloride concentration of the sample in milligrams per litre directly from the calibration curve.

29. Precision and Bias [6]

29.1 *Precision*—The overall and single-operator precision of this test method, within its designated range, varies with the quantity tested, as shown in Fig. 1, for reagent water, and in Fig. 2, for selected water matrices. These matrices included natural and waste waters.

29.2 *Bias*—Recoveries of known amounts of chloride from reagent water and selected water matrices are as shown in Table 1.

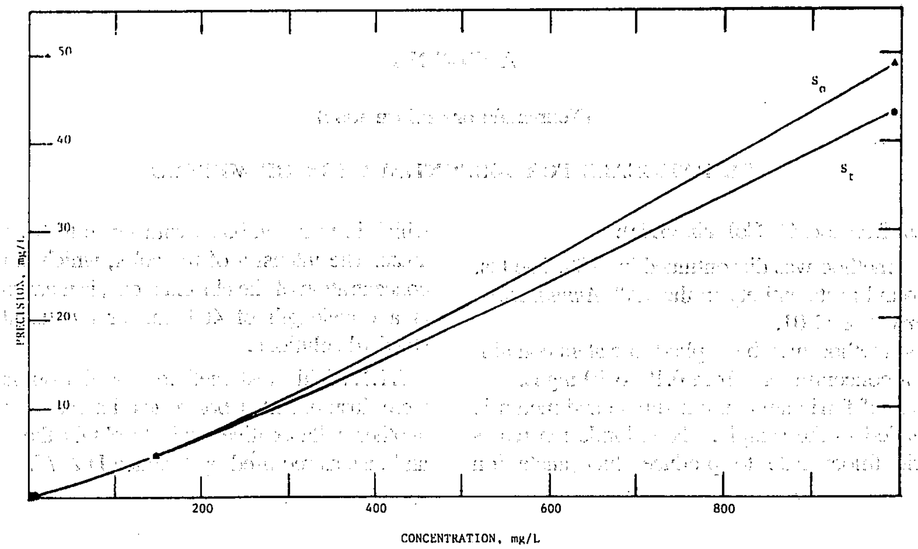


FIG. 1 Interlaboratory Precision for Chloride Found in Reagent Water (Ion Selective Electrode)

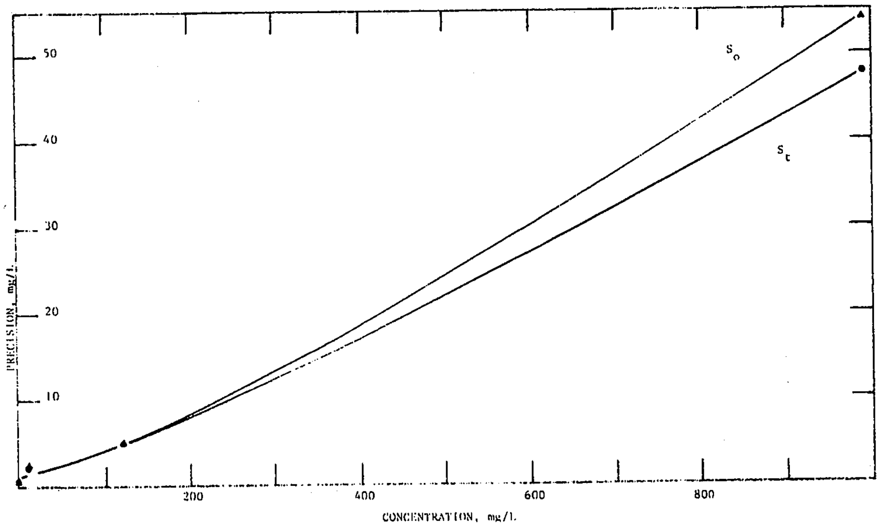


FIG. 2 Interlaboratory Precision for Chloride Found in Selected Water Matrices (Ion Selective Electrode)

TABLE 1 Determination of Bias (Ion Selective Electrode)

	Amount Added, mg/L	Amount Found, mg/L	Bias, ± %	Statistically Significant (95 % Confidence Level)
Reagent water	1.1	1.03	−6.4	no
	10.0	9.94	−0.6	no
	150	148.8	−0.8	no
	1000	992	−0.8	no
Water matrices	1.1	1.04	−5.5	no
	10.0	10.24	+2.4	no
	150	146.0	−2.7	no
	1000	991	−0.9	no

29.3 The information in Table 1 is derived from round-robin testing in which five laboratories, including seven operators, participated. Of seven data sets ranked as described in Practice D 2777, none was rejected in the case of reagent water and one was rejected in the case of selected water matrices. Eight "outlier" data points within the sets were also rejected. Four sample levels were run on three days, and blanks were obtained for the water used.

29.4 It is the responsibility of the analyst to assure the validity of this test method for untested matrices.

29.5 Precision and bias for this test method conforms to Practice D 2777 – 77, which was in place at the time of collaborative testing. Under the allowances made in 1.5 of Practice D 2777 – 86, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D-19 test methods.

30. Keywords

30.1 analysis; chloride; electrode; titrametric; water

APPENDIX

(Nonmandatory Information)

X1. RATIONALE FOR DISCONTINUATION OF METHOD

X1.1 Former Test Method C (Colorimetric):

X1.1.1 This test method was discontinued in 1988. The test method may be found in its entirety in the 1988 *Annual Book of ASTM Standards*, Vol 11.01.

X1.1.2 This test method may be applied to waters containing chloride ion in concentrations from 0.10 to 10 mg/L.

X1.1.3 Solutions of ferric ammonium sulfate and mercuric thiocyanate are added to the sample. The chloride ion reacts with the mercuric thiocyanate to produce thiocyanate ion which in turn combines with ferric to form red ferric thiocyanate. The intensity of the color, which is proportional to the concentration of the chloride ions, is measured photometrically at a wavelength of 463 nm, or by visual comparison with standard solutions.

X1.1.4 This test method was discontinued because there were insufficient laboratories interested in participating in another collaborative study to obtain the necessary precision and bias as required by Practice D 2777.

SUMMARY OF CHANGES

This section identifies the location of selected changes to these test methods that have been incorporated since the last issue. For the convenience of the user, Committee D-19 has highlighted those changes that may impact the use of these test methods. This section may also include descriptions of the changes or reasons for the changes, or both.

(1) Subsequent to round robin testing, waters tested and ranges used were revised.

(2) The equation in 20.1 was revised from a previously rounded value.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D611: Standard Test Method for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents

CFR Section(s): 21 CFR 177.1520(b)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

[redacted]

Designation: D 611 – 82 (Reapproved 1998)

An American National Standard

Designation: 2/98

Standard Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents[1]

This standard is issued under the fixed designation D 611; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods were adopted as a joint ASTM-IP standard in 1964.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the determination of the aniline point of petroleum products and hydrocarbon solvents. Method A is suitable for transparent samples with an initial boiling point above room temperature and where the aniline point is below the bubble point and above the solidification point of the aniline-sample mixture. Method B, a thin-film method, is suitable for samples too dark for testing by Method A. Methods C and D are for samples that may vaporize appreciably at the aniline point. Method D is particularly suitable where only small quantities of sample are available. Method E describes a procedure using an automatic apparatus suitable for the range covered by Methods A and B.

1.2 These test methods also cover the determination of the mixed aniline point of petroleum products and hydrocarbon solvents having aniline points below the temperature at which aniline will crystallize from the aniline-sample mixture.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Sections 7.1 and 7.3.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1015 Test Method for Freezing Points of High-Purity Hydrocarbons[2]
D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[2]
D 1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids[2]
D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)[2]
D 2700 Test Method for Knock Characteristics of Motor and Aviation Fuels by the Motor Method[3]
E 1 Specification for ASTM Thermometers[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *aniline point*—the minimum equilibrium solution temperature for equal volumes of aniline and sample.

3.1.2 *mixed aniline point*—the minimum equilibrium solution temperature of a mixture of two volumes of aniline, one volume of sample, and one volume of *n*-heptane of specified purity.

4. Summary of Test Methods

4.1 Specified volumes of aniline and sample, or aniline and sample plus *n*-heptane, are placed in a tube and mixed mechanically. The mixture is heated at a controlled rate until the two phases become miscible. The mixture is then cooled at a controlled rate and the temperature at which two phases separate is recorded as the aniline point or mixed aniline point.

5. Significance and Use

5.1 The aniline point (or mixed aniline point) is useful as an aid in the characterization of pure hydrocarbons and in the analysis of hydrocarbon mixtures. Aromatic hydrocarbons exhibit the lowest, and paraffins the highest values. Cycloparaffins and olefins exhibit values that lie between those for paraffins and aromatics. In homologous series the aniline points increase with increasing molecular weight. Although it occasionally is used in combination with other physical properties in correlative methods for hydrocarbon analysis, the aniline point is most often used to provide an estimate of the aromatic hydrocarbon content of mixtures.

---

[1] These test methods are under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand are the direct responsibility of D02.04on Hydrocarbon Analysis.

Current edition approved Aug. 27, 1982. Published January 1983. Originally published as D 611 – 41 T. Last previous edition D 611 – 77.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 05.04.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

6. Apparatus

6.1 For details of the aniline point apparatus required for each method see:

Annex A1 for Method A
Annex A2 for Method B
Annex A3 for Method C
Annex A4 for Method D
Annex A5 for Method E

NOTE 1—Alternative apparatus may be used, such as the U-tube method for dark oils, provided it has been shown to give results of the same precision and accuracy as those described in the Annexes.

6.2 *Heating and Cooling Bath*—A suitable air bath, a nonvolatile, transparent liquid bath, or an infrared lamp (250 to 375 W), provided with means for controlling the rate of heating.

NOTE 2—Water should not be used as either a heating or cooling medium since aniline is hygroscopic and moist aniline will give erroneous test results. For example, the aniline point of the *n*-heptane reagent as measured with aniline containing 0.1 volume % water is approximately 0.5°C (0.9°F) higher than that measured with dry aniline. If the aniline point is below the dew point of the atmosphere, pass a slow stream of dry inert gas into the aniline point tube to blanket the aniline-sample mixture.

6.3 *Thermometers*, having the following ranges and conforming to the requirements of the designated ASTM or IP specification:

Range	ASTM (Specification E 1)	IP
−38 to + 42°C (−36.5 to + 107.5°F)	33C, 33F	20C
25 to 105°C (77 to 221°F)	34C, 34F	21C
90 to 170°C (194 to 338°F)	35C, 35F	59C

6.4 *Pipets*, with capacities of 10 ± 0.04 mL, 5 ± 0.02 mL, the latter equipped with a long, fine tip. Provide a rubber suction bulb for use with pipets when measuring aniline.

6.5 *Balance*—A laboratory balance sensitive to 0.01 g, suitable for weighing the tube and sample when the sample cannot be pipetted conveniently.

6.6 *Safety Goggles.*

6.7 *Plastic Gloves*, impervious to aniline.

7. Reagents

7.1 *Aniline* (**Warning**—See Note 3.) Dry chemically pure aniline over potassium hydroxide pellets, decant, and distill fresh on the day of use, discarding the first and last 10 %. Aniline thus prepared when tested with *n*-heptane according to Section 9 shall give an aniline point of 69.3 ± 0.2°C (156.7 ± 0.4°F) as determined from the average of two independent tests having a difference of not more than 0.1°C (0.2°F).

NOTE 3—**Warning:** Aniline should not be pipetted directly by mouth because of its extreme toxicity. Aniline is also toxic by absorption through the skin even in *very small quantities,* and should be handled with great caution.

NOTE 4—For routine purposes the distillation process is not mandatory provided the aniline meets the requirements of the test with *n*-heptane.

NOTE 5—The aniline point of aniline and *n*-heptane determined with automatic apparatus (Method E) shall be 69.3 ± 0.2°C (156.7°F ± 0.4°F) when corrected in accordance with the equation in Section A5.2.1

NOTE 6—As an alternative to distilling the aniline on the day of use, the aniline may be distilled as described in 7.1, collecting the distillate in ampoules, sealing the ampoules under vacuum or dry nitrogen, and storing in a cool dark place for future use. In either case, rigid precaution must be taken to avoid contamination from atmospheric moisture (Note 2). It is believed that under these conditions the aniline will remain unchanged for a period exceeding 6 months.

7.2 *Calcium Sulfate*, anhydrous.

7.3 *n-Heptane* (**Warning**—See Note 7), conforming to the requirements listed in Table 1.[5]

NOTE 7—**Warning:** Flammable. Harmful if inhaled. See Annex A6.1.

8. Sample

8.1 Dry the sample by shaking vigorously for 3 to 5 min with about 10 volume % of a suitable drying agent such as anhydrous calcium sulfate or anhydrous sodium sulfate. Reduce the viscosity of viscous samples by warming to a temperature below that which would cause the loss of light ends or the dehydration of the drying agent. Remove any suspended drying agent by use of a centrifuge or by filtration. Heat samples containing separated wax until they are homogeneous and keep heated during filtration or centrifugation to ensure against separation of wax. When suspended water is visibly present and the sample material is known to dissolve less than 0.03 mass % of water, the use of a centrifuge for the removal of suspended water is an acceptable procedure.

9. Procedure for Aniline Point

9.1 The following methods, to be used as applicable, are covered as follows:

9.1.1 *Method A*, described in detail in Annex A1, is applicable to clear samples or to samples not darker than No. 6.5 ASTM color, as determined by Test Method D 1500, having initial boiling points well above the expected aniline point.

9.1.2 *Method B*, described in detail in Annex A2, is applicable to light-colored samples, moderately dark samples, and to very dark samples. It is suitable for samples that are too dark to be tested by Method A.

9.1.3 *Method C*, described in detail in Annex A3, is applicable to clear samples or to samples not darker than No. 6.5 ASTM color, as determined by Test Method D 1500, having initial boiling points sufficiently low as to give incorrect aniline point readings by Method A, for example, aviation gasoline.

9.1.4 *Method D*, described in detail in Annex A4, is applicable to the same type of sample as Method C. It is particularly useful when only limited quantities of sample are available.

TABLE 1 Requirements for *n*-Heptane

		ASTM Method
ASTM Motor Octane Number	0.0 ± 0.2	D 2700
Density at 20°C, g/mL	0.68380 ± 0.00015	D 1217
Refractive index, $n_D^{20°C}$	1.38770 ± 0.00015	D 1218
Freezing point, °C	−90.710 min	D 1015
Distillation, 50 % recovered at 1.013 bar (760 mm Hg), °C	98.427 ± 0.025	[A]
Differential, 80 % recovered minus 20 % recovered, °C	0.020 max	

[A] For equipment and method used, see *Journal of Research,* National Institute of Standards and Technology, Vol 44, No. 3, 1950, pp. 309 and 310 (*RP2079*).

---

[5] These requirements for *n*-heptane are identical, except for tetraethyl lead, with those prescribed in the *1987 Annual Book of ASTM Standards,* Vol 05.04.

9.1.5 *Method E* is applicable when using automatic apparatus in accordance with the instructions in Annex A5.

10. Procedure for Mixed Aniline Point

10.1 This procedure is applicable to samples having aniline points below the temperature at which aniline crystallizes from the mixture. Pipet 10 mL of aniline (**Warning:** See Note 3), 5 mL of sample, and 5 mL of *n*-heptane into a clean, dry apparatus. Determine the aniline point of the mixture by Method A or B as described in Annex A1 or Annex A2.

11. Report

11.1 If the range of three successive observations of the aniline point temperature is not greater than 0.1°C (0.2°F) for light-colored samples or 0.2°C (0.4°F) for dark samples, report the average temperature of these observations, corrected for thermometer calibration errors, to the nearest 0.05°C (0.1°F) as the aniline point.

11.2 If such a range is not obtained after five observations, repeat the test using fresh quantities of aniline and sample in a clean, dry apparatus, and if consecutive temperature observations show a progressive change, or if the range of observations is greater than the repeatability given in 12.1, report the method as being inapplicable.

12. Precision and Bias

12.1 The precision of these test methods as obtained by statistical examination of interlaboratory test results is as follows:

12.1.1 *Repeatability*—The difference between successive test results (two average temperatures obtained in a series of observations as described in Section 11 obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

	Repeatability	
Aniline point of:		
Clear, light-colored samples	0.16°C	(0.3°F)
Moderately dark to very dark samples	0.3°C	(0.6°F)^A
Mixed aniline point of:		
Clear, light-colored samples	0.16°C	(0.3°F)^A
Moderately dark to very dark samples	0.3°C	(0.6°F)^A

---

^{A}Not determined from recent cooperative tests; however, the ratios with those given in the 1953 version are believed to apply.

12.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators, working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

12.2 *Bias*—A statement of bias is now being developed by the subcommittee.

	Reproducibility
Aniline point of:	
Clear, light-colored samples	0.5°C (0.9°F)
Moderately dark to very dark samples	1.0°C (1.8°F)^A
Mixed aniline point of:	
Clear, light-colored samples	0.7°C (1.3°F)^A
Moderately dark to very dark samples	1.0°C (1.8°F)^A

---

^{A}Not determined from recent cooperative tests; however, the ratios with those given in the 1953 version are believed to apply.

12.3 The precision of this test was not obtained in accordance with Committee D-2 Research Report RR:D02-1007, "Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants."

13. Keywords

13.1 aniline point; aromatics; mixed aniline point

ANNEXES

(Mandatory Information)

A1. METHOD A

A1.1 Apparatus

A1.1.1 The apparatus shown in Fig. A1.1 shall consist of the following:

A1.1.1.1 *Test Tube*, approximately 25 mm in diameter and 150 mm in length, made of heat-resistant glass.

A1.1.1.2 *Jacket*, approximately 37 to 42 mm in diameter and 175 mm in length, made of heat-resistant glass.

A1.1.1.3 *Stirrer*, manually operated, metal, approximately 2 mm in diameter (14 B&S gage) metal wire as shown in Fig. A1.1. A concentric ring shall be at the bottom, having a diameter of approximately 19 mm. The length of the stirrer to a right-angle bend shall be approximately 200 mm. The right-angle bend shall be approximately 55 mm long. A glass sleeve approximately 65 mm in length of 3-mm inside diameter shall be used as a guide for the stirrer. Any suitable mechanical device for operating the stirrer as specified is an approved alternative for the manual operation.

A1.2 Procedure

A1.2.1 Clean and dry the apparatus. Pipet 10 mL of aniline (**Warning—** see 7.1) and 10 mL of the dried sample (8.1) into the test tube fitted with stirrer and thermometer. If the material is too viscous for pipetting, weigh to the nearest 0.01 g a quantity of the sample corresponding to 10 mL at room temperature. Center the thermometer in the test tube so that the immersion mark is at the liquid level, making sure that the thermometer bulb does not touch the side of the tube. Center the test tube in the jacket tube. Stir the mixture rapidly using a 50-mm (2-in.) stroke, avoiding the introduction of air bubbles.

A1.2.2 If the aniline-sample mixture is not miscible at room temperature, apply heat directly to the jacket tube so that the temperature rises at a rate of 1 to 3°C (2 to 5°F)/min by

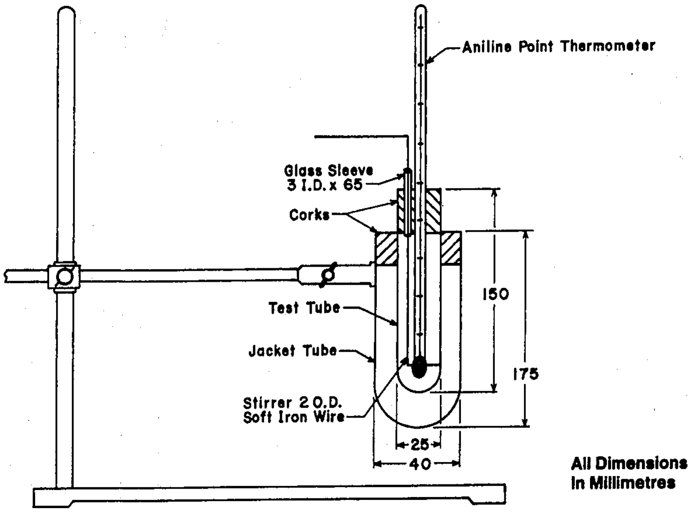


FIG. A1.1 Aniline Point Apparatus (Method A)

removing or reducing the heat source until complete miscibility is obtained. Continue stirring and allow the mixture to cool at a rate of 0.5 to 1.0°C (1.0 to 1.8°F)/min. Continue cooling to a temperature of 1 to 2°C (2.0 to 3.5°F) below the first appearance of turbidity, and record as the aniline point the temperature at which the mixture suddenly becomes cloudy throughout (Note A1.1). This temperature, and not the temperature of separation of small amounts of material, is the minimum equilibrium solution temperature.

NOTE A1.1—The true aniline point is characterized by a turbidity that is so cloudy as to obscure the thermometer bulb in reflected light.

A1.2.3 If the aniline-sample mixture is completely miscible at room temperature, substitute a non-aqueous cooling bath for the heating source, allow to cool at the rate specified in A1.2.2 and determine the aniline point as described.

A1.2.4 Repeat the observation of aniline point temperature by heating and cooling repeatedly until a report as directed in Section 11 can be made.

A2. METHOD B

A2.1 Apparatus

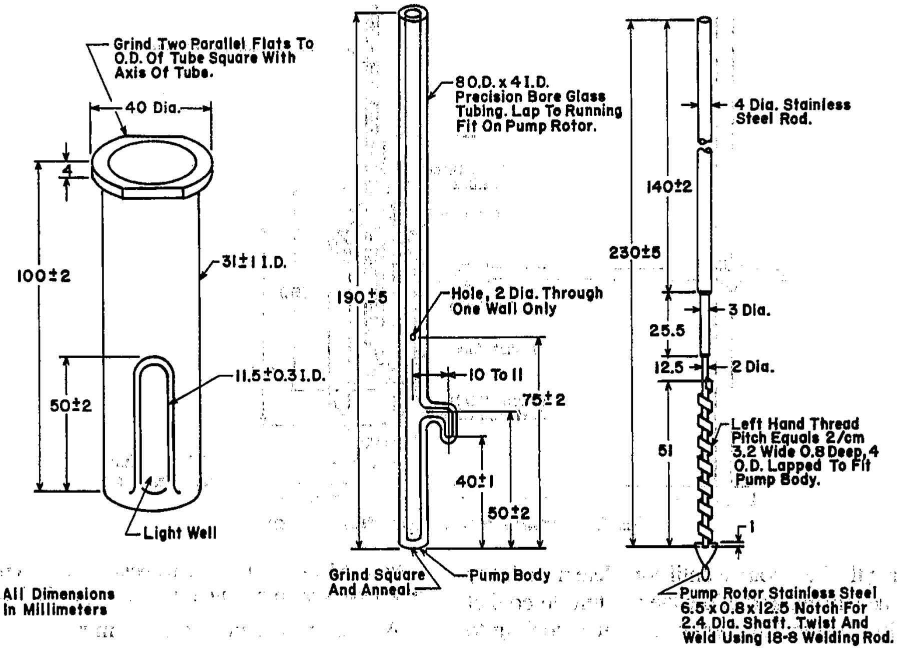
A2.1.1 *Thin-Film Apparatus*, made of heat-resistant glass and stainless steel, conforming to the dimensions given in Fig. A2.1. A suggested assembly is shown in Fig. A2.2.

A2.2 Procedure

A2.2.1 Clean and dry the apparatus. Pipet 10 mL of aniline (**Warning**— see Note 3) and 10 mL of the dried sample (8.1) into the tube fitted with pump-stirrer and thermometer. If the material is too viscous for pipetting, weigh to the nearest 0.01 g a quantity of sample corresponding to 10 mL at room temperature. Place the thermometer in the tube so that the contraction chamber is below the liquid level and so that the mercury bulb does not touch the side of the tube. Assemble the apparatus as shown in Fig. A2.2.

A2.2.2 Adjust the speed of the pump to produce a continuous stream of the oil-aniline mixture in the form of a thin film flowing over the light well. With extremely dark oils, operate the pump slowly and lower it so that the delivery tube nearly touches the top of the light well, so as to obtain a continuous film thin enough to permit observation of the aniline point. Adjust the voltage on the lamp until just enough light is given for the filament to be visible through the film. Raise the temperature of the mixture at a rate of 1 to 2°C (2.0 to 3.5°F)/min until the aniline point has just been passed, as denoted by a definite, sudden brightening of the lamp filament, and by the disappearance of the more or less opalescent condition of the film (Note A2.1). Discontinue heating and adjust the lamp voltage so that the filament appears clear and distinct but not uncomfortably bright to the eye. Adjust the temperature of the bath so that the sample-aniline mixture cools at a rate of 0.5 to 1.0°C (1.0 to 1.8°F)/min and note the appearance of the film and light filament. Record as the aniline point the temperature at which a second phase appears as evidenced by the reappearance of the opalescent condition of the film (usually causing a halo to appear around the lamp filament) or by a sudden dimming of the lamp filament, or both. At temperatures above the aniline point the edges of the light filament appear clear and distinct. At the aniline point temperature a halo or haze forms around the filament, replacing the distinct lines of the filament edge with lines that appear cloudy or hazy in appearance. Further darkening of the cloud over the filament occurs at lower temperature, but is not to be confused with the aniline point.

NOTE A2.1—For those making the test for the first time, the following procedure may be helpful: Make preliminary operational adjustments and tests using a colorless sample-aniline mixture, and observing changes taking place in the body of the liquid and film. Make rough tests with dark oils to become familiar with the appearance of the film and light source as the mixture passes from the clear state above the aniline point, to the



DETAIL OF TUBE DETAIL OF PUMP BODY AND ROTOR

FIG. A2.1 Details of Aniline Point Thin-Film Apparatus (Method B)

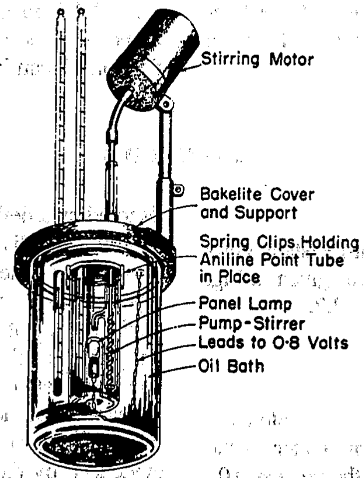


FIG. A2.2 Assembly of Thin-Film Apparatus (Method B)

translucent state below. If the sample is such that there is difficulty in observing the exact point of the phase change, make experiments with the sample, using various intensities of light and paying particular attention to the appearance of the light in the immediate vicinity of the lamp filament.

A2.2.3 Repeat the observation of aniline point temperature by heating and cooling repeatedly until a report as directed in Section 11 can be made.

A3. METHOD C

A3.1 Apparatus

A3.1.1 *Aniline-Point Tube*, of heat-resistant glass, of the shape and dimensions shown in Fig. A3.1, and fitted internally with a thin-walled glass thermometer tube, sealed at the lower end. The latter tube accommodates a tight-fitting cork stopper carrying the thermometer, the bulb of which rests on a cork ring or disk placed at the bottom of the tube; the tube contains sufficient light transformer oil to cover the bulb of the thermometer. The inner tube is held in the top of the aniline-point tube by a tightly fitting stopper, and a clamp is provided to hold the stopper in position to prevent loss of vapor from the sample.

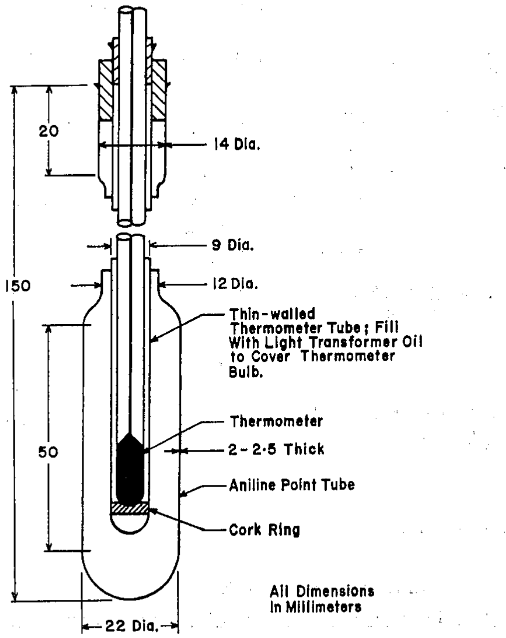


FIG. A3.1 Apparatus for Volatile Samples (Method C)

NOTE A3.1—Any other suitable arrangement, such as a screwed plastic gland carrying the thermometer, that will prevent the loss of vapor from the apparatus, may be used. In such cases it may be possible to omit the thermometer tube and immerse the thermometer bulb in the aniline-sample mixture.

A3.1.2 *Guard*, of stout metal gauze and surrounding the aniline point tube. It should preferably be combined with the clamp for holding the thermometer tube in place.

A3.2 Procedure

A3.2.1 Clean and dry the apparatus. Pipet 5 mL of aniline (Note A3.2 Precaution see Note 3) and 5 mL of the dried sample (8.1), both cooled to a temperature at which the sample may be measured without loss of vapor. Close the tube by means of the stopper and fit the thermometer tube centrally so that the bottom is 5 mm from the bottom of the aniline point tube. Clamp the stopper in position and attach the guard.

NOTE A3.2—**Precaution:** Put on goggles of safety glass and plastic gloves impervious to aniline.

A3.2.2 Follow the procedure described in A1.2.2 and A1.2.3 but mix the sample and aniline by shaking the tube. If the rate of change of temperature is greater than 1°C (2°F)/min when the aniline point is being approached, place the tube in a jacket that has previously been warmed or cooled to an appropriate temperature.

A3.2.3 Repeat the observation of aniline point by heating and cooling repeatedly until a report as directed in Section 11 can be made.

A4. METHOD D

A4.1 Apparatus

A4.1.1 *Bulb*, 1.5 to 2.0-mL capacity, blown from heat-resistant glass tubing, 5 mm in external diameter and 3 mm in internal diameter.

A4.1.2 *Guard*, as for Method C.

A4.2 Procedure

A4.2.1 Dry the bulb thoroughly in an oven at 105 ± 5°C, allow it to cool to room temperature, and charge it by means of the pipets with 0.5 mL of aniline (**Warning**— see Note 3) and 0.5 mL of the dried sample (8.1). Cool the mixture thoroughly and rapidly draw out and seal the open end of the bulb at about 10 mm from the center of the bulb.

NOTE A4.1—**Precaution:** Put on goggles of safety glass and plastic gloves impervious to aniline.

A4.2.2 Attach the bulb to the thermometer by rubber bands so that the bulb is adjacent to the thermometer bulb. Attach the mesh guard and follow the procedure described in A1.2.2 and A1.2.3 but mix the sample and aniline by shaking.

A4.2.3 Repeat the observation of aniline point temperature by heating and cooling repeatedly until a report as directed in Section 11 can be made.

A5. METHOD E

A5.1 Apparatus

A5.1.1 *Automatic Aniline Point Apparatus*, commercially available, using a modified thin film technique and direct heating of the sample-aniline mixture with electrical immersion heater. Detection of change of sample turbidity at the aniline point is by response of a photoelectric cell to collimated light directed through the thin film of sample.

A5.2 Procedure

A5.2.1 Determine the automatic aniline point in accordance with instructions provided with the apparatus. Correct the aniline point as follows:

$$\text{Corrected aniline point} = (X_a - A)/B \qquad (A5.1)$$

where:

X_a = automatic aniline point, and

A and B = constants determined for each apparatus as described in A5.2.2

NOTE A5.1—It has been established by cooperative tests that observed aniline points determined by some automatic apparatus are lower than the determinations by Methods A and B. The difference is greater for automatic apparatus when relatively high sample-cooling rates are used, and increases as the aniline point increases.

A5.2.2 Determine the aniline point by either Method A or Method B and also using the automatic apparatus for three or more samples with aniline points in each of the ranges 43 to 49°C (110 to 120°F), 60 to 66°C (140 to 150°F), and 77 to 82°C (170 to 180°F). Calculate the constants A and B by the least squares method by simultaneous solution of the following equations:

$$\Sigma(X_a) = NA + B\Sigma(X_c) \qquad (A5.2)$$

$$\Sigma(X_aX_c) = A\Sigma(X_c) + B\Sigma(X_c^2)$$

where:

$\Sigma(X_a)$ = sum of all aniline point data by automatic apparatus,

$\Sigma(X_c)$ = sum of all aniline point data by either Method A or B,

$\Sigma(X_c^2)$ = sum of the squares of all aniline point data by either Method A or B,

$\Sigma(X_aX_c)$ = sum of the products of aniline points determined by either Method A or B and by using the automatic apparatus for each sample, and

N = number of samples.

NOTE A5.2—Cooperative data were obtained from five laboratories for five samples with aniline points in the range from 34 to 87°C (93 to 188°F). Constants A and B were calculated for the composite data as 0.79 and 0.991 respectively. Although a minimum number of nine samples is specified in this method, constants A and B in the preceding equation may be obtained with a slightly greater precision if data for a larger number of samples are used.

A6. PRECAUTIONARY STATEMENT

A6.1 *n*-Heptane

Warning—Flammable. Harmful if inhaled.
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D665: Standard Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water

CFR Section(s): 46 CFR 61.20-17(a)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 665 – 98$^{\epsilon 1}$

An American National Standard

Designation: 135/93

Standard Test Method for Rust-Preventing Characteristics of Inhibited Mineral Oil in the Presence of Water[1]

This standard is issued under the fixed designation D 665; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

ϵ^1 Note—Paragraph 6.3.2 was corrected editorially in August 1999.

1. Scope

1.1 This test method is used to evaluate the ability of inhibited mineral oils, particularly steam-turbine oils, to aid in preventing the rusting of ferrous parts should water become mixed with the oil. This test method is also used for testing other oils, such as hydraulic oils and circulating oils. Provision is made in the procedure for testing heavier-than-water fluids.

Note 1—For synthetic fluids, such as phosphate ester types, the plastic holder and beaker cover should be made of a chemically resistant material, such as polytetrafluoroethylene (PTFE).

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

A 108 Specification for Steel Bars, Carbon, Cold-Finished, Standard Quality[2]

A 240/A 240M Specification for Heat-Resisting Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels[3]

D 91 Test Method for Precipitation Number of Lubricating Oils[4]

D 1193 Specification for Reagent Water[5]

D 1401 Test Method for Water Separability of Petroleum Oils and Synthetic Fluids[4]

D 2422 Classification of Industrial Fluid Lubricants by Viscosity System[4]

D 3603 Test Method for Rust-Preventing Characteristics of Steam Turbine Oil in the Presence of Water (Horizontal Disk Method)[6]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[6]

E 1 Specification for ASTM Thermometers[7]

Motor Fuels, Section I, Annex A2, Reference Materials and Blending Accessories[8]

2.2 *Other Documents:*

Specifications-IP Standard Thermometers, Appendix A[9]

Specifications for IP Standard Reference Liquids, Appendix B[9]

SAE Standard J 405 Chemical Composition of SAE Wrought Stainless Steels[10]

BS 871 Specification for abrasive papers and cloths[9]

BS 970: Part 1: Carbon and Carbon Manganese Steels Including Free Cutting Steels[9]

3. Summary of Test Method

3.1 A mixture of 300 mL of the oil under test is stirred with 30 mL of distilled water or synthetic sea water, as required, at a temperature of 60°C (140°F) with a cylindrical steel test rod completely immersed therein. It is customary to run the test for 24 h; however, the test period may, at the discretion of the contracting parties, be for a shorter or longer period. The test rod is observed for signs of rusting and, if desired, degree of rusting.

4. Significance and Use

4.1 In many instances, such as in the gears of a steam turbine, water can become mixed with the lubricant, and

---

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee DO2.C on Turbine Oils.

In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM – IP standard in 1964.

Current edition approved June 10, 1998. Published September 1998. Originally published as D 665 – 42 T. Last previous edition D 665 – 95.

[2] *Annual Book of ASTM Standards*, Vol 01.05.

[3] *Annual Book of ASTM Standards*, Vol 01.03.

[4] *Annual Book of ASTM Standards*, Vol 05.01.

[5] *Annual Book of ASTM Standards*, Vol 11.01.

[6] *Annual Book of ASTM Standards*, Vol 05.02.

[7] *Annual Book of ASTM Standards*, Vol 14.03.

[8] *Annual Book of ASTM Standards*, Vol 05.04.

[9] *Annual Book of IP Standard Methods for Analysis and Testing of Petroleum and Related Products*, Vol 2.

[10] *1995 SAE Handbook*, Vol 1.

rusting of ferrous parts can occur. This test indicates how well inhibited mineral oils aid in preventing this type of rusting. This test method is also used for testing hydraulic and circulating oils, including heavier-than-water fluids. It is used for specification of new oils and monitoring of in-service oils.

NOTE 2—This test method was used as a basis for Test Method D 3603. Test Method D 3603 is used to test the oil on separate horizontal and vertical test rod surfaces, and can provide a more discriminating evaluation.

5. Apparatus

5.1 *Oil Bath*—A thermostatically controlled liquid bath capable of maintaining the test sample at a temperature of 60 ± 1°C (140 ± 2°F) (see Note 3). An oil having a viscosity of approximately ISO VG 32 (see Classification D 2422) is suitable for the bath. The bath shall have a cover with holes to accommodate the test beakers.

NOTE 3—ASTM Thermometer 9C (9F), as prescribed in Specification E 1, or IP Thermometer 21C in accordance with IP Volume 2, Appendix A, is suitable to indicate the temperature. Alternatively, calibrated thermocouples may be used.

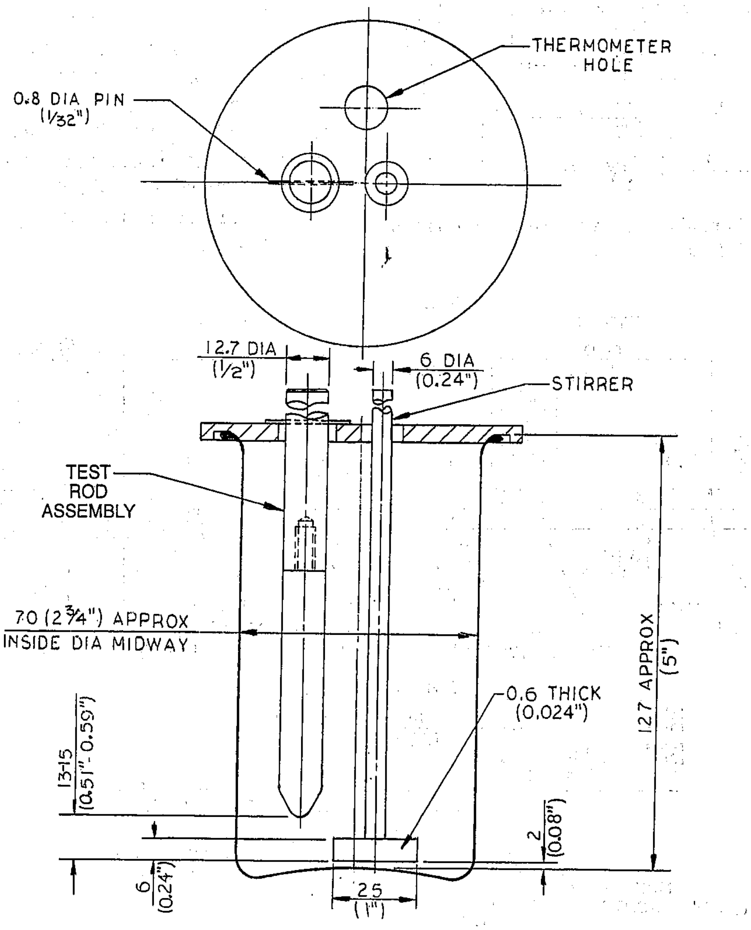
5.2 *Beaker*—A 400-mL, Berzelius-type, tall-form heat-resistant glass[11] beaker without pourout, as shown in Fig. 1, approximately 127 mm (5 in.) in height measured from the inside bottom center and approximately 70 mm (2¾ in.) in inside diameter measured at the middle.

5.3 *Beaker Cover*— A flat beaker cover of glass or poly(methyl methacrylate) (PMMA) (Note 4), kept in position by suitable means such as a rim or groove. Two holes shall be provided on any diameter of the cover; one for a stirrer 12 mm (15⁄32 in.) in diameter with its center 6.4 mm (¼ in.) from the center of the cover; and the other, on the opposite side of the center of the cover, for the test rod assembly (see Section 8), 18 mm (45⁄64 in.) in diameter with its center 16 mm (⅝ in.) from the center of the cover. In addition, a third hole 12 mm (15⁄32 in.) in diameter shall be provided for a temperature measuring device, with its center 22.5 mm (⅞ in.) from the center of the cover and on a diameter of the cover at right angles to the diameter through the other two holes.

NOTE 4—An inverted petri dish makes a suitable cover, as the sides of the dish aid in keeping it in position. Fig. 2 shows a PMMA resin cover

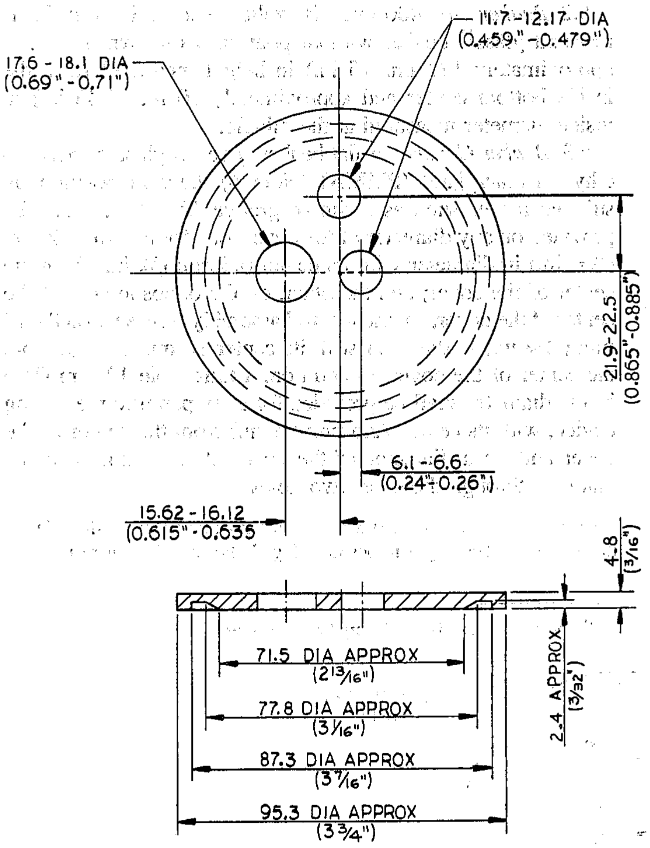
---

[11] Borosilicate glass is satisfactory for this purpose.



NOTE 1—All units are in millimetres, unless otherwise specified.

FIG. 1 Rusting Test Apparatus



NOTE 1—All units are in millimetres, unless otherwise specified.

FIG. 2 Beaker Cover

for the beaker which has been found to be suitable. An optional feature is shown, consisting of a slot, 1.6 by 27 mm (1/16 by 1 1/16 in.), which is centered on a diameter of the stirrer hole at right angles to the cover diameter through the test rod hole and stirrer hole. This feature allows withdrawal of the stirrer while the beaker cover is in place. When the test method test is used for other fluids such as synthetics, the beaker cover should be made from chemically resistant material such as polymonochlorotrifluoroethylene (PCTFE).

5.4 The plastic holder shall be made of PMMA resin in accordance with the dimensions shown in Fig. 3 (two types of holders are illustrated). When testing synthetic fluids, the plastic holder should be made from chemically resistant material such as polytetrafluoroethylene (PTFE).

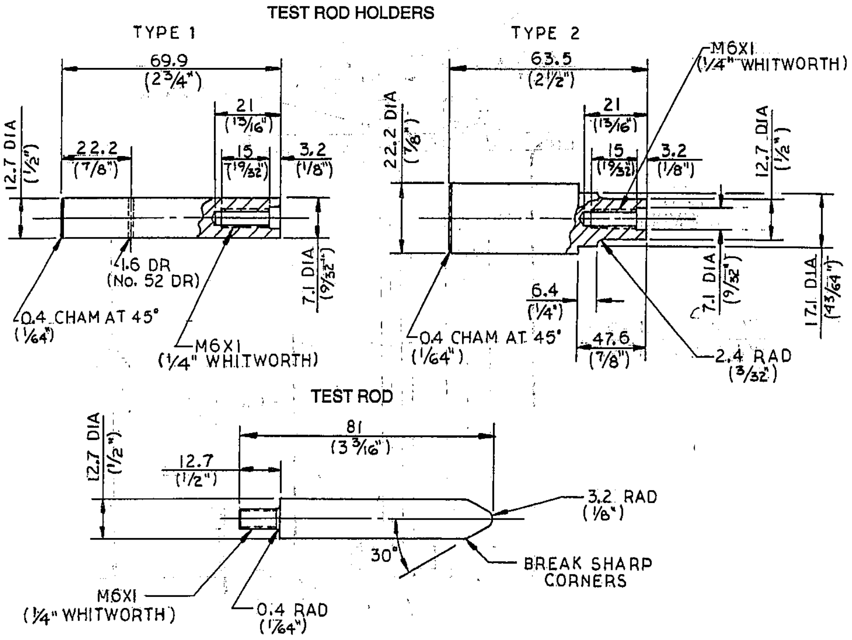
5.5 *Stirrer*—A stirrer constructed entirely from stainless steel (Note 5 and Note 6) in the form of an inverted T. A flat blade 25 by 6 by 0.6 mm (1 by 0.24 by 0.024 in.) shall be attached to a 6-mm (0.24-in.) rod in such a way that the blade is symmetrical with the rod and has its flat surface in the vertical plane.

NOTE 5—A suitable material is an 18 % chromium, 8 % nickel alloy steel conforming to Type 304, of Specification A 240, or SAE No. 30304 (see SAE J405), or BS 970: Part 1: 1983: 302S31.[9]

NOTE 6—If stainless steel is not obtainable, stirrers made of heat-resistant glass[11] and having approximately the same dimensions as the stainless steel stirrers specified can be used.

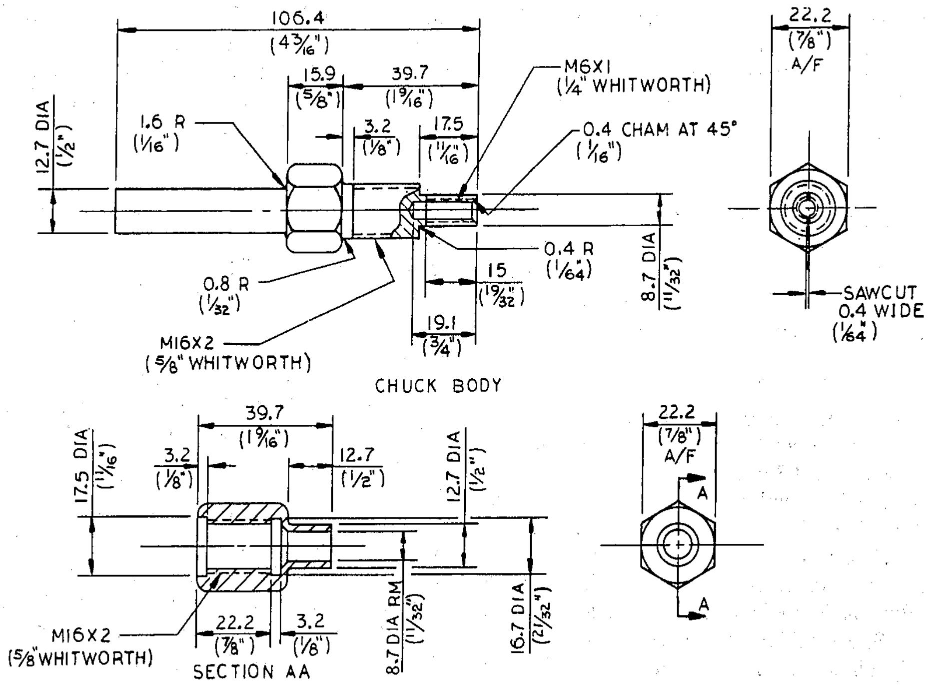
5.6 *Stirring Apparatus*—Any convenient form of stirring apparatus capable of maintaining a speed of 1000 ± 50 rpm.

5.7 *Grinding and Polishing Equipment*—A 150-grit[9] (99-μm) and a 240-grit[9] (58-μm) in accordance with BS 871 or its equivalent, metalworking aluminum oxide abrasive cloth coat on a jeans backing, a suitable chuck (see Fig. 4) for holding the test rod, and a means of rotating the test rod at a speed of 1700 to 1800 rpm.



NOTE 1—All units are in millimetres, unless otherwise specified.

FIG. 3 Test Rod and Holders



NOTE 1—All units are in millimetres, unless otherwise specified.

FIG. 4 Chuck for Polishing Test Rods

5.8 *Oven*, capable of maintaining a temperature of 65°C (150°F).

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[12] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

6.3 The synthetic sea water shall have the following composition:

Salt	g/L
NaCl	24.54
$MgCl_2 \cdot 6H_2O$	11.10
Na_2SO_4	4.09
$CaCl_2$	1.16
KCl	0.69
$NaHCO_3$	0.20
KBr	0.10
H_3BO_3	0.03
$SrCl_2 \cdot 6H_2O$	0.04
NaF	0.003

6.3.1 The solution can be conveniently prepared as follows. This procedure avoids any precipitation in concentrated solutions with subsequent uncertainty of complete resolution. Using certified pure (cp) chemicals and distilled water, prepare the following stock solutions (also see Test Method D 1401, Section 6):

Stock Solution No. 1:

$MgCl_2 \cdot 6H_2O$	3885 g
$CaCl_2$(anhydrous)	406 g
$SrCl_2 \cdot 6H_2O$	14 g

Dissolve and dilute to 7 L

Stock Solution No. 2:

KCl	483 g
$NaHCO_3$	140 g
KBr	70 g
H_3BO_3	21 g
NaF	2.1 g

Dissolve and dilute to 7 L

6.3.2 To prepare the synthetic sea water, dissolve 245.4 g of NaCl and 40.94 g of Na_2SO_4 in a few litres of distilled water, add 200 mL of Stock Solution No. 1 and 100 mL of Stock Solution No. 2 and dilute to 10 L. Stir the 10-L batch and add 0.1 N Na_2CO_3 solution until the pH is between 7.8 and 8.2. One or two millilitres of the carbonate solution will be required.

6.4 *Precipitation Naphtha*, as specified in Test Method D 91. (**Warning**—Flammable. Health hazard.)

6.5 *Isooctane*, as specified in Table 32, Motor Fuels Section

---

[12] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

I, Annex A2, Reference Materials and Blending Accessories. (**Warning**—Flammable. Health hazard.)

6.6 *IP 60/80 Petroleum Spirit*, as specified by IP Standard Reference Liquids. (**Warning**—Flammable. Health hazard.)

6.7 *Steel Test Rod*, as specified in Section 8.

7. Sampling

7.1 Sample for this test can come from tanks, drums, small containers, or even operating equipment, and they should be truly representative of the entire quantity. Therefore, use the applicable apparatus and techniques described in Practice D 4057, or other comparable standard practice.

8. Test Rod and Its Preparation

8.1 For each test oil, prepare two steel test rods. These may either be new or from a previous test (seeNote 7) and shall be prepared in accordance with 8.2 and 8.3.

8.2 The test rod assembly shall consist of a round steel test rod fitted to a plastic holder. The round steel test rod when new shall be 12.7 mm (0.5 in.) in diameter and approximately 68 mm ($2^{11}/_{16}$ in.) in length exclusive of the threaded portion which screws into the plastic holder and shall be tapered at one end as shown in Fig. 3. It shall be made of steel conforming to Grade 10180 of Specification A 108 or to BS 970 Part I: 1983-070M20. If these steels are not available, other equivalent steels may be used, provided they are found to be satisfactory by comparative test using this Test Method D 665 – IP 135.

NOTE 7—When making a check test, the steel test rod that showed rust should not be reused. Test rods that repeatedly show rust in tests of various oils can be imperfect. Such test rods should be used with oils known to pass the test. If rusting occurs in repeat tests, these test rods should be discarded.

8.3 *Preliminary Grinding*—If the test rod has been used previously and is free of rust or other irregularities, the preliminary grinding may be omitted, and it may be subjected only to final polishing as prescribed in 8.4. If the test rod is new or if any part of its surface shows rust or other irregularities, clean it with ASTM precipitation naphtha or *iso*octane or IP 60/80 petroleum spirit and grind with medium 150-grit aluminum oxide cloth to remove all irregularities, pits, and scratches, as determined by visual inspection (Note 8). Perform the grindings by mounting the test rod in the chuck of the grinding and polishing apparatus, and turning it at a speed of 1700 to 1800 rpm while applying the 150-grit aluminum oxide cloth. Old 150-grit aluminum oxide cloth may be used to remove rust or major irregularities, but complete the grinding with new cloth. Proceed at once with the final polishing with 240-grit aluminum oxide cloth, or remove the test rod from the chuck and store in *iso*octane until needed. Discard reused test rods when the diameter is reduced to 9.5 mm (0.375 in.).

NOTE 8—Do not touch the test rods with the hands at any stage after cleaning with naphtha or *iso*octane or the petroleum spirit (which precedes either preliminary grinding or final polishing) until the test is completed. Forceps or a clean, lintless cloth may be used.

8.4 *Final Polishing*:

8.4.1 Just before the test is to be made, subject the test rod to final polishing with 240-grit aluminum oxide cloth. If the preliminary grinding has just been completed, stop the motor that rotates the test rod. Otherwise, remove the test rod from the *iso*octane (previously used unrusted test rods shall be stored in this reagent), dry with a clean cloth, and place in the chuck. Rub a new piece of 240-grit aluminum oxide cloth longitudinally over the static test rod until the rounded end, and the entire surface show visible scratches. Rotate the test rod at a speed of 1700 to 1800 rpm. Take the cloth and place it halfway around the test rod, and apply a firm but gentle downward pull to the loose ends of the cloth for about 1 to 2 min so as to produce a uniform finely scratched surface free of longitudinal scratches. Carry out the final stages of the polishing with new cloth.

8.4.2 To ensure that the flat shoulder (that portion of the test rod perpendicular to the threaded stem) is free of rust, polish this area. This can be done by holding a strip of 240-grit aluminum oxide cloth between the chuck and the shoulder while rotating the test rod for a brief period.

8.4.3 Remove the test rod from the chuck without touching with the fingers; wipe lightly with a clean, dry, lintless cloth or tissue (or brush the test rod lightly with a camel's hair brush); attach to the plastic holder; and immediately immerse in the oil to be tested. This can be either the hot oil sample (see 9.1) or a clean test tube containing a portion of the sample. The test rod can be removed later from this tube and allowed to drain briefly before being placed in the hot oil.

9. Procedure A for Distilled Water

9.1 Clean the beaker in accordance with good laboratory procedure, wash with distilled water, and dry in an oven. Clean glass beaker cover and a glass stirrer by the same procedure. To clean a stainless steel stirrer and a PMMA cover, use ASTM precipitation naphtha or *iso*octane or IP 60/80 petroleum spirit, wash thoroughly with hot water and finally with distilled water, and dry in an oven at a temperature not over 65°C (150°F). Pour 300 mL of the oil to be tested (see Practice D 4057) into the beaker and place the beaker in the oil bath held at a temperature that will maintain the oil sample at 60 ± 1°C (140 ± 2°F). Insert the beaker into a hole of the bath cover and suspend in the hole with the beaker rim resting on the bath cover. The oil level in the bath shall not be below the oil level in the test beaker. Cover the beaker with the beaker cover with the stirrer in position in the proper opening. Adjust the stirrer so that the shaft is 6 mm (0.24 in.) off center in the beaker containing the oil sample and the blade is not more than 2 mm (0.08 in.) from the bottom of the beaker. Then suspend a temperature measuring device through the hole in the cover intended for that purpose so that it is immersed to a depth of 56 mm (2.2 in.). Start the stirrer and when the temperature reading reaches 60 ± 1°C (140± 2°F), insert the steel test rod prepared in accordance with Section 8.

9.2 Insert the test rod assembly through the test rod hole in the beaker cover and suspend so that its lower end is 13 to 15 mm (0.51 to 0.59 in.) from the bottom of the beaker. Either type of plastic test rod holder (see Fig. 3) may be used. The hole through which the test rod is suspended shall be unobstructed (see Note 9).

NOTE 9—Fig. 1 shows the arrangement of the apparatus.

9.3 Continue stirring for 30 min to ensure complete wetting of the steel test rod. With the stirrer in motion, remove the temperature measuring device temporarily and add 30 mL of distilled water through this hole, discharging the water on the bottom of the beaker, and replace the temperature measuring device. Continue stirring at a speed of 1000 ± 50 rpm for 24 h (see Note 10) from the time water was added, maintaining the temperature of the oil-water mixture at 60 ± 1°C (140 ± 2°F). Stop stirring at the end of the 24-h period, remove the test rod, allow to drain, and then wash with ASTM precipitation naphtha or *iso*octane, or IP 60/80 petroleum spirit. If desired, the test rod may be preserved by lacquering.

Note 10—In general, observations of rusting made at the end of the 12-h testing time are indicative as to whether the oil will pass or fail. It is customary to run the test for 24 h; however, the test period may, at the discretion of the contracting parties, be for a shorter or longer period.

10. Procedure B for Synthetic Sea Water

10.1 The procedure for rust-preventing characteristics of inhibited mineral oils in the presence of synthetic sea water shall be identical with that described in 9.1, 9.2, and 9.3, except use synthetic sea water in place of distilled water in that portion of the procedure described in 9.3.

11. Procedure C for Heavier-Than-Water Fluids

11.1 The stirring action provided by the stirrer prescribed in 5.4 is not sufficient to mix thoroughly the water and test sample when the fluid under test is heavier than water. This section is meant to define changes in the test method to accommodate heavier-than-water fluids. Except as specifically stated, all the requirements of the previous Sections 1-10 shall apply. Since this procedure can be performed with distilled water or synthetic sea water, take care to include this information in the report.

11.2 *Apparatus*:

11.2.1 *Beaker Cover*— Same as prescribed in 5.3 (see Note 11).

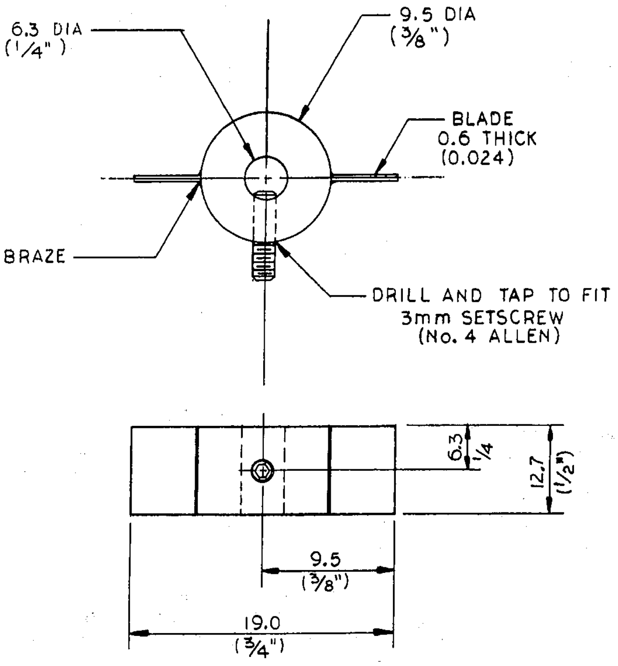
Note 11—Some heavier-than-water fluids can attack or dissolve PMMA beaker covers and test rod holders. It is recommended that PCTFE beaker covers and PTFE test rod holders be used when testing heavier-than-water fluids.

11.2.2 *Stirrer*—Same as prescribed in 5.4 but with an auxiliary blade attached to the stirrer shaft. The auxiliary blade shall be of stainless steel, 19.0 by 12.7 by 0.6 mm (0.75 by 0.50 by 0.25 in.) as shown in Fig. 5. The auxiliary blade shall be positioned on the stirrer shaft so that the bottom edge of the auxiliary blade is 57 mm (2.25 in.) above the top edge of the fixed lower blade, and so that the flat surfaces of both blades are in the same vertical plane.

11.3 *Test Rod and Its Preparation*—Same as prescribed in Section 8.

12. Interpretation of Results

12.1 Perform all inspections at the end of the test to determine the condition of test rods without magnification under normal light. For the purpose of this test, normal light is considered to be illumination of about 60 footcandles (650 lx). Within the meaning of this test method, a rusted test rod is one on which any rust spot or rust streak is visible by the above inspection procedure.



Note 1—All units are in millimetres, unless otherwise specified.

FIG. 5 Auxiliary Stirrer Blade (not to scale)

12.2 For the purpose of this test method, rust is an area of corrosion of the test surface that is identified by color and is confirmed by the presence of pits or roughness if the surface is wiped with a lintless cloth or tissue paper. Neither surface discoloration nor specks, which can easily be removed with a lintless or tissue paper cloth with no evidence of pitting or roughness, shall be considered to be rust.

12.3 In order to report an oil as passing or failing, conduct the test in duplicate. Report an oil as passing the test if both test rods are rust-free at the end of the test period. Report an oil as failing the test if both test rods are rusted at the end of the test period (see Note 12). If one test rod is rusted while the other is free of rust, test two additional test rods (see Note 7). If either of these latter test rods shows rusting, report the oil as not passing the test. If neither of these latter test rods shows rusting, report the oil as passing the test.

Note 12—An indication of the degree of rusting occurring in this test may be desired. For uniformity in such cases, use of the following classifications of rusting severity is recommended:

Light Rusting—Rusting confined to not more than six spots, each of which is 1 mm or less in diameter.

Moderate Rusting—Rusting in excess of the above but confined to less than 5% of the surface of the test rod.

Severe Rusting—Rusting covering more than 5% of the surface of the test rod.

12.4 A reference oil with a Pass in Procedure A and a Fail in Procedure B can be prepared as follows: Add 0.0150 mass %

of an additive concentrate[13] to a white mineral oil.[14] The additive concentrate consists of 60 mass % of dodecenyl succinic acid and 40 mass % of normal paraffin oil, ISO VG 22 (see Classification D 2422).[15]

13. Report

13.1 The test report shall contain the following:

13.1.1 The type and identification of the product used.

13.1.2 The date of the test.

13.1.3 A reference to this ASTM-IP standard, indicating whether Procedure A, B, or C was used. Since Procedure C may be followed with either distilled water or sea water, be sure to include the type of water used in this case.

13.1.4 The duration of the tests.

13.1.5 Any deviation from the procedure specified.

13.1.6 The result of the test, including degree of severity of rusting, if desired.

14. Precision and Bias

14.1 *Precision*—It is not practicable to specify the precision of the procedure in this test method because the results of the test are only intended to give a pass/fail rating to the oil being tested.

14.2 *Bias*—The procedure in this test method has no bias because the value of rust-preventing characteristics is defined only in terms of this test method.

15. Keywords

15.1 circulating oils; heavier-than-water fluids; hydraulic oils; inhibited mineral oil; rust-preventing characteristics; steam-turbine oils

---

[13] A suitable additive concentrate is Lubrizol 850, as used in the ASTM round robin. Lubrizol 850 is available from Lubrizol Corporation, Wickliffe, OH.

[14] A suitable white mineral oil is USP Mineral Oil with a viscosity in the order of ISO VG 32 (Penreco Drakeol 19 was used in the ASTM round robin and is available from Penreco, Karns City, PA).

[15] The results of the cooperative test program using this reference oil are available from ASTM Headquarters as RR:D02-1284.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name:	ASTM D814: Standard Test Method for Rubber Property--Vapor Transmission of Volatile Liquids
CFR Section(s):	40 CFR 1051.245(e)(1)
Standards Body:	American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 814 – 95

Standard Test Method for Rubber Property— Vapor Transmission of Volatile Liquids[1]

1. Scope

1.1 This test method covers the measurement of the rate of vapor transmission of volatile liquids through a rubber sheet, disk, or diaphragm.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard:*

D 3767 Practice for Rubber—Measurement of Dimensions[2]

3. Significance and Use

3.1 The rate of transmission of a liquid through a rubber sheet, that acts as a barrier, is important in rubber diaphragm performance and other similar industrial applications. This transmission is referred to as vapor transmission since the liquid diffuses through the rubber in a molecular sense and escapes into free atmosphere in vapor form.

3.2 This test method is applicable only to the materials in sheet form of moderate thickness and is principally useful for comparing the relative vapor transmission of different liquids in the same rubber or of the same liquid through different rubbers. Comparisons should not be made in which both different rubbers and different liquids are used.

4. Apparatus

4.1 The test apparatus shall consist essentially of glass vapor transmission jars (4.2) for each liquid, on which the test specimens are mounted, and a suitable rack for supporting the jars in an inverted position in such a way as to allow free circulation of air over the surface of the specimen. The apparatus shall be kept in a constant temperature cabinet or room at 23 ± 2°C (73.4 ± 3.6°F).

4.2 *Vapor Transmission Jar*—A glass jar of approximately 236-cm^3 (½-pt) capacity with an opening of 60.3 ± 0.4 mm (2.375 ± 0.015 in.) in inside diameter and a wall 3.2 mm (0.125 in.) in thickness at the opening, which shall be ground flat without rounded edges to a smooth ground-glass finish at the contact surface. The opening shall be equipped with a metal screw ring clamp holding a smooth-edged, flat, sheet-metal ring 55.6 ± 0.4 mm (2.188 ± 0.015 in.) in inside diameter and approximately 68.3 mm (2.69 in.) in outside diameter. The ring clamp and ring serve as a clamping device for holding the specimen against the ground-glass-edge of the jar with a circular specimen area 55.6 mm (2.188 in.) in diameter exposed to the air while permitting the screw clamp to be tightened to form a leak-proof seal without subjecting the specimen to torsional strain. When testing extensible materials, a circular disk of 16 to 20-mesh screen shall be placed inside the screw ring clamp between it and the flat metal ring to prevent stretching of the specimen during the test. The screen disk may be soldered in place on top of the sheet metal ring if desired.

NOTE 1—A suitable vapor transmission jar may be prepared from a ½-pt Mason jar equipped with a Kerr-type screw cap, provided the edge of the jar is properly ground and finished with fine emery and the cap is cut with smooth, flattened edges to form the required circular opening.

4.3 *Dial Micrometer*—The dial micrometer shall conform to the requirements of Method A of Practice D 3767.

4.4 *Balance*—The balance shall be of suitable capacity capable of weighing to 0.005 g.

5. Test Specimens

5.1 The test specimens shall consist of circular disks 68 mm (2.69 in.) in diameter, cut with a sharp-edged die from a sheet of the material being tested. The thickness of the sheet, which shall not exceed 3.2 mm (0.125 in.), shall be measured using the dial micrometer described in 4.3.

5.2 For evaluating the vapor transmission of a particular liquid with respect to a given rubber, three specimens having the same nominal thickness within a tolerance of ±3 % shall be tested. The vapor transmission of the liquid shall be taken as the average of the values obtained from the two rubber specimens giving the lowest results, providing these check within ±20 % of the average so determined.

6. Procedure

6.1 With the vapor transmission jar in an upright position, place 200 cm^3 of the test liquid in it, after which fit the test specimen over the mouth of the jar, place the ring and wire screen in position, and screw down the screw ring clamp lightly. Hold the assembly in the constant temperature cabinet or room at 23 ± 2°C (73.4 ± 3.6°F) with the test disk lightly in place until temperature equilibrium is obtained. Then screw down the ring tightly and allow the assembly to stand in an upright position for 2 h. Make a tare assembly using a duplicate jar and a test specimen of the same size but without the addition of the test liquid. Place suitable weighting material such as lead shot in the tare jar to bring its

---

[1] This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

Current edition approved May 15, 1995. Published July 1995. Originally published as D 814 – 44 T. Last previous edition D 814 – 86 (1991)$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

mass to approximately that of the assembled jar and contents. Condition the tare jar at the test temperature, seal it, and handle throughout the test in a manner similar to that used for the test jar. Place the test jar on a balance of suitable capacity capable of weighing to 0.005 g, and counterbalance it against the tare jar, adding weights as required for equilibrium. Take the original mass of the test assembly as that of the tare corrected for the added weights and record to the nearest 0.005 g.

6.2 After weighing, invert each test jar assembly and place on the rack, supporting each in such a way that the entire inner surface of the specimen remains in contact with the liquid throughout the test. If the rack is located in a conditioning cabinet, take care to remove diffused vapors from the chamber and to assure free circulation of fresh air by means of a fan or other suitable device. Twenty-four hours after inverting the jar, again weigh it against the tare jar. Any excessive loss of mass indicates that leakage due to improper seal is occurring. Remove such test assemblies and replace them by new assemblies properly prepared. Return the test assembly to the rack and reweigh against the tare after periods of 120 ± 2 h and 192 ± 2 h from the initial inversion. Consider the difference in mass between those obtained at 120 h (5 days) and at 192 h (8 days) divided by 3 as the average mass of liquid lost per 24 h.

NOTE 2—In this test, the specimens are allowed to remain in contact with the liquid for 5 days before starting measurement of the loss in order to permit the rate of diffusion of the liquid to become uniform.

7. Calculation

7.1 Express the vapor transmission rate (VTR) of each test liquid with respect to the rubber employed and calculate as follows:

$$\text{Vapor transmission rate (VTR), mg/(s}\cdot\text{m}^2) = 4.77 \times \text{average mass in grams of liquid lost per 24 h}$$

8. Report

8.1 The report shall include the following:

8.1.1 Identification of the test rubber,

8.1.2 Thickness of the test rubber to the nearest 0.025 mm (0.001 in.),

8.1.3 Identification of the test liquid, and

8.1.4 Vapor transmission rate (VTR) of the liquid, expressed in milligrams per second per square metre of wetted area.

9. Precision

9.1 Due to a lack of laboratories willing to participate in round-robin testing, no precision data are available for this test method.

10. Keywords

10.1 vapor transmission; volatile liquids

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *__HEED THIS NOTICE__*: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM D975: Standard Specification for Diesel Fuel Oils

CFR Section(s): 46 CFR 160.176-13(r)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 975 – 98b

An American National Standard

Standard Specification for Diesel Fuel Oils[1]

This standard is issued under the fixed designation D 975; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers five grades of diesel fuel oils suitable for various types of diesel engines. These grades are described as follows:

1.1.1 *Grade Low Sulfur No. 1-D*—A special-purpose, light distillate fuel for automotive diesel engines requiring low sulfur fuel and requiring higher volatility than that provided by Grade Low Sulfur No. 2-D.[2]

1.1.2 *Grade Low Sulfur No. 2-D*— A general-purpose, middle distillate fuel for automotive diesel engines requiring low sulfur fuel. It is also suitable for use in non-automotive applications, especially in conditions of varying speed and load.[2]

1.1.3 *Grade No. 1-D*—A special-purpose, light distillate fuel for automotive diesel engines in applications requiring higher volatility than that provided by Grade No. 2-D fuels.

1.1.4 *Grade No. 2-D*—A general-purpose, middle distillate fuel for automotive diesel engines, which is also suitable for use in non-automotive applications, especially in conditions of frequently varying speed and load.

1.1.5 *Grade No. 4-D*—A heavy distillate fuel, or a blend of distillate and residual oil, for low- and medium-speed diesel engines in non-automotive applications involving predominantly constant speed and load.

NOTE 1—A more detailed description of the grades of diesel fuel oils is given in Appendix X1.2.

1.2 This specification, unless otherwise provided by agreement between the purchaser and the supplier, prescribes the required properties of diesel fuels at the time and place of delivery.

1.2.1 Nothing in this specification shall preclude observance of federal, state, or local regulations which may be more restrictive.

NOTE 2—The generation and dissipation of static electricity can create problems in the handling of distillate diesel fuel oils. For more information on the subject, see Guide D 4865.

1.3 Values are stated in SI units and are regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 56 Test Method for Flash Point by Tag Closed Tester[3]
D 86 Test Method for Distillation of Petroleum Products[3]
D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester[3]
D 97 Test Method for Pour Point of Petroleum Products[3]
D 129 Test Method for Sulfur in Petroleum Products (General Bomb Method)[3]
D 130 Test Method for Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test[3]
D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[3]
D 482 Test Method for Ash from Petroleum Products[3]
D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products[3]
D 613 Test Method for Cetane Number of Diesel Fuel Oil[4]
D 976 Test Methods for Calculated Cetane Index of Distillate Fuels[3]
D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)[3]
D 1319 Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption[3]
D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)[3]
D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)[3]
D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)[3]
D 2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)[3]
D 2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling[3]
D 2500 Test Method for Cloud Point of Petroleum Oils[3]
D 2622 Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry[5]

---

[1] This specification is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E on Burner, Diesel, Non–aviation Gas Turbine, and Marine Fuels.

Current edition approved Dec.10, 1998. Published February 1999. Originally published as D975 – 48 T. Last previous edition D975 – 98a.

[2] This fuel complies with 40 CFR Part 80—Regulation of Fuels and Fuel Additives: Fuel Quality Regulations for Highway Diesel Fuel Sold in 1993 and Later Calendar Years.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 05.04.

[5] *Annual Book of ASTM Standards*, Vol 05.02.

D 2709 Test Method for Water and Sediment in Distillate Fuels by Centrifuge[5]
D 2880 Specification for Gas Turbine Fuel Oils[5]
D 3117 Test Method for Wax Appearance Point of Distillate Fuels[5]
D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry[5]
D 3828 Test Methods for Flash Point by Small Scale Closed Tester[5]
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[5]
D 4294 Test Method for Sulfur in Petroleum Products by Energy–Dispersive X-Ray Fluorescence Spectrometry[5]
D 4539 Test Method for Filterability of Diesel Fuels by Low Temperature Flow Test (LTFT)[2]
D 4737 Test Method for Calculated Cetane Index by Four Variable Equation[6]
D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems[6]
D 5001 Test Method for Measurement of Lubricity of Aviation Turbine Fuels by the Ball-on-Cylinder Lubricity Evaluator (BOCLE)[6]
D 5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)[6]
D 5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)[6]
D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)[6]
D 6078 Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball–on–Cylinder Lubricity Evaluation (SLBOCLE)
D 6079 Test Method for Evaluating Lubricity of Diesel Fuels by the High–Fequency Reciprocating Rig (HFFR)

2.2 *Other Documents:*

26 CFR Part 48 Manufacturers and Realtors Excise Taxes
26 CFR Part 48 Diesel Fuel Excise Tax; Dye Color and Concentration
40 CFR Part 80 Regulation of Fuels and Fuel Additives
IP 309 Diesel and domestic heating fuels–Determination of cold filter plugging point

3. Test Methods

3.1 The requirements enumerated in this specification shall be determined in accordance with the following methods:

3.1.1 *Flash Point*—Test Method D 93, except where other methods are prescribed by law. For all grades, Test Method D 3828 can be used as an alternate with the same limits. For Grades Low Sulfur No. 1-D, Low Sulfur No. 2-D, No. 1-D, and No. 2-D, Test Method D 56 can be used as an alternate with the same limits, provided the flash point is below 93°C and the viscosity is below 5.5 mm^2/s at 40°C. This test method will give slightly lower values. In cases of dispute, Test Method D 93 shall be used as the referee method.

3.1.2 *Cloud Point*—Test Method D 2500. For all grades, the automatic Test Method D 5771, D 5772, or D 5773 can be used as alternates with the same limits.Test Method D 3117 can also be used since it is closely related to Test Method D 2500. In case of dispute, Test Method D 2500 shall be the referee method.

3.1.3 *Water and Sediment*—Test Method D 2709 is used for Grades Low Sulfur No. 1-D, Low Sulfur No. 2-D, No. 1-D, and No. 2-D. Test Method D 1796 is used for Grade No. 4-D.

3.1.4 *Carbon Residue*—Test Method D 524.

3.1.5 *Ash*—Test Method D 482.

3.1.6 *Distillation of Low Sulfur No. 1-D, Low Sulfur No. 2-D, No. 1-D, and No. 2-D Fuel Oils*—Test Method D 86.

3.1.7 *Viscosity*—Test Method D 445.

3.1.8 *Sulfur*—Test Method D 2622 is used for Grades Low Sulfur No. 1-D and Low Sulfur No. 2-D. Test Methods D 1266, D3120 and D4294 are also suitable for determining up to 0.05 % sulfur in diesel fuels. Test Method D 129 is used for Grades No. 1-D, No. 2-D and No. 4-D. Test Methods D 1552, D 2622, and D4294 can also be used for these grades. In addition, Test Method D 1266 can be used for Grade No. 1, but only with samples having sulfur contents of 0.4 mass % and less (down to 0.01 %). In case of dispute, Test Method D 2622 is the referee sulfur test method for Grades Low Sulfur No. 1-D and Low Sulfur No. 2-D. Test Method D 129 is the referee sulfur test method for Grades No. 1-D, No. 2-D, and No. 4-D.

3.1.9 *Corrosion*—Test Method D 130, 3 h test at 50°C.

3.1.10 *Cetane Number*—Test Method D 613.

3.1.11 *Cetane Index*—Method D 976 – 80.

3.1.12 *Aromaticity*—Test Method D 1319. This test method provides an indication of the aromatics content of fuels. For fuels with a maximum final boiling point of 315°C, this method is a measurement of the aromatic content of the fuel.

4. Workmanship

4.1 The diesel fuel shall be visually free of undissolved water, sediment, and suspended matter.

5. Requirements

5.1 The grades of diesel fuel oils herein specified shall be hydrocarbon oils conforming to the detailed requirements shown in Table 1.

6. Keywords

6.1 diesel; fuel oil; petroleum and petroleum products; specification

---

[6] *Annual Book of ASTM Standards*, Vol 05.03.

TABLE 1 Detailed Requirements for Diesel Fuel Oils[A]

Property	ASTM Test Method[B]	Grade Low Sulfur No. 1-D[C]	Grade Low Sulfur No. 2-D[C]	Grade No. 1-D[D]	Grade No. 2-D[D]	Grade No. 4-D[D]
Flash Point, °C, min.	D 93	38	52	38	52	55
Water and Sediment, % vol, max	D 2709	0.05	0.05	0.05	0.05	...
	D 1796	...	...	...	...	0.50
Distillation Temperature, °C 90 % % vol Recovered	D 86					
min		...	282[E]	...	282[E]	...
max		288	338	288	338	...
Kinematic Viscosity, mm^2/S at 40°C	D 445					
min.		1.3	1.9	1.3	1.9	5.5
max	...	2.4	4.1	2.4	4.1	24.0
Ash % mass, max	D 482	0.01	0.01	0.01	0.01	0.10
Sulfur, % mass, max[F]	D 2622[G]	0.05	0.05	...	...	...
	D 129	...	...	0.50	0.50	2.00
Copper strip corrosion rating max 3 h at 50°C	D 130	No. 3	No. 3	No. 3	No. 3	...
Cetane number, min[H]	D 613	40[I]	40[I]	40[I]	40[I]	30[I]
One of the following properties must be met:						
(1) Cetane index, min.	D 976[F]	40	40	...	...	...
(2) Aromaticity, % vol, max	D 1319[F]	35	35	...	...	...
Cloud point, °C, max	D 2500	J	J	J	J	J
Ramsbottom carbon residue on 10 % distillation residue, % mass, max	D 524	0.15	0.35	0.15	0.35	...

[A] To meet special operating conditions, modifications of individual limiting requirements may be agreed upon between purchaser, seller, and manufacturer.

[B] The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 3.1.

[C] Under United States regulations, if Grades Low Sulfur No. 1-D or Low Sulfur No. 2-D are sold for tax exempt purposes then, at or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lbs per thousand barrels of the solid dye standard Solvent Red 26, or the tax must be collected.

[D] Under United States regulations, Grades No.1–D, No. 2–D, and No. 4–D are required by 40 CFR Part 80 to contain a sufficient amount of the dye Solvent Red 164 so its presence is visually apparent. At or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lbs per thousand barrels of the solid dye standard Solvent Red 26.

[E] When a cloud point less than – 12°C is specified, the minimum flash point shall be 38°C, the minimum viscosity at 40°C shall be 1.7 mm^2/s, and the minimum 90 % recovered temperature shall be waived.

[F] Other sulfur limits can apply in selected areas in the United States and in other countries.

[G] These test methods are specified in 40 CFR Part 80.

[H] Where cetane number by Test Method D 613 is not available, Test Method D 4737 can be used as an approximation.

[I] Low ambient temperatures as well as engine operation at high altitudes may require the use of fuels with higher cetane ratings.

[J] It is unrealistic to specify low temperature properties that will ensure satisfactory operation at all ambient conditions. In general, cloud point (or wax appearance point) may be used as an estimate of operating temperature limits for Grades Low Sulfur No. 1; Low Sulfur No. 2; and No. 1 and No. 2 diesel fuel oils. However, satisfactory operation below the cloud point (or wax appearance point) may be achieved depending on equipment design, operating conditions, and the use of flow-improver additives as described in X4.1.2. Tenth percentile minimum air temperatures for U.S. locations are provided in Appendix X4 as a means of estimating expected regional temperatures. This guidance is general. Some equipment designs or operation may allow higher or require lower cloud point fuels. Appropriate low temperature operability properties should be agreed upon between the fuel supplier and purchaser for the intended use and expected ambient temperatures.

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF ASTM SPECIFICATION FOR DIESEL FUEL OILS

X1.1 Introduction

X1.1.1 The properties of commercial fuel oils depend on the refining practices employed and the nature of the crude oils from which they are produced. Distillate fuel oils, for example, may be produced within the boiling range of 150 and 400°C having many possible combinations of various properties, such as volatility, ignition quality, viscosity, and other characteristics.

X1.2 Grades

X1.2.1 This specification is intended as a statement of permissible limits of significant fuel properties used for specifying the wide variety of commercially available diesel fuel oils. Limiting values of significant properties are prescribed for five grades of diesel fuel oils. These grades and their general applicability for use in diesel engines are broadly indicated as follows:

X1.2.2 *Grade Low Sulfur No. 1-D*—Grade Low Sulfur No. 1-D comprises the class of low-sulfur, volatile fuel oils from kerosine to the intermediate distillates. Fuels within this grade are applicable for use in high-speed engines that require low sulfur fuel and in services involving frequent and relatively wide variations in loads and speeds, and also for use in cases where abnormally low fuel temperatures are encountered.

X1.2.3 *Grade Low Sulfur No. 2-D*—Grade Low Sulfur No. 2-D includes the class of low-sulfur, distillate gas oils of lower volatility than Grade Low Sulfur No. 1-D. These fuels are applicable for use in high-speed engines that require low sulfur fuels and in services involving relatively high loads and uniform speeds, or in engines not requiring fuels having the higher volatility or other properties specified for Grade Low Sulfur No. 1-D.

X1.2.4 *Grade No. 1-D*— Grade No. 1-D comprises the class of volatile fuel oils from kerosine to the intermediate distillates. Fuels within this grade are applicable for use in high-speed engines in services involving frequent and relatively wide variations in loads and speeds, and also for use in cases where abnormally low fuel temperatures are encountered.

X1.2.5 *Grade No. 2-D*— Grade No. 2-D includes the class of distillate gas oils of lower volatility. These fuels are applicable for use in high-speed engines in services involving relatively high loads and uniform speeds, or in engines not requiring fuels having the higher volatility or other properties specified for Grade No. 1-D.

X1.2.6 *Grade No. 4-D*— Grade No. 4-D covers the class of more viscous distillates and blends of these distillates with residual fuel oils. These fuels are applicable for use in low- and medium-speed engines employed in services involving sustained loads at substantially constant speed.

X1.3 Selection of Particular Grade

X1.3.1 The selection of a particular diesel fuel oil from one of these three ASTM grades for use in a given engine requires consideration of the following factors:

X1.3.1.1 Fuel price and availability,

X1.3.1.2 Maintenance considerations,

X1.3.1.3 Engine size and design,

X1.3.1.4 Emission control systems,

X1.3.1.5 Speed and load ranges,

X1.3.1.6 Frequency of speed and load changes, and

X1.3.1.7 Atmospheric conditions. Some of these factors can influence the required fuel properties outlined as follows:

X1.4 Cetane Number

X1.4.1 Cetane number is a measure of the ignition quality of the fuel and influences combustion roughness. The cetane number requirements depend on engine design, size, nature of speed and load variations, and on starting and atmospheric conditions. Increase in cetane number over values actually required does not materially improve engine performance. Accordingly, the cetane number specified should be as low as possible to assure maximum fuel availability.

X1.5 Distillation

X1.5.1 The fuel volatility requirements depend on engine design, size, nature of speed and load variations, and starting and atmospheric conditions. For engines in services involving rapidly fluctuating loads and speeds as in bus and truck operation, the more volatile fuels may provide best performance, particularly with respect to smoke and odor. However, best fuel economy is generally obtained from the heavier types of fuels because of their higher heat content.

X1.6 Viscosity

X1.6.1 For some engines it is advantageous to specify a minimum viscosity because of power loss due to injection pump and injector leakage. Maximum viscosity, on the other hand, is limited by considerations involved in engine design and size, and the characteristics of the injection system.

X1.7 Carbon Residue

X1.7.1 Carbon residue gives a measure of the carbon depositing tendencies of a fuel oil when heated in a bulb under prescribed conditions. While not directly correlating with engine deposits, this property is considered an approximation.

X1.8 Sulfur

X1.8.1 The effect of sulfur content on engine wear and deposits appears to vary considerably in importance and depends largely on operating conditions. Fuel sulfur can affect emission control systems performance. To assure maximum availability of fuels, the permissible sulfur content should be specified as high as is practicable, consistent with maintenance considerations.

X1.9 Flash Point

X1.9.1 The flash point as specified is not directly related to engine performance. It is, however, of importance in connection with legal requirements and safety precautions involved in fuel handling and storage, and is normally specified to meet insurance and fire regulations.

X1.10 Cloud Point

X1.10.1 Cloud point is of importance in that it defines the temperature at which a cloud or haze of wax crystals appears in the oil under prescribed test conditions which generally relates to the temperature at which wax crystals begin to precipitate from the oil in use.

X1.11 Ash

X1.11.1 Ash-forming materials may be present in fuel oil in two forms: *(1)* abrasive solids, and *(2)* soluble metallic soaps. Abrasive solids contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear but may contribute to engine deposits.

X1.12 Copper Strip Corrosion

X1.12.1 This test serves as a measure of possible difficulties with copper and brass or bronze parts of the fuel system.

X1.13 Aromaticity

X1.13.1 This test is used as an indication of the aromatic contents of diesel fuel. Aromatic content is specified to prevent an increase in the average aromatics in Grades Low Sulfur No. 1-D and Low Sulfur No. 2-D fuels. Increases in aromatic content of fuels over current levels may have a negative impact on emissions.

X1.14 Cetane Index

X1.14.1 Cetane Index is specified as a limitation on the amount of high aromatic components in Grades Low Sulfur No. 1-D and Low Sulfur No. 2-D.

X2. LONG-TERM STORAGE OF DISTILLATE FUELS

X2.1 Scope

X2.1.1 This appendix provides guidance for consumers of distillate fuels who may wish to store quantities of fuels for extended periods. Fuels containing residual components are excluded. Consistently successful long-term fuel storage requires attention to fuel selection, storage conditions, and monitoring of properties prior to and during storage.

X2.1.2 Normally produced fuels have adequate stability properties to withstand normal storage without the formation of troublesome amounts of insoluble degradation products. Fuels that are to be stored for prolonged periods should be selected to avoid formation of sediments, which can overload filters or plug combustor nozzles or injectors. Selection of these fuels should result from supplier-user discussions.

X2.1.3 These suggested practices are general in nature and should not be considered substitutes for any requirements imposed by the warranty of the distillate fuel equipment manufacturer or by federal, state, or local government regulations. Although they cannot replace a knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the distillate fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when, and how fuel quality should be monitored.

X2.2 Definitions

X2.2.1 *long-term storage*—storage of fuel for longer than 12 months after it is received by the user.

X2.2.2 *bulk fuel*—fuel in the storage facility.

X2.2.3 *combustor fuel*— fuel entering the combustion zone of the burner or engine after filtration or other treatment of bulk fuel.

X2.2.4 *fuel contaminants*—foreign materials that make fuel less suitable or unsuitable for the intended use. Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

X2.2.5 *fuel-degradation products*—those materials that are formed in fuel during extended storage. Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (soluble gums) are less volatile than fuel and may carbonize to form in fuels due to complex interactions and oxidation of small amounts of olefinic, sulfurous, oxygenated, and nitrogenous compounds present in fuels. The formation of degradation products may be catalyzed by dissolved metals, especially copper salts.

X2.3 Fuel Selection

X2.3.1 Certain distilled refinery products are generally more suitable for long-term storage than others. The stability properties of distillates are highly dependent on the crude oil sources, severity of processing, and whether additional refinery treatment has been carried out.

X2.3.2 The composition and stability properties of distillate fuels produced at specific refineries may be different. Any special requirements of the user, such as long-term storage, should be discussed with the supplier.

X2.3.3 Blends of fuels from various sources may interact to give stability properties worse than expected based on the characteristics of the individual fuels.

X2.4 Fuel Additives

X2.4.1 Available fuel additives can improve the suitability of marginal fuels for long-term storage but may be unsuccessful for fuels with markedly poor stability properties. Most additives should be added at the refinery or during the early weeks of storage to obtain maximum benefits.

X2.4.2 Biocides or biostats destroy or inhibit the growth of fungi and bacteria, which can grow at fuel-water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in both the fuel and water or in the water phase only.

X2.5 Tests for Fuel Quality

X2.5.1 At the time of manufacture, the storage stability of fuel may be assessed using Test Method D 2274. However, this accelerated stability test may not correlate well with field storage stability due to varying field conditions and to fuel composition.

X2.5.2 Performance criteria for accelerated stability tests that assure satisfactory long-term storage of fuels have not been established.

X2.6 Fuel Monitoring

X2.6.1 A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

X2.6.2 Stored fuel should be periodically sampled and its quality assessed. Practice D 4057 provides guidance for sampling. Fuel contaminants and degradation products will usually settle to the bottom of a quiescent tank. A "Bottom" or "Clearance" sample, as defined in Practice D 4057, should be included in the evaluation along with an "All Level" sample.

X2.6.3 The quantity of insoluble fuel contaminants present in fuel can be determined using Test Method D 2276, Procedure A.

X2.6.4 Other quality tests like fuel color (see Test Method D 1500) and stability tests (see Test Method D 2274) after storage may have value. Correlations of these tests with fuel suitability are tenuous.

X2.7 Fuel Storage Conditions

X2.7.1 Contamination levels in fuel can be reduced by storage in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface. Underground storage is preferred to avoid temperature extremes; above-ground storage tanks should be sheltered or painted with reflective paint. High storage temperatures accelerate fuel degradation. Fixed roof tanks should be kept full to limit oxygen supply and tank breathing.

X2.7.2 Copper and copper-containing alloys should be avoided. Copper can promote fuel degradation and may produce mercaptide gels. Zinc coatings can react with water or organic acids in the fuel to form gels which rapidly plug filters.

X2.7.3 Appendix X3 of Specification D 2880 discusses fuel contaminants as a general topic.

X2.8 Use of Degraded Fuels

X2.8.1 Fuels that have undergone mild-to-moderate degradation can often be consumed in a normal way, depending on the fuel system requirements. Filters and other cleanup equipment can require special attention and increased maintenance. Burner nozzle or injector fouling can occur more rapidly.

X2.8.2 Fuels containing very large quantities of fuel degradation products and other contaminants or with runaway microbiological growth require special attention. Consultation with experts in this area is desirable. It can be possible to drain the sediment or draw off most of the fuel above the sediment layer and use it with the precautions described in X2.8.1. However, very high soluble gum levels or corrosion products from microbiological contamination can cause severe operational problems.

X3. DIESEL FUEL LUBRICITY

X3.1 Introduction

X3.1.1 Diesel fuel functions as a lubricant in certain items of fuel injection equipment such as rotary/distributor fuel pumps and injectors. In limited cases, fuel with very specific properties can have insufficient lubricating properties which can lead to a reduction in the normal service life of fuel pumps or injectors.

X3.2 Fuel Characteristics Affecting Equipment Wear

X3.2.1 Two fuel characteristics that affect equipment wear are low viscosity and lack of sufficient quantities of trace components, which have an affinity for metal surfaces. If fuel viscosity meets the requirements of a particular engine, a fuel film is maintained between the moving surfaces of the fuel system components. This prevents excessive metal-to-metal contact and avoids premature failure due to wear. Similarly, certain surface active molecules in the fuel adhere to, or combine with, metallic surfaces to produce a protective film which also can protect surfaces against excessive wear.

X3.3 Fuel Lubricity

X3.3.1 The concern about fuel lubricity is limited to situations in which fuels with lower viscosities than those specified for a particular engine are used or with fuels which have been processed in a manner that results in the elimination of the trace levels of the surface active species that act as lubricating agents. Presently the only fuels of the latter type shown to have lubricity problems resulted from sufficiently severe processing to reduce aromatics substantially below current levels. Research is in progress to identify the characteristics of such fuels and where the use of a lubricity improver additive is required, to ensure satisfactory operation in the sensitive areas of the vehicle fuel system.

X3.3.2 Work in the area of diesel fuel lubricity is ongoing by several organizations, such as the International Standard Organization (ISO) and the ASTM Diesel Fuel Lubricity Task Force. These groups include representatives from the fuel injection equipment manufacturers, fuel producers, and additive suppliers. The charge of the ASTM task force has been the recommendation of test methods and a fuel specification for Specification D 975. Two test methods were proposed and approved. These are Test Method D 6078, a scuffing load ball-on-cylinder lubricity evaluator method, SLBOCLE, and Test Method D 6079, a high frequency reciprocating rig method, HFRR. The inclusion of a single fuel specification in the main table for Grade No. 2 requires further research because 1) the correlation of the data among the two test methods and the fuel injection equipment needs further clarification, 2) both methods in their current from do not apply to all fuel-additive combinations, and 3) the reproducibility values for both test methods are large. In the meantime, the following information may be of use and serve as a general guideline to fuel suppliers and users.

X3.3.3 SAE Technical Paper 952369[7] indicates that users should monitor their fuel injection pumps for possible trends of abnormal wear rates if the fuel has a scuffing load value between 2000 and 2800 g in Test Method D 6078. According to this paper, fuels with values below 2000 g will in all probability cause accelerated wear in fuel lubricated rotary-type fuel injection pumps. It should be noted that fuels with a sufficient level of an effective lubricity additive may protect the equipment, but may not be recognized by the test method.

X3.3.4 Work at ISO, documented in SAE Technical Paper 952372,[8] indicates that a fuel with a 450-micron wear scar diameter or lower value at 60 °C in Test Method D 6079 (380 micron at 25 °C) should protect all fuel injection equipment. Other SAE publications present data to show that some fuels and fuel/additive combinations can have values above this level and still provide sufficient lubricity protection to the equipment. The current ISO test program is addressing this issue.

X3.3.5 Pump stand testing of fuels, although more expensive and time consuming, is a more accurate means of evaluating the lubricity of diesel fuel. Although several fuel injection manufacturers have pump-stand tests, no single test method has become standardized and no standard industry-approved type procedure is available at this time.

---

[7] Westbrook, S.R., "Survey of Low Sulfur Diesel Fuels and Aviation Kerosenes from U.S. Military Installations", SAE Technical Paper 952369, 1995.

[8] Nikanjam, M., "ISO Diesel Fuel Lubricity Round Robin Program", SAE Technical Paper 952372, 1995.

X4. TENTH PERCENTILE MINIMUM AMBIENT AIR TEMPERATURES FOR THE UNITED STATES (EXCEPT HAWAII)

X4.1 Introduction

X4.1.1 The tenth percentile minimum ambient air temperatures shown on the following maps (Figs. X4.1-X4.12) and in Table X4.1 were derived from an analysis of historical hourly temperature readings recorded over a period of 15 to 21 years from 345 weather stations in the United States. This study was conducted by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, Aberdeen Proving Ground, MD 21005. The tenth percentile minimum ambient air temperature is defined as the lowest ambient air temperature which will not go lower on average more than 10 % of the time. In other words, the daily minimum ambient air temperature would on average not be expected to go below the monthly tenth percentile minimum ambient air temperature more than 3 days for a 30 day month.

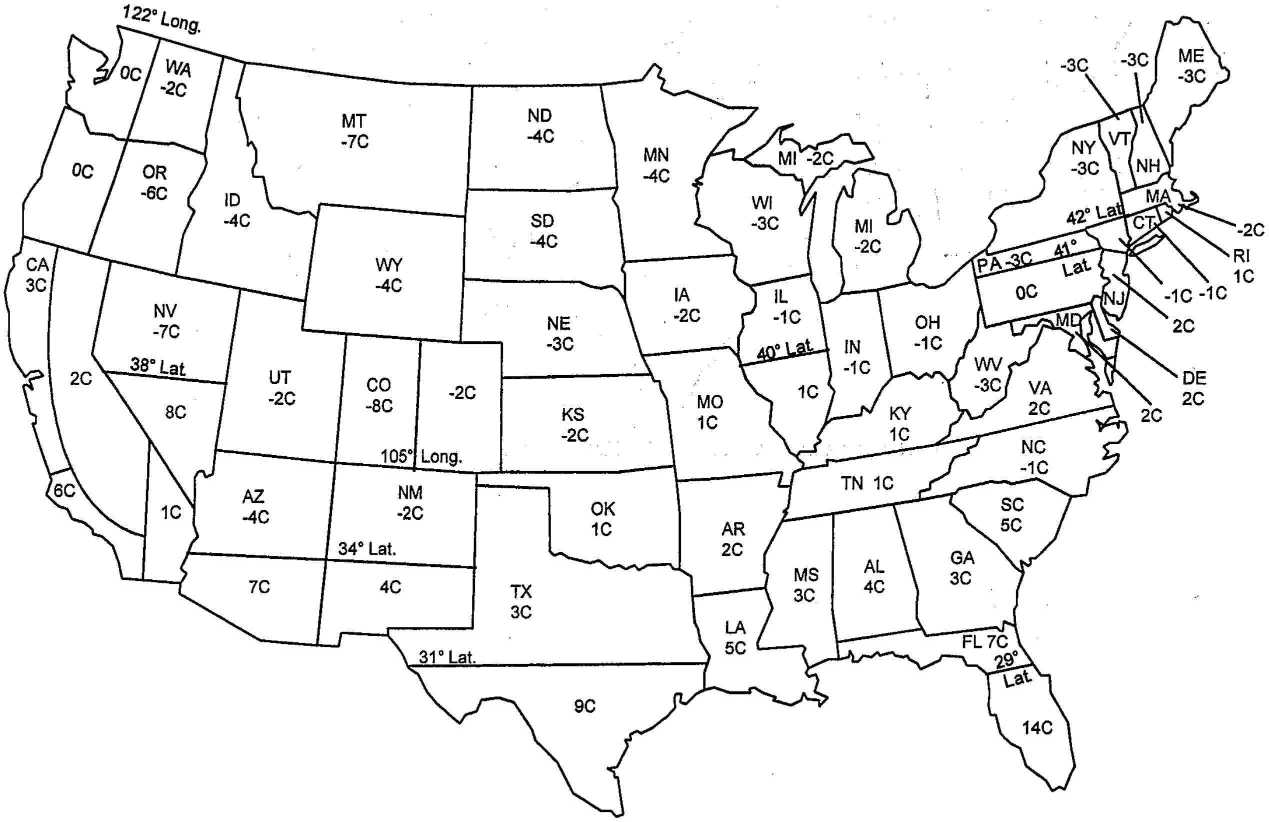


FIG. X4.1 October—10th Percentile Minimum Temperatures

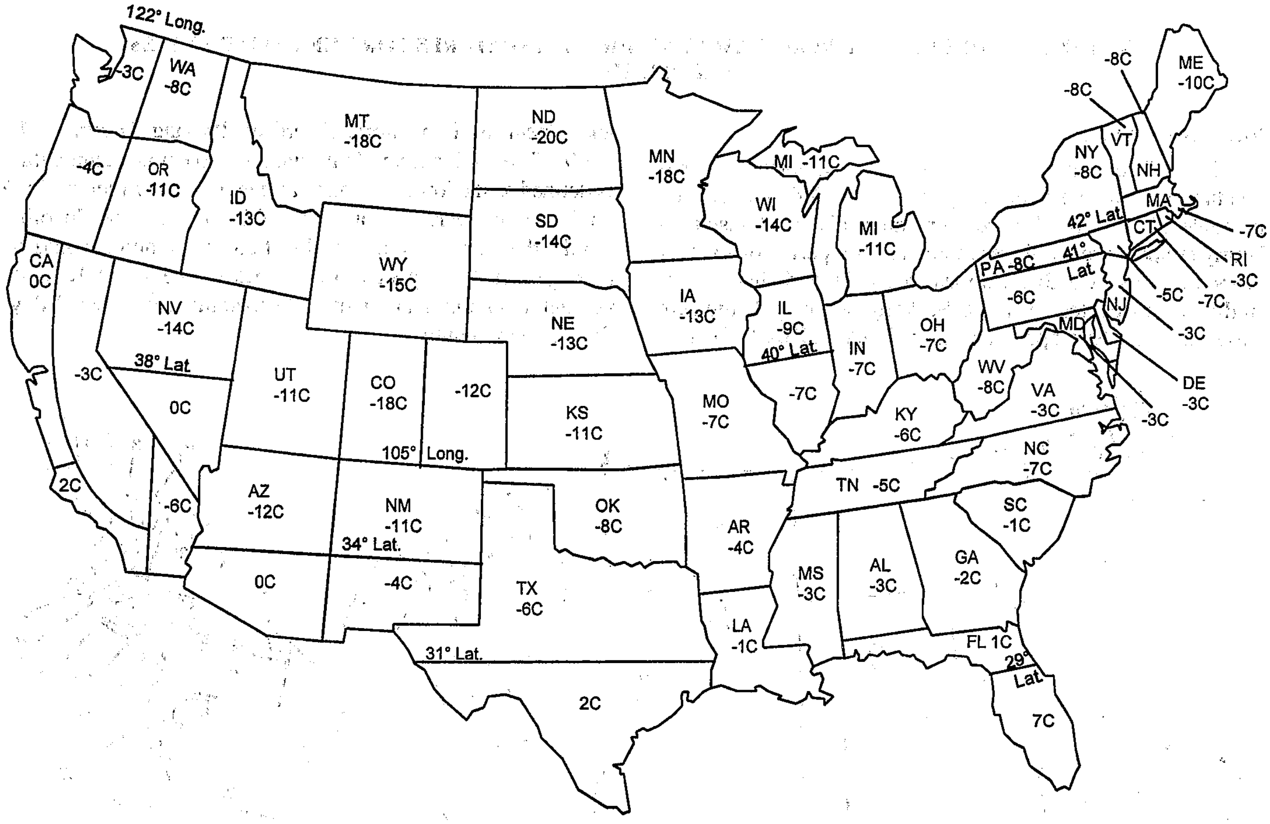


FIG. X4.2 November—10th Percentile Minimum Ambient Air Temperatures

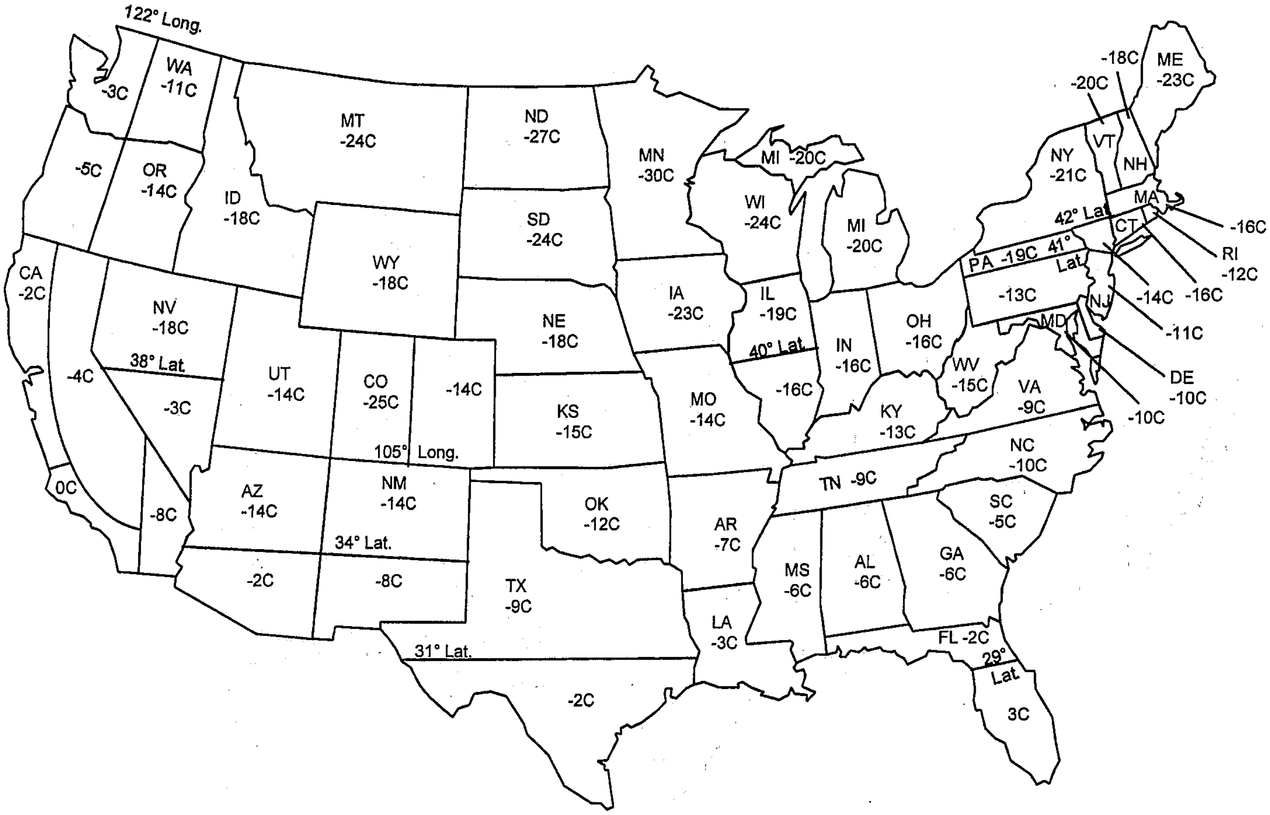


FIG. X4.3 December—10th Percentile Minimum Ambient Air Temperatures

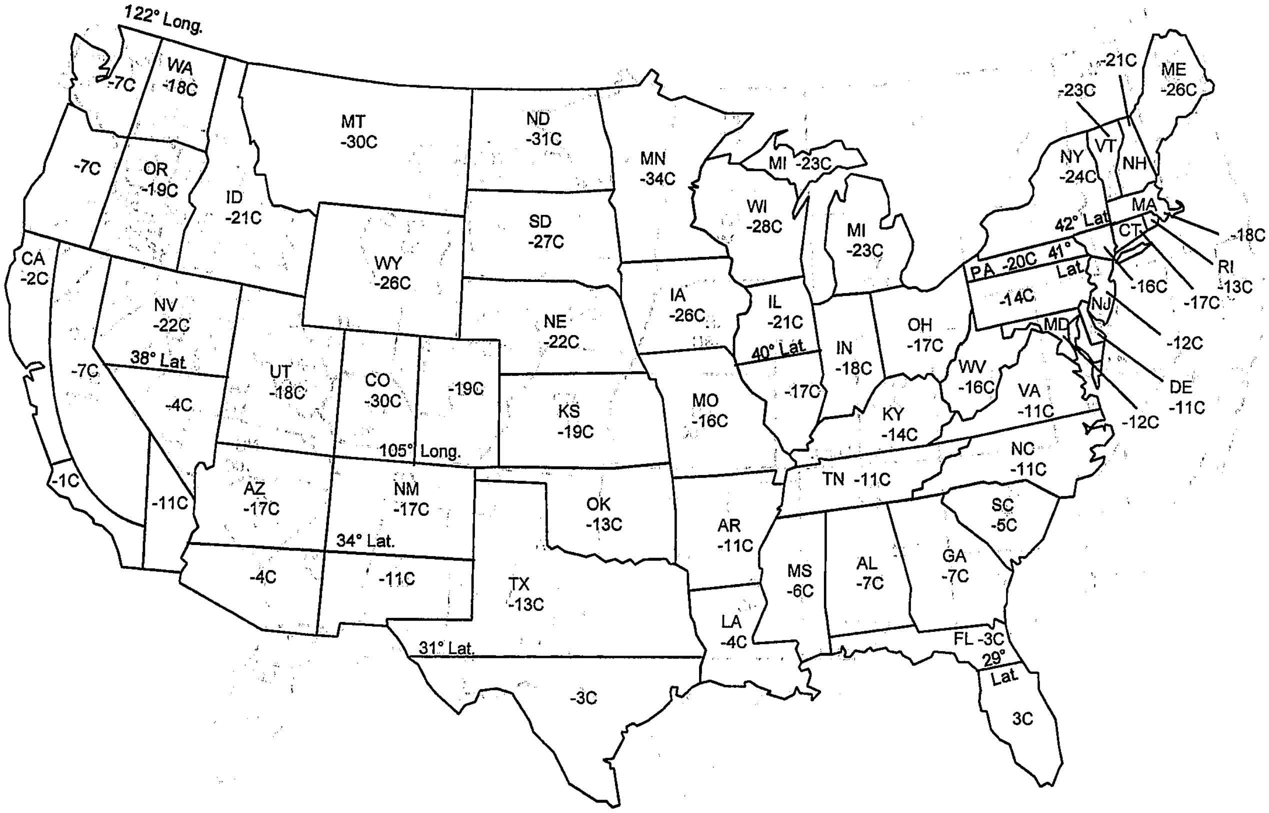


FIG. X4.4 January—10th Percentile Minimum Ambient Air Temperatures

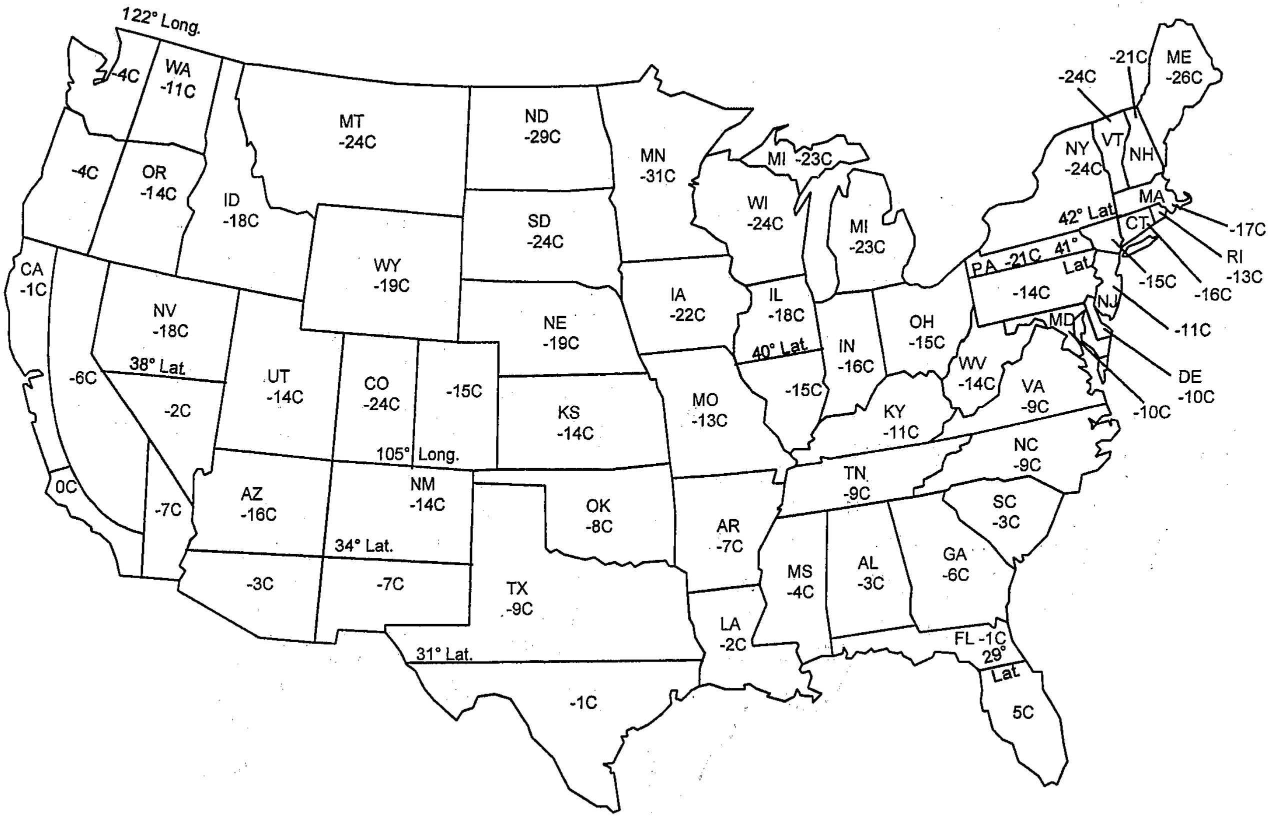


FIG. X4.5 February—10th Percentile Minimum Ambient Air Temperatures

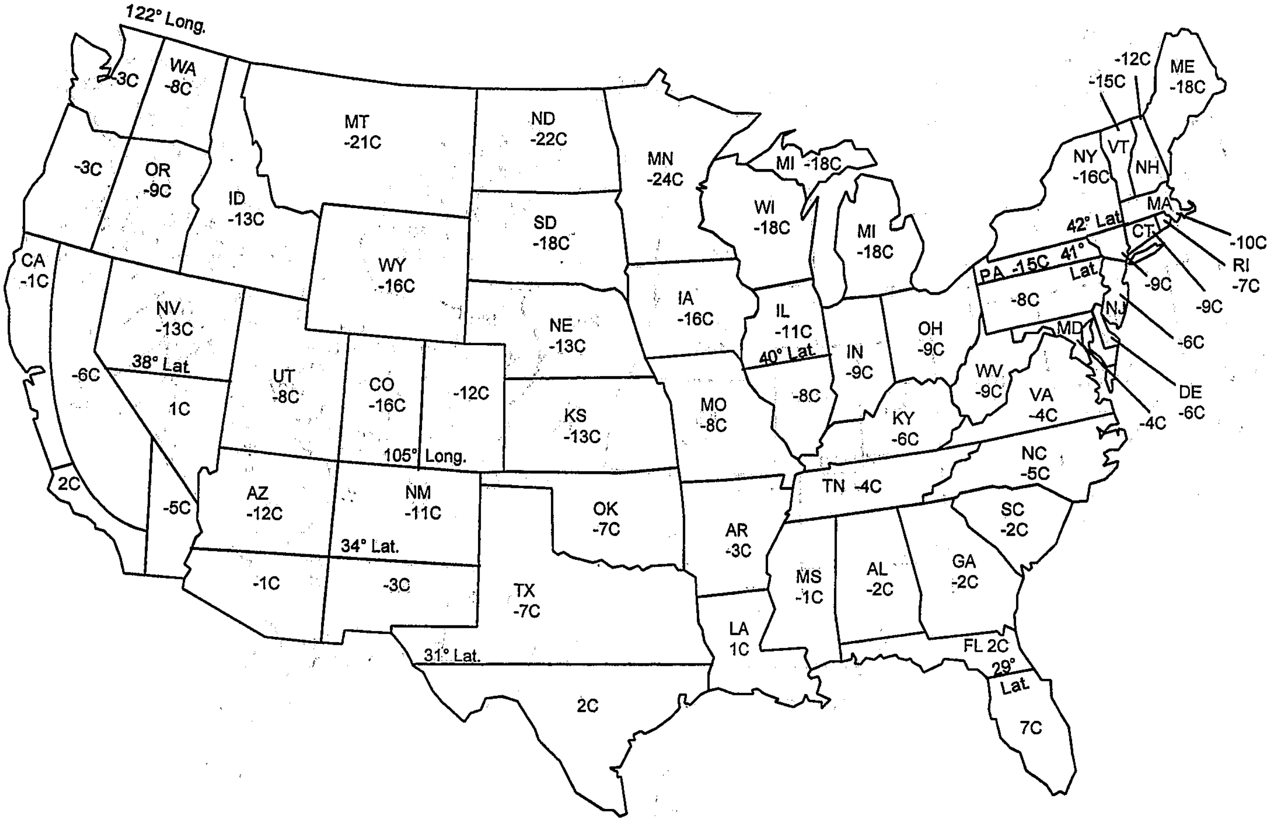


FIG. X4.6 March—10th Percentile Minimum Ambient Air Temperatures

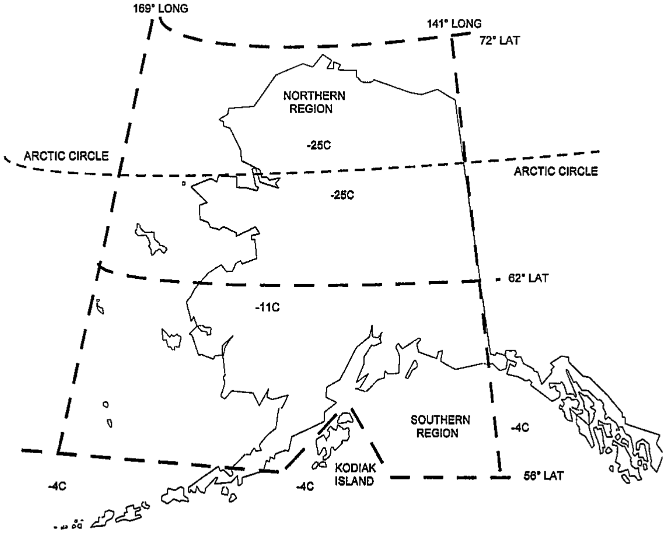


FIG. X4.7 October—10th Percentile Minimum Ambient Air Temperatures

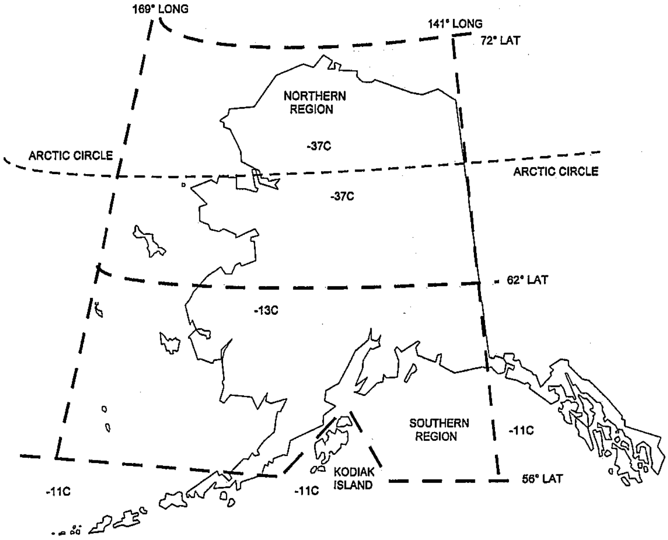


FIG. X4.8 November—10th Percentile Minimum Ambient Air Temperatures

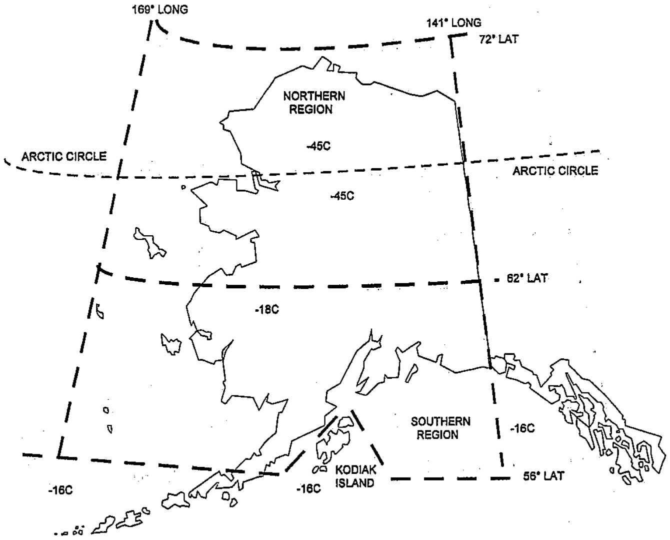


FIG. X4.9 December—10th Percentile Minimum Ambient Air Temperatures

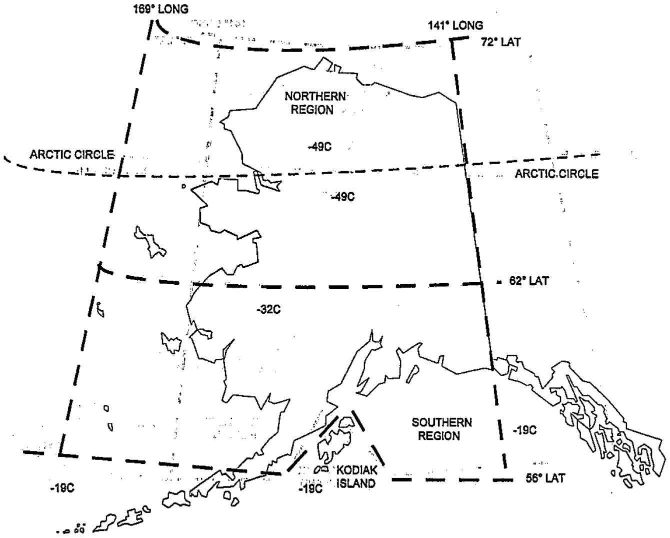


FIG. X4.10 January—10th Percentile Minimum Ambient Air Temperatures

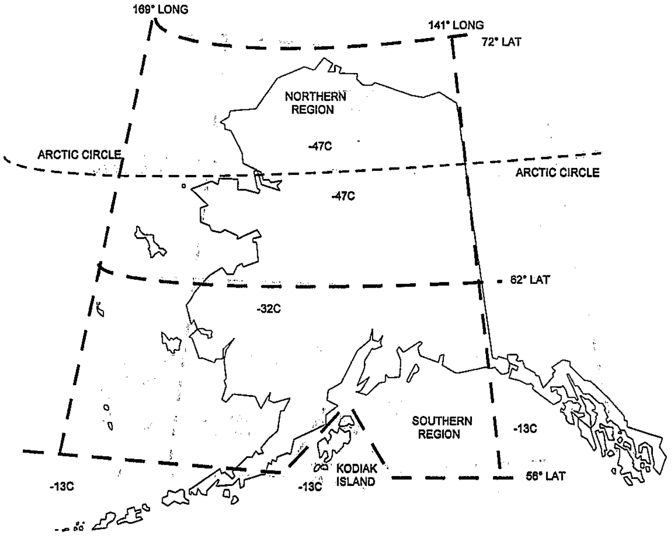


FIG. X4.11 February—10th Percentile Minimum Ambient Air Temperatures

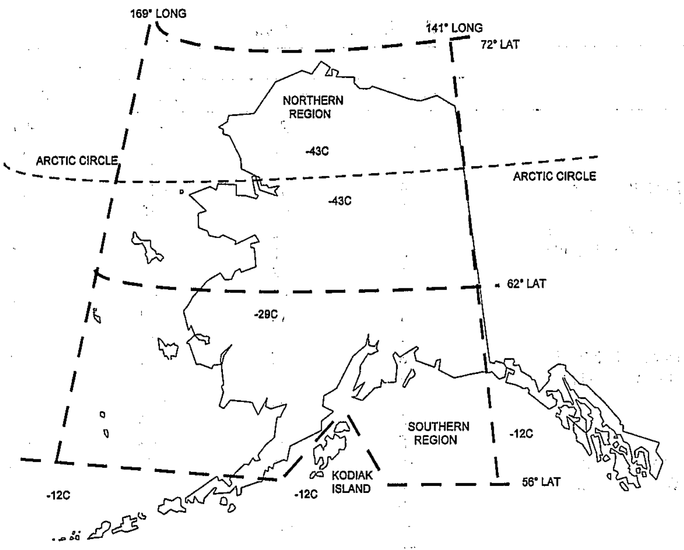


FIG. X4.12 March—10th Percentile Minimum Ambient Air Temperatures

TABLE X4.1 Tenth Percentile Minimum Ambient Air Temperatures for the United States (except Hawaii)

State		10th Percentile Temperature°C, min					
		Oct.	Nov.	Dec.	Jan.	Feb.	March
Alabama		4	−3	−6	−7	−3	−2
Alaska	Northern	−25	−37	−45	−49	−47	−43
	Southern	−11	−13	−18	−32	−32	−29
	South East	−4	−11	−16	−19	−13	−12
Arizona	North 34° latitude	−4	−12	−14	−17	−16	−12
	South 34° latitude	7	0	−2	−4	−3	−1
Arkansas		2	−4	−7	−11	−7	−3
California	North Coast	3	0	−2	−2	−1	−1
	Interior	2	−3	−4	−7	−6	−6
	South Coast	6	2	0	−1	0	2
	Southeast	1	−6	−8	−11	−7	−5
Colorado	East 105° long	−2	−12	−14	−19	−15	−12
	West 105° long	−8	−18	−25	−30	−24	−16
Connecticut		−1	−7	−16	−17	−16	−9
Delaware		2	−3	−10	−11	−10	−6
Florida	North 29° latitude	7	1	−2	−3	−1	2
	South 29° latitude	14	7	3	3	5	7
Georgia		3	−2	−6	−7	−6	−2
Idaho		−4	−13	−18	−21	−18	−13
Illinois	North 40° latitude	−1	−9	−19	−21	−18	−11
	South 40° latitude	1	−7	−16	−17	−15	−8
Indiana		−1	−7	−16	−18	−16	−9
Iowa		−2	−13	−23	−26	−22	−16
Kansas		−2	−11	−15	−19	−14	−13
Kentucky		1	−6	−13	−14	−11	−6
Louisiana		5	−1	−3	−4	−2	1
Maine		−3	−10	−23	−26	−26	−18
Maryland		2	−3	−10	−12	−10	−4
Massachusetts		−2	−7	−16	−18	−17	−10
Michigan		−2	−11	−20	−23	−23	−18
Minnesota		−4	−18	−30	−34	−31	−24
Mississippi		3	−3	−6	−6	−4	−1
Missouri		1	−7	−14	−16	−13	−8
Montana		−7	−18	−24	−30	−24	−21
Nebraska		−3	−13	−18	−22	−19	−13
Nevada	North 38° latitude	−7	−14	−18	−22	−18	−13
	South 38° latitude	8	0	−3	−4	−2	1
New Hampshire		−3	−8	−18	−21	−21	−12
New Jersey		2	−3	−11	−12	−11	−6
New Mexico	North 34° latitude	−2	−11	−14	−17	−14	−11
	South 34° latitude	4	−4	−8	−11	−7	−3
New York	North 42° latitude	−3	−8	−21	−24	−24	−16
	South 42° latitude	−1	−5	−14	−16	−15	−9
North Carolina		−1	−7	−10	−11	−9	−5

TABLE X4.1 *Continued*

State		10th Percentile Temperature°C, min					
		Oct.	Nov.	Dec.	Jan.	Feb.	March
North Dakota		−4	−20	−27	−31	−29	−22
Ohio		−1	−7	−16	−17	−15	−9
Oklahoma		1	−8	−12	−13	−8	−7
Oregon	East 122° long	−6	−11	−14	−19	−14	−9
	West 122° long	0	−4	−5	−7	−4	−3
Pennsylvania	North 41° latitude	−3	−8	−19	−20	−21	−15
	South 41° latitude	0	−6	−13	−14	−14	−8
Rhode Island		1	−3	−12	−13	−13	−7
South Carolina		5	−1	−5	−5	−3	−2
South Dakota		−4	−14	−24	−27	−24	−18
Tennessee		1	−5	−9	−11	−9	−4
Texas	North 31° latitude	3	−6	−9	−13	−9	−7
	South 31° latitude	9	2	−2	−3	−1	2
Utah		−2	−11	−14	−18	−14	−8
Vermont		−3	−8	−20	−23	−24	−15
Virginia		2	−3	−9	−11	−9	−4
Washington	East 122° long	−2	−8	−11	−18	−11	−8
	West 122° long	0	−3	−3	−7	−4	−3
West Virginia		−3	−8	−15	−16	−14	−9
Wisconsin		−3	−14	−24	−28	−24	−18
Wyoming		−4	−15	−18	−26	−19	−16

X4.1.2 These data may be used to estimate low temperature operability requirements. In establishing low temperature operability requirements, consideration should be given to the following. These factors, or any combination, may make low temperature operability more or less severe than normal. As X4.1.2.1-X4.1.2.12 indicate, field work suggests that cloud point (or was appearance point) is a fair indication of the low temperature operability limit of fuels without cold flow additives in most vehicles.

X4.1.2.1 Long term weather patterns (Average winter low temperatures will be exceeded on occasion).

X4.1.2.2 Short term local weather conditions (Unusual cold periods do occur).

X4.1.2.3 Elevation (High locations are usually colder than surrounding lower areas).

X4.1.2.4 Specific engine design.

X4.1.2.5 Fuel system design (Recycle rate, filter location, filter capacity, filter porosity, and so forth.)

X4.1.2.6 Fuel viscosity at low temperatures

X4.1.2.7 Equipment add-ons (Engine heaters, radiator covers, fuel line and fuel filter heaters and so forth.)

X4.1.2.8 Types of operation (Extensive idling, engine shutdown, or unusual operation).

X4.1.2.9 Low temperature flow improver additives in fuel.

X4.1.2.10 Geographic area for fuel use and movement between geographical areas.

X4.1.2.11 General housekeeping (Dirt and/or water in fuel or fuel supply system).

X4.1.2.12 Impact failure for engine to start or run (Critical vs. non-critical application).

X4.1.3 *Historical Background*—A field test conducted by the CRC in 1981 resulted in two documents that provide insight into correlating laboratory tests to vehicle performance in the field. Prior to the 1981 field test, it was thought that in most cases vehicle would operate 6°C below the cloud point (or wax appearance point) due to the fuel temperature lagging behind the air temperature and the premise that more wax than would be produced at the cloud point would be necessary to block a filter or plug a fuel line. CRC Report No. 537[9] indicated that in overnight cool down, 30 % of the vehicles tested had final fuel tank temperatures within 2°C of the overnight minimum ambient temperature. CRC Report No. 528[10] concluded that of the several laboratory tests examined (cloud point, pour point, CEPP, I.TET), cloud point (see Test Method D 2500) predicted the bahavior of the untreated fuels. The combination of these reports would suggest the cloud point (or wax appearance point), in most cases, would be closer to the vehicle low temperature operability limit for untreated fuels. In addition, CRC Report No. 528 found that the Low Temperature Flow Test (LTFT), (see Test Method D 4539) provided the best correlation with vehicle performance for untreated fuel and flow improver treated fuel. Pour point, (see Test Method D 97) and Cold Filter Plugging Point (CFPP), (see Test Method IP 309) tests were not sufficiently severe enough to predict the performance of the test fuels in the diesel vehicles used in the study.

X4.1.3.1 *Current Practices*—It is recognized that fuel distributors, producers, and end users in the United States use cloud point, wax appearance point, CFPP, and LTFT to estimate vehicle low temperature operability limits for diesel fuel. No independent data has been published in recent years to determine test applicability for today's fuels and vehicles.

X4.2 Maps

X4.2.1 The maps in the following figures were derived from CCL Report No. 316, "A Predictive Study for Defining Limiting Temperatures and Their Application in Petroleum Product Specifications," by John P. Doner. This report was published by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, and it is available from the National Technical Information Service, Springfield, VA 22151, by requesting Publication No. AD756-420.

X4.2.2 Where states are divided the divisions are noted on the maps and table with the exception of California, which is divided by counties as follows:

California, North Coast—Alameda, Contra Costa, Del Norte, Humbolt, Lake, Marin, Mendocino, Monterey, Napa, San Benito, San Francisco, San Mateo, Santa Clara, Santa Cruz, Solano, Sonoma, Trinity.

California, Interior—Lassen, Modoc, Plumas, Sierra, Siskiyou, Alpine, Amador, Butte, Calaveras, Colusa, El Dorado, Fresno, Glenn, Kern (except that portion lying east of the Los Angeles County Aqueduct), Kings, Madera, Mariposa, Merced, Placer, Sacramento, San Joaquin, Shasta, Stanislaus, Sutter, Tehama, Tulare, Tuolumne, Yolo, Yuba, Nevada.

California, South Coast—Orange, San Diego, San Luis Obispo, Santa Barbara, Ventura, Los Angeles (except that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct).

California, Southeast—Imperial, Riverside, San Bernardino, Los Angeles (that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct), Mono, Inyo, Kern (that portion lying east of the Los Angeles County Aqueduct).

X4.2.3 The temperatures in CCL Report No. 316 were in degrees Fahrenheit. The degree Celsius temperatures in Appendix X4 were obtained by converting the original degree Fahrenheit temperatures.

---

[9] CRC Report No. 537, "The Relationship Between Vehicle Fuel Temperature and Ambient Temperature, 1981 CRC Kapuskasing Field Test", December 1983.

[10] CRC Report No. 528, "1981 CRC Diesel Fuel Low-Temperature Operability Field Test", September 1983.

ASTM Logo Removed

D 975

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D975: Standard Specification for Diesel Fuel Oils

CFR Section(s): 40 CFR 1065.701

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 975 – 07

An American National Standard

Standard Specification for Diesel Fuel Oils[1]

This standard is issued under the fixed designation D 975; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This specification covers seven grades of diesel fuel oils suitable for various types of diesel engines. These grades are described as follows:

1.1.1 *Grade No. 1-D S15*—A special-purpose, light middle distillate fuel for use in diesel engine applications requiring a fuel with 15 ppm sulfur (maximum) and higher volatility than that provided by Grade No. 2-D S15 fuel.[2]

1.1.2 *Grade No. 1-D S500*—A special-purpose, light middle distillate fuel for use in diesel engine applications requiring a fuel with 500 ppm sulfur (maximum) and higher volatility than that provided by Grade No. 2-D S500 fuel.[2]

1.1.3 *Grade No. 1-D S5000*—A special-purpose, light middle distillate fuel for use in diesel engine applications requiring a fuel with 5000 ppm sulfur (maximum) and higher volatility than that provided by Grade No. 2-D S5000 fuels.

1.1.4 *Grade No. 2-D S15*—A general purpose, middle distillate fuel for use in diesel engine applications requiring a fuel with 15 ppm sulfur (maximum). It is especially suitable for use in applications with conditions of varying speed and load.[2]

1.1.5 *Grade No. 2-D S500*—A general-purpose, middle distillate fuel for use in diesel engine applications requiring a fuel with 500 ppm sulfur (maximum). It is especially suitable for use in applications with conditions of varying speed and load.[2]

1.1.6 *Grade No. 2-D S5000*—A general-purpose, middle distillate fuel for use in diesel engine applications requiring a fuel with 5000 ppm sulfur (maximum), especially in conditions of varying speed and load.

1.1.7 *Grade No. 4-D*—A heavy distillate fuel, or a blend of distillate and residual oil, for use in low- and medium-speed diesel engines in applications involving predominantly constant speed and load.

NOTE 1—A more detailed description of the grades of diesel fuel oils is given in X1.2.

NOTE 2—The Sxxx designation has been adopted to distinguish grades by sulfur rather than using words such as "Low Sulfur" as previously because the number of sulfur grades is growing and the word descriptions were thought to be not precise. S5000 grades correspond to the so-called "regular" sulfur grades, the previous No. 1-D and No. 2-D. S500 grades correspond to the previous "Low Sulfur" grades. S15 grades were not in the previous grade system and are commonly referred to as "Ultra-Low Sulfur" grades or ULSD.

1.2 This specification, unless otherwise provided by agreement between the purchaser and the supplier, prescribes the required properties of diesel fuels at the time and place of delivery.

1.2.1 Nothing in this specification shall preclude observance of federal, state, or local regulations which may be more restrictive.

NOTE 3—The generation and dissipation of static electricity can create problems in the handling of distillate diesel fuel oils. For more information on the subject, see Guide D 4865.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

D 56 Test Method for Flash Point by Tag Closed Cup Tester

D 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D 129 Test Method for Sulfur in Petroleum Products (General Bomb Method)

D 130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D 482 Test Method for Ash from Petroleum Products

---

[1] This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.E0.02 on Diesel Fuel Oils.

Current edition approved Feb. 1, 2007. Published March 2007. Originally approved in 1948. Last previous edition approved in 2006 as D 975–06b.

[2] This fuel complies with 40 CFR Part 80—Control of Air Pollution from New Motor Vehicles: Heavy-Duty Engines and Vehicle Standards and Highway Diesel Fuel Sulfur Control Requirements: Final Rule. Regulation of Fuels and Fuel Additives: Fuel Quality Regulations for Highway Diesel Fuel Sold in 1993 and Later Calendar Years.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard.**

D 524 Test Method for Ramsbottom Carbon Residue of Petroleum Products
D 613 Test Method for Cetane Number of Diesel Fuel Oil
D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)
D 1319 Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption
D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)
D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)
D 2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
D 2500 Test Method for Cloud Point of Petroleum Products
D 2622 Test Method for Sulfur in Petroleum Products by Wavelength Dispersive X-ray Fluorescence Spectrometry
D 2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge
D 2880 Specification for Gas Turbine Fuel Oils
D 2887 Test Method for Boiling Range Distribution of Petroleum Fractions by Gas Chromatography
D 3117 Test Method for Wax Appearance Point of Distillate Fuels
D 3120 Test Method for Trace Quantities of Sulfur in Light Liquid Petroleum Hydrocarbons by Oxidative Microcoulometry
D 3828 Test Methods for Flash Point by Small Scale Closed Cup Tester
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D 4294 Test Method for Sulfur in Petroleum and Petroleum Products by Energy-Dispersive X-ray Fluorescence Spectrometry
D 4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination
D 4539 Test Method for Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT)
D 4737 Test Method for Calculated Cetane Index by Four Variable Equation
D 4865 Guide for Generation and Dissipation of Static Electricity in Petroleum Fuel Systems
D 5453 Test Method for Determination of Total Sulfur in Light Hydrocarbons, Spark Ignition Engine Fuel, Diesel Engine Fuel, and Engine Oil by Ultraviolet Fluorescence
D 5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)
D 5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)
D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)
D 5842 Practice for Sampling and Handling of Fuels for Volatility Measurement
D 5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
D 6078 Test Method for Evaluating Lubricity of Diesel Fuels by the Scuffing Load Ball-on-Cylinder Lubricity Evaluator (SLBOCLE)
D 6079 Test Method for Evaluating Lubricity of Diesel Fuels by the High-Frequency Reciprocating Rig (HFRR)
D 6217 Test Method for Particulate Contamination in Middle Distillate Fuels by Laboratory Filtration
D 6371 Test Method for Cold Filter Plugging Point of Diesel and Heating Fuels
D 6468 Test Method for High Temperature Stability of Distillate Fuels
D 6469 Guide for Microbial Contamination in Fuels and Fuel Systems
D 6890 Test Method for Determination of Ignition Delay and Derived Cetane Number (DCN) of Diesel Fuel Oils by Combustion in a Constant Volume Chamber
D 6898 Test Method for Evaluating Diesel Fuel Lubricity by an Injection Pump Rig

2.2 *Other Documents:*

26 CFR Part 48 Manufacturers and Realtors Excise Taxes[4]
40 CFR Part 80 Regulation of Fuels and Fuel Additives[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *S(numerical specification maximum)*—indicates the maximum sulfur content, in weight ppm (µg/g), allowed by this specification in a diesel fuel grade.

3.1.1.1 *Discussion*—Of the seven diesel fuel grades specified in this standard, six have important distinguishing maximum sulfur regulatory requirements. These are Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D S15, No. 2-D S500 and No. 2-D S5000. The seventh grade, No. 4-D, is distinguished from these other grades by many major properties in addition to sulfur (unregulated maximum), and therefore is not included in this designation system. Thus, Grade No. 4-D does not have the designation S20000 as part of its grade name.

4. Sampling, Containers, and Sample Handling

4.1 It is strongly advised to review all test methods prior to sampling to understand the importance and effects of sampling technique, proper containers, and special handling required for each test method.

4.2 Correct sampling procedures are critical to obtaining a representative sample of the diesel fuel oil to be tested. Refer to Appendix X2 for recommendations. The recommended procedures or practices provide techniques useful in the proper sampling or handling of diesel fuels.

5. Test Methods

5.1 The requirements enumerated in this specification shall be determined in accordance with the following methods:

5.1.1 *Flash Point*—Test Methods D 93, except where other methods are prescribed by law. For all grades, Test Method D 3828 may be used as an alternate with the same limits. For Grades No. 1-D S15, No. 1-D S500 , No. 1-D S5000, No. 2-D

---

[4] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

S15, No. 2-D S500, and No. 2-D S5000, Test Method D 56 may be used as an alternate with the same limits, provided the flash point is below 93°C and the viscosity is below 5.5 mm^2/s at 40°C. This test method will give slightly lower values. In cases of dispute, Test Methods D 93 shall be used as the referee method. Test Method D 56 can not be used as the alternate method for Grade No. 4-D because its minimum viscosity limit is 5.5 mm^2/s at 40°C.

5.1.2 *Cloud Point*—Test Method D 2500. For all fuel grades in Table 1, the automatic Test Methods D 5771, D 5772, or D 5773 can be used as alternates with the same limits. Test Method D 3117 can also be used since it is closely related to Test Method D 2500. In case of dispute, Test Method D 2500 shall be the referee method.

5.1.3 *Water and Sediment*—Test Method D 2709 is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D S15, No. 2-D S500, and No. 2-D S5000. Test Method D 1796 is used for Grade No. 4-D.

5.1.4 *Carbon Residue*—Test Method D 524 is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D

TABLE 1 Detailed Requirements for Diesel Fuel Oils[A]

Property	ASTM Test Method[B]	Grade						
		No. 1-D S15	No. 1-D S500[C]	No. 1-D S5000[D]	No. 2-D S15	No. 2-D S500[C,E]	No. 2-D S5000[D,E]	No. 4-D[D]
Flash Point, °C, min.	D 93	38	38	38	52[E]	52[E]	52[E]	55
Water and Sediment, % vol, max	D 2709	0.05	0.05	0.05	0.05	0.05	0.05	...
	D 1796	...	...	...	...	...	...	0.50
Distillation: one of the following requirements shall be met:								
1. Physical Distillation	D 86							
Distillation Temperature, °C 90 % , % vol recovered								
min		...	...	...	282[E]	282[E]	282[E]	...
max		288	288	288	338	338	338	...
2. Simulated Distillation	D 2887							
Distillation Temperature, °C 90 %, % vol recovered								
min						300[E]	300[E]	
max			304	304		356	356	
Kinematic Viscosity, mm^2/S at 40°C	D 445							
min		1.3	1.3	1.3	1.9[E]	1.9[E]	1.9[E]	5.5
max	...	2.4	2.4	2.4	4.1	4.1	4.1	24.0
Ash % mass, max	D 482	0.01	0.01	0.01	0.01	0.01	0.01	0.10
Sulfur, ppm (µg/g)[F] max	D 5453	15	...	...	15	...	...	...
% mass, max	D 2622[G]	...	0.05	...	...	0.05	...	...
% mass, max	D 129	...	...	0.50	...	...	0.50	2.00
Copper strip corrosion rating max 3 h at 50°C	D 130	No. 3	No. 3	No. 3	No. 3	No. 3	No. 3	...
Cetane number, min[H]	D 613	40[I]	40[I]	40[I]	40[I]	40[I]	40[I]	30[I]
One of the following properties must be met:								
(1) Cetane index, min.	D 976–80[G]	40	40	...	40	40	...	...
(2) Aromaticity, % vol, max	D 1319[G]	35	35	...	35	35	...	...
Operability Requirements								
Cloud point, °C, max	D 2500	[J]	[J]	[J]	[J]	[J]	[J]	...
or								
LTFT/CFPP, °C, max	D 4539/ D 6371							
Ramsbottom carbon residue on 10 % distillation residue, % mass, max	D 524	0.15	0.15	0.15	0.35	0.35	0.35	...
Lubricity, HFRR @ 60°C, micron, max	D 6079	520	520	520	520	520	520	...

[A] To meet special operating conditions, modifications of individual limiting requirements may be agreed upon between purchaser, seller, and manufacturer.

[B] The test methods indicated are the approved referee methods. Other acceptable methods are indicated in 5.1.

[C] Under United States regulations, if Grades No. 1–D S500 or No. 2–D S500 are sold for tax exempt purposes then, at or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lb per thousand barrels of the solid dye standard Solvent Red 26, or the tax must be collected.

[D] Under United States regulations, Grades No.1–D S5000, No. 2–D S5000, and No. 4–D are required by 40 CFR Part 80 to contain a sufficient amount of the dye Solvent Red 164 so its presence is visually apparent. At or beyond terminal storage tanks, they are required by 26 CFR Part 48 to contain the dye Solvent Red 164 at a concentration spectrally equivalent to 3.9 lb per thousand barrels of the solid dye standard Solvent Red 26.

[E] When a cloud point less than −12°C is specified, as can occur during cold months, it is permitted and normal blending practice to combine Grades No. 1 and No. 2 to meet the low temperature requirements. In that case, the minimum flash point shall be 38°C, the minimum viscosity at 40°C shall be 1.7 mm^2/s, and the minimum 90 % recovered temperature shall be waived.

[F] Other sulfur limits can apply in selected areas in the United States and in other countries.

[G] These test methods are specified in 40 CFR Part 80.

[H] Where cetane number by Test Method D 613 is not available, Test Method D 4737 can be used as an approximation.

[I] Low ambient temperatures as well as engine operation at high altitudes may require the use of fuels with higher cetane ratings.

[J] It is unrealistic to specify low temperature properties that will ensure satisfactory operation at all ambient conditions. In general, cloud point (or wax appearance point) Low Temperature Flow Test, and Cold Filter Plugging Point Test may be used as an estimate of operating temperature limits for Grades No. 1–D S500; No. 2–D S500; and No. 1–D S5000 and No. 2–D S5000 diesel fuel oils. However, satisfactory operation below the cloud point (or wax appearance point) may be achieved depending on equipment design, operating conditions, and the use of flow-improver additives as described in X5.1.2. Appropriate low temperature operability properties should be agreed upon between the fuel supplier and purchaser for the intended use and expected ambient temperatures. Test Methods D 4539 and D 6371 may be especially useful to estimate vehicle low temperature operability limits when flow improvers are used. Due to fuel delivery system, engine design, and test method differences, low temperature operability tests may not provide the same degree of protection in various vehicle operating classes. Tenth percentile minimum air temperatures for U.S. locations are provided in Appendix X5 as a means of estimating expected regional temperatures. The tenth percentile minimum air temperatures may be used to estimate expected regional target temperatures for use with Test Methods D 2500, D 4539, and D 6371. Refer to X5.1.3 for further general guidance on test application.

S15, No. 2-D S500 and No. 2-D S5000. Grade No. 4-D does not have a limit for carbon residue.

5.1.5 *Ash*—Test Method D 482 is used for all grades in Table 1.

5.1.6 *Distillation*—Test Method D 86 is used for Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D S15, No. 2-D S500 and No. 2-D S5000. For all grades, Test Method D 2887 can be used as an alternate with the limits listed in Table 1. In case of dispute, Test Method D 86 shall be the referee method. Grade No. 4-D does not have distillation requirements.

5.1.7 *Viscosity*—Test Method D 445 is used for all fuel grades in Table 1.

5.1.8 *Sulfur*—The following list shows the referee test methods and alternate test methods for sulfur, the range over which each test method applies and the corresponding fuel grades.

Sulfur Test Method	Range	Grades
D 129 (referee)	>0.1 mass %	No. 1-D S5000, No. 2-D S5000, No. 4-D
D 1266	0.0005 to 0.4 mass % 5 to 4000 mg/kg (wt ppm)	No. 1-D S500, No. 2-D S500
D 1552	>0.06 mass %	No. 1- D S5000, No. 2-D S5000, No. 4-D
D 2622 (referee for S500 Grades)	0.0003 to 5.3 mass % 3 to 53 000 mg/kg (wt ppm)	All Grades
D 3120	3.0 to 100 mg/kg (wt ppm)	No. 1-D S15, No. 2-D S15 No. 1-D S500, No. 2-D S500 (S500 grades must be diluted before testing)
D 4294	0.0150 to 5.00 mass % 150 to 50 000 mg/kg (wt ppm)	No. 1- D S5000, No. 2-D S5000, No. 4-D
D 5453 (referee for S15 grades)	0.0001 to 0.8 mass % 1.0 to 8000 mg/kg (wt ppm)	All Grades

NOTE 4—The units used to report results in the above test methods are:

D 129	mass %
D 1266	mass %
D 1552	mass %
D 2622	mass %
D 3120	ppm (µg/g)
D 4294	mass %
D 5453	ppm (µg/g)

Results reported in mg/kg and in ppm (µg/g) are numerically the same. The units used in Table 1 for the sulfur requirements are the units in which results for the referee test are reported.

5.1.9 *Copper Corrosion*—Test Method D 130, 3 h test at 50°C. This test method is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 1-D S5000, No. 2-D S15, No. 2-D S500 and No. 2-D S5000. Grade No. 4-D does not have a copper corrosion requirement.

5.1.10 *Cetane Number*—Test Method D 613 is used for all fuel grades in Table 1. Test Method D 6890 is used for all No. 1-D and No. 2-D grades with the DCN result being compared to the cetane number specification requirement of 40. Test Method D 613 shall be the referee method.

5.1.11 *Cetane Index*—Test Methods D 976–80 is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 2-D S15 and No. 2-D S500. Grades No. 1-D S5000, No. 2-D S5000 and No. 4-D do not have an aromatics content requirement, so do not use this test method as a surrogate for aromatics content.

5.1.12 *Aromaticity*—Test Method D 1319. This test method provides an indication of the aromatics content of fuels. For fuels with a maximum final boiling point of 315°C, this method is a measurement of the aromatic content of the fuel. This test method is used for fuel Grades No. 1-D S15, No. 1-D S500, No. 2-D S15 and No. 2-D S500. Grades No. 1-D S5000, No. 2-D S5000 and No. 4-D do not have an aromatics content requirement.

5.1.13 *Lubricity*—Test Method D 6079.

6. Workmanship

6.1 The diesel fuel shall be visually free of undissolved water, sediment, and suspended matter.

7. Requirements

7.1 The grades of diesel fuel oils herein specified shall be hydrocarbon oils conforming to the detailed requirements shown in Table 1.

7.2 *Grades No. 2-D S15, No. 2-D S500 and No. 2–D S5000*—When a cloud point less than −12°C is specified, as can occur during cold months, it is permitted and normal blending practice to combine Grades No. 1 and No. 2 to meet the low temperature requirements. In that case, the minimum flash point shall be 38°C, the minimum viscosity at 40°C shall be 1.7 mm^2/s, and the minimum 90 % recovered temperature shall be waived.

8. Keywords

8.1 diesel; fuel oil; petroleum and petroleum products

APPENDIXES

(Nonmandatory Information)

X1. SIGNIFICANCE OF ASTM SPECIFICATION FOR DIESEL FUEL OILS

X1.1 Introduction

X1.1.1 The properties of commercial fuel oils depend on the refining practices employed and the nature of the crude oils from which they are produced. Distillate fuel oils, for example, may be produced within the boiling range of 150 and 400°C having many possible combinations of various properties, such as volatility, ignition quality, viscosity, and other characteristics.

X1.2 Grades

X1.2.1 This specification is intended as a statement of permissible limits of significant fuel properties used for specifying the wide variety of commercially available diesel fuel oils. Limiting values of significant properties are prescribed for seven grades of diesel fuel oils. These grades and their general applicability for use in diesel engines are broadly indicated as follows:

X1.2.2 *Grade No. 1-D S15*—Grade No. 1-D S15 comprises the class of very low sulfur, volatile fuel oils from kerosine to the intermediate middle distillates. Fuels within this grade are applicable for use in *(1)* high-speed diesel engines and diesel engine applications that require ultra-low sulfur fuels, *(2)* applications necessitating frequent and relatively wide variations in loads and speeds, and *(3)* applications where abnormally low operating temperatures are encountered.

X1.2.3 *Grade No. 1-D S500*—Grade No. 1-D S500 comprises the class of low-sulfur, volatile fuel oils from kerosine to the intermediate middle distillates. Fuels within this grade are applicable for use in *(1)* high-speed diesel engines that require low sulfur fuels, *(2)* in applications necessitating frequent and relatively wide variations in loads and speeds, and *(3)* in applications where abnormally low operating temperatures are encountered.

X1.2.4 *Grade No. 1-D S5000*—Grade No. 1-D S5000 comprises the class of volatile fuel oils from kerosine to the intermediate middle distillates. Fuels within this grade are applicable for use in high-speed diesel engines applications necessitating frequent and relatively wide variations in loads and speeds, and also for use in cases where abnormally low operating temperatures are encountered.

X1.2.5 *Grade No. 2-D S15*—Grade No. 2-D S15 includes the class of very low sulfur, middle distillate gas oils of lower volatility than Grade No. 1-D S15. These fuels are applicable for use in *(1)* high speed diesel engines and diesel engine applications that require ultra-low sulfur fuels, *(2)* applications necessitating relatively high loads and uniform speeds, or *(3)* diesel engines not requiring fuels having higher volatility or other properties specified in Grade No. 1-D S15.

X1.2.6 *Grade No. 2-D S500*—Grade No. 2-D S500 includes the class of low-sulfur, middle distillate gas oils of lower volatility than Grade No. 1-D S500. These fuels are applicable for use in *(1)* high-speed diesel engine applications that require low sulfur fuels, *(2)* applications necessitating relatively high loads and uniform speeds, or *(3)* diesel engines not requiring fuels having higher volatility or other properties specified for Grade No. 1-D S500.

X1.2.7 *Grade No. 2-D S5000*—Grade No. 2-D S5000 includes the class of middle distillate gas oils of lower volatility than Grade No. 1-D S5000. These fuels are applicable for use in *(1)* high-speed diesel engines in applications necessitating relatively high loads and uniform speeds, or *(2)* in diesel engines not requiring fuels having higher volatility or other properties specified for Grade No. 1-D S5000.

X1.2.8 *Grade No. 4-D*—Grade No. 4-D comprises the class of more viscous middle distillates and blends of these middle distillates with residual fuel oils. Fuels within this grade are applicable for use in low- and medium-speed diesel engines in applications necessitating sustained loads at substantially constant speed.

X1.3 Selection of Particular Grade

X1.3.1 The selection of a particular diesel fuel oil from one of these seven ASTM grades for use in a given engine requires consideration of the following factors:

X1.3.1.1 Fuel price and availability,

X1.3.1.2 Maintenance considerations,

X1.3.1.3 Engine size and design,

X1.3.1.4 Emission control systems,

X1.3.1.5 Speed and load ranges,

X1.3.1.6 Frequency of speed and load changes, and

X1.3.1.7 Atmospheric conditions. Some of these factors can influence the required fuel properties outlined as follows:

X1.4 Cetane Number

X1.4.1 Cetane number is a measure of the ignition quality of the fuel and influences combustion roughness. The cetane number requirements depend on engine design, size, nature of speed and load variations, and on starting and atmospheric conditions. Increase in cetane number over values actually required does not materially improve engine performance. Accordingly, the cetane number specified should be as low as possible to assure maximum fuel availability.

X1.5 Distillation

X1.5.1 The fuel volatility requirements depend on engine design, size, nature of speed and load variations, and starting and atmospheric conditions. For engines in services involving rapidly fluctuating loads and speeds as in bus and truck operation, the more volatile fuels may provide best performance, particularly with respect to smoke and odor. However, best fuel economy is generally obtained from the heavier types of fuels because of their higher heat content.

X1.6 Viscosity

X1.6.1 For some engines it is advantageous to specify a minimum viscosity because of power loss due to injection pump and injector leakage. Maximum viscosity, on the other hand, is limited by considerations involved in engine design and size, and the characteristics of the injection system.

X1.7 Carbon Residue

X1.7.1 Carbon residue gives a measure of the carbon depositing tendencies of a fuel oil when heated in a bulb under prescribed conditions. While not directly correlating with engine deposits, this property is considered an approximation.

X1.8 Sulfur

X1.8.1 The effect of sulfur content on engine wear and deposits appears to vary considerably in importance and depends largely on operating conditions. Fuel sulfur can affect emission control systems performance. To assure maximum availability of fuels, the permissible sulfur content should be specified as high as is practicable, consistent with maintenance considerations.

X1.9 Flash Point

X1.9.1 The flash point as specified is not directly related to engine performance. It is, however, of importance in connection with legal requirements and safety precautions involved in fuel handling and storage, and is normally specified to meet insurance and fire regulations.

X1.10 Cloud Point

X1.10.1 Cloud point is of importance in that it defines the temperature at which a cloud or haze of wax crystals appears in the oil under prescribed test conditions which generally relates to the temperature at which wax crystals begin to precipitate from the oil in use.

X1.11 Ash

X1.11.1 Ash-forming materials may be present in fuel oil in two forms: *(1)* abrasive solids, and *(2)* soluble metallic soaps. Abrasive solids contribute to injector, fuel pump, piston and ring wear, and also to engine deposits. Soluble metallic soaps have little effect on wear but may contribute to engine deposits.

X1.12 Copper Strip Corrosion

X1.12.1 This test serves as a measure of possible difficulties with copper and brass or bronze parts of the fuel system.

X1.13 Aromaticity

X1.13.1 This test is used as an indication of the aromatics content of diesel fuel. Aromatics content is specified to prevent an increase in the average aromatics content in Grades No. 1-D S15, No. 1-D S500, No. 2-D S15 and No. 2-D S500 fuels and is required by 40 CFR Part 80. Increases in aromatics content of fuels over current levels may have a negative impact on emissions.

X1.14 Cetane Index

X1.14.1 Cetane Index is specified as a limitation on the amount of high aromatic components in Grades No. 1-D S15, No. 1-D S500, No. 2-D S15 and No. 2-D S500.

X1.15 Other

X1.15.1 *Microbial Contamination*—Refer to Guide D 6469 for a discussion of this form of contamination.

X2. SAMPLING, CONTAINERS AND SAMPLE HANDLING

X2.1 Introduction

X2.1.1 This appendix provides guidance on methods and techniques for the proper sampling of diesel fuel oils. As diesel fuel oil specifications become more stringent and contaminants and impurities become more tightly controlled, even greater care needs to be taken in collecting and storing samples for quality assessment.

X2.2 Sampling, Containers and Sample Handling Recommendations

X2.2.1 Appropriate manual method sampling procedures can be found in Practice D 4057 and automatic method sampling is covered in Practice D 4177.

X2.2.2 The correct sample volume and appropriate container selection are also important decisions that can impact test results. Practice D 4306 for aviation fuel container selection for tests sensitive to trace contamination may be useful. Practice D 5854 for procedures on container selection and sample mixing and handling is recommended. For cetane number determination protection from light is important. Collection and storage of diesel fuel oil samples in an opaque container, such as a dark brown glass bottle, metal can, or a minimally reactive plastic container to minimize exposure to UV emissions from sources such as sunlight or fluorescent lamps, is recommended. According to Paragraph 8.2 of Test Method D 6079, "Because of sensitivity of lubricity measurements to trace materials, sample containers shall be only fully epoxy-lined metal, amber borosilicate glass, or polytetrafluoroethylene as specified in Practice D 4306."

X2.2.3 For volatility determination of a sample, Practice D 5842 for special precautions recommended for representative sampling and handling techniques may be appropriate.

X3. STORAGE AND THERMAL STABILITY OF DIESEL FUELS

X3.1 Scope

X3.1.1 This appendix provides guidance for consumers of diesel fuels who may wish to store quantities of fuels for extended periods or use the fuel in severe service or high temperature applications. Fuels containing residual components are excluded. Consistently successful long-term fuel storage or use in severe applications requires attention to fuel selection, storage conditions, handling and monitoring of properties during storage and prior to use.

X3.1.2 Normally produced fuels have adequate stability properties to withstand normal storage and use without the formation of troublesome amounts of insoluble degradation products. Fuels that are to be stored for prolonged periods or used in severe applications should be selected to avoid formation of sediments or gums, which can overload filters or plug injectors. Selection of these fuels should result from supplier-user discussions.

X3.1.3 These suggested practices are general in nature and should not be considered substitutes for any requirements imposed by the warranty of the distillate fuel equipment manufacturer or by federal, state, or local government regulations. Although they cannot replace a knowledge of local conditions or good engineering and scientific judgment, these suggested practices do provide guidance in developing an individual fuel management system for the middle distillate fuel user. They include suggestions in the operation and maintenance of existing fuel storage and handling facilities and for identifying where, when, and how fuel quality should be monitored or selected for storage or severe use.

X3.2 Definitions

X3.2.1 *bulk fuel*—fuel in the storage facility.

X3.2.2 *fuel contaminants*—foreign materials that make fuel less suitable or unsuitable for the intended use.

X3.2.2.1 *Discussion*—Fuel contaminants include materials introduced subsequent to the manufacture of fuel and fuel degradation products.

X3.2.3 *fuel-degradation products*—those materials that are formed in fuel during extended storage or exposure to high temperatures.

X3.2.3.1 *Discussion*—Insoluble degradation products may combine with other fuel contaminants to reinforce deleterious effects. Soluble degradation products (soluble gums) are less volatile than fuel and may carbonize to form deposits due to complex interactions and oxidation of small amounts of olefinic or sulfur-, oxygen- or nitrogen-containing compounds present in fuels. The formation of degradation products may be catalyzed by dissolved metals, especially copper salts. When dissolved copper is present it can be deactivated with metal deactivator additives.

X3.2.4 *long-term storage*—storage of fuel for longer than 12 months after it is received by the user.

X3.2.5 *severe use*—use of the fuel in applications which may result in engines operating under high load conditions that may cause the fuel to be exposed to excessive heat.

X3.3 Fuel Selection

X3.3.1 Certain distilled refinery products are generally more suitable for long-term storage and severe service than others. The stability properties of middle distillates are highly dependent on the crude oil sources, severity of processing, use of additives and whether additional refinery treatment has been carried out.

X3.3.2 The composition and stability properties of middle distillate fuels produced at specific refineries may be different. Any special requirements of the user, such as long-term storage or severe service, should be discussed with the supplier.

X3.3.3 Blends of fuels from various sources may interact to give stability properties worse than expected based on the characteristics of the individual fuels.

X3.4 Fuel Additives

X3.4.1 Available fuel additives can improve the suitability of marginal fuels for long-term storage and thermal stability, but may be unsuccessful for fuels with markedly poor stability properties. Most additives should be added at the refinery or during the early weeks of storage to obtain maximum benefits.

X3.4.2 Biocides or biostats destroy or inhibit the growth of fungi and bacteria, which can grow at fuel-water interfaces to give high particulate concentrations in the fuel. Available biocides are soluble in both the fuel and water or in the water phase only.

X3.5 Tests for Fuel Quality

X3.5.1 At the time of manufacture, the storage stability of fuel may be assessed using Test Method D 2274 or D 5304. However, these accelerated stability tests may not correlate well with field storage stability due to varying field conditions and to fuel composition.

X3.5.2 Performance criteria for accelerated stability tests that assure satisfactory long-term storage of fuels have not been established.

X3.5.3 Test Method D 6468, provides an indication of thermal oxidative stability of middle distillate fuels when heated to temperatures near 150°C.

X3.6 Fuel Monitoring

X3.6.1 A plan for monitoring the quality of bulk fuel during prolonged storage is an integral part of a successful program. A plan to replace aged fuel with fresh product at established intervals is also desirable.

X3.6.2 Stored fuel should be periodically sampled and its quality assessed. Practice D 4057 provides guidance for sampling. Fuel contaminants and degradation products will usually settle to the bottom of a quiescent tank. A "Bottom" or "Clearance" sample, as defined in Practice D 4057, should be included in the evaluation along with an "All Level" sample.

X3.6.3 The quantity of insoluble fuel contaminants present in fuel can be determined using Test Method D 6217.

X3.6.4 Test Method D 6468, can be used for investigation of operational problems that might be related to fuel thermal stability. Testing samples from the fuel tank or from bulk storage may give an indication as to the cause of filter plugging. It is more difficult to monitor the quality of fuels in vehicle tanks since operation may be on fuels from multiple sources.

X3.6.5 Some additives exhibit effects on fuels tested in accordance with Test Method D 6468 that may or may not be observed in the field. Data have not been developed that correlate results from the test method for various engine types and levels of operating severity.

X3.7 Fuel Storage Conditions

X3.7.1 Contamination levels in fuel can be reduced by storage in tanks kept free of water, and tankage should have provisions for water draining on a scheduled basis. Water promotes corrosion, and microbiological growth may occur at a fuel-water interface. Underground storage is preferred to avoid temperature extremes; above-ground storage tanks should be sheltered or painted with reflective paint. High storage temperatures accelerate fuel degradation. Fixed roof tanks should be kept full to limit oxygen supply and tank breathing.

X3.7.2 Copper and copper-containing alloys should be avoided. Copper can promote fuel degradation and may produce mercaptide gels. Zinc coatings can react with water or organic acids in the fuel to form gels that rapidly plug filters.

X3.7.3 Appendix X2 of Specification D 2880 discusses fuel contaminants as a general topic.

X3.8 Fuel Use Conditions

X3.8.1 Many diesel engines are designed so that the diesel fuel is used for heat transfer. In modern heavy-duty diesel engines, for example, only a portion of the fuel that is circulated to the fuel injectors is actually delivered to the combustion chamber. The remainder of the fuel is circulated back to the fuel tank, carrying heat with it. Thus adequate high temperature stability can be a necessary requirement in some severe applications or types of service.

X3.8.2 Inadequate high temperature stability may result in the formation of insoluble degradation products.

X3.9 Use of Degraded Fuels

X3.9.1 Fuels that have undergone mild-to-moderate degradation can often be consumed in a normal way, depending on the fuel system requirements. Filters and other cleanup equipment can require special attention and increased maintenance. Burner nozzle or injector fouling can occur more rapidly.

X3.9.2 Fuels containing very large quantities of fuel degradation products and other contaminants or with runaway microbiological growth require special attention. Consultation with experts in this area is desirable. It can be possible to drain the sediment or draw off most of the fuel above the sediment layer and use it with the precautions described in X3.9.1. However, very high soluble gum levels or corrosion products from microbiological contamination can cause severe operational problems.

X3.10 Thermal Stability Guidelines

X3.10.1 Results from truck fleet experience suggests that Test Method D 6468 can be used to qualitatively indicate whether diesel fuels have satisfactory thermal stability performance properties.[5,6]

X3.10.2 Performance in engines has not been sufficiently correlated with results from Test Method D 6468 to provide definitive specification requirements. However, the following guidelines are suggested.

X3.10.2.1 Fuels giving a Test Method D 6468 reflectance value of 70 % or more in a 90 minute test at the time of manufacture should give satisfactory performance in normal use.

X3.10.2.2 Fuels giving a Test Method D 6468 reflectance value of 80 % or more in a 180 minute test at the time of manufacture should give satisfactory performance in severe use.

X3.10.3 Thermal stability as determined by Test Method D 6468 is known to degrade during storage.[7] The guidance above is for fuels used within six months of manufacture.

X4. DIESEL FUEL LUBRICITY

X4.1 Introduction

X4.1.1 Diesel fuel functions as a lubricant in most components of fuel injection equipment such as pumps and injectors. In limited cases, fuel with specific properties will have insufficient lubricating properties which will lead to a reduction in the normal service life and functional performance of diesel fuel injection systems.

X4.2 Fuel Characteristics Affecting Equipment Wear

X4.2.1 Currently, two fuel characteristics affect equipment wear. These are low viscosity and lack of sufficient quantities of trace components that have an affinity for surfaces. If fuel viscosity meets the requirements of a particular engine, a fuel film is maintained between the moving surfaces of the fuel system components. This prevents excessive metal-to-metal

---

[5] Bacha, John D., and Lesnini, David G., "Diesel Fuel Thermal Stability at 300°F," *Proceedings of the 6th International Conference on Stability and Handling of Liquid Fuels*, Vancouver, B.C., October 1997.

[6] Schwab, Scott D., Henly, Timothy J., Moxley, Joel F., and Miller, Keith, "Thermal Stability of Diesel Fuel," *Proceedings of the 7th International Conference on Stability and Handling of Liquid Fuels* , Graz, Austria September 2000.

[7] Henry, C. P., "The DuPont F21 149°C (300°F) Accelerated Stability Test," *Distillate Fuel Stability and Cleanliness, ASTM STP 751*, 1981, pp. 22-33.

contact and avoids premature failure due to wear. Similarly, certain surface active molecules in the fuel adhere to, or combine with, surfaces to produce a protective film which also can protect surfaces against excessive wear.

X4.3 Fuel Lubricity

X4.3.1 The concern about fuel lubricity is limited to situations in which fuels with lower viscosities than those specified for a particular engine are used or in which fuels that have been processed in a manner that results in severe reduction of the trace levels of the surface active species that act as surface protecting agents. Presently the only fuels of the latter type shown to have lubricity problems resulted from sufficiently severe processing to reduce aromatics or sulfur.

X4.3.2 Work in the area of diesel fuel lubricity is ongoing by several organizations, such as the International Organization for Standardization (ISO), the ASTM Diesel Fuel Lubricity Task Force, and the Coordinating Research Council (CRC) Diesel Performance Group. These groups include representatives from the fuel injection equipment manufacturers, fuel producers, and additive suppliers. The charge of the ASTM task force has been the recommendation of test methods and fuel lubricity requirements for Specification D 975. Two test methods were proposed and approved. These are Test Method D 6078, a scuffing load ball-on-cylinder lubricity evaluator method, SLBOCLE, and Test Method D 6079, a high frequency reciprocating rig (HFRR) method. Use of these tests raises three issues: 1) The correlation of the data among the two test methods and the fuel injection equipment is not perfect, 2) Both methods in their current form do not apply to all fuel-additive combinations, and 3) The reproducibility values for both test methods are large. In order to protect diesel fuel injection equipment, an HFRR Wear Scar Diameter (WSD) of 520 microns has been placed in Specification D 975.[8]

X4.3.3 Most experts agree that fuels having a SLBOCLE lubricity value below 2000 g might not prevent excessive wear in injection equipment[9] while fuels with values above 3100 g should provide sufficient lubricity in all cases.[10] Experts also agree that if HFFR test at 60°C is used, fuels with values above 600 microns might not prevent excessive wear,[11] while fuels with values below 450 microns should provide sufficient lubricity in all cases.[10] More accurately, an industry-accepted long-term durability pump test, such as Test Method D 6898, can be used to evaluate the lubricity of a diesel fuel. A poor result in such a test indicates that the fuel has low lubricity and may not be able to provide sufficient protection.

NOTE X4.1—Some injection equipment can be fitted with special components that can tolerate low lubricity fuels.

---

[8] Mitchell, K., "Diesel Fuel Lubricity—Base Fuel Effects," SAE Technical Paper 2001–01–1928, 2001.

[9] Westbrook, S. R., "Survey of Low Sulfur Diesel Fuels and Aviation Kerosenes from U.S. Military Installations," SAE Technical Paper 952369, 1995.

[10] Nikanjam, M., "ISO Diesel Fuel Lubricity Round Robin Program," SAE Technical Paper 952372, 1995.

[11] Nikanjam, M., "*Diesel Fuel Lubricity: On the Path to Specifications*," SAE Technical Paper 1999-01-1479, 1999.

X5. TENTH PERCENTILE MINIMUM AMBIENT AIR TEMPERATURES FOR THE UNITED STATES (EXCEPT HAWAII)

X5.1 Introduction

X5.1.1 The tenth percentile minimum ambient air temperatures shown on the following maps (Figs. X5.1-X5.12) and in Table X5.1 were derived from an analysis of historical hourly temperature readings recorded over a period of 15 to 21 years from 345 weather stations in the United States. This study was conducted by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, Aberdeen Proving Ground, MD 21005. The tenth percentile minimum ambient air temperature is defined as the lowest ambient air temperature which will not go lower on average more than 10 % of the time. In other words, the daily minimum ambient air temperature would on average not be expected to go below the monthly tenth percentile minimum ambient air temperature more than 3 days for a 30-day month. See Table X5.1.

X5.1.2 These data may be used to estimate low temperature operability requirements. In establishing low temperature operability requirements, consideration should be given to the following. These factors, or any combination, may make low temperature operability more or less severe than normal. As X5.1.2.1 through X5.1.2.12 indicate, field work suggests that cloud point (or wax appearance point) is a fair indication of the low temperature operability limit of fuels without cold flow additives in most vehicles.

X5.1.2.1 Long term weather patterns (Average winter low temperatures will be exceeded on occasion).

X5.1.2.2 Short term local weather conditions (Unusual cold periods do occur).

X5.1.2.3 Elevation (High locations are usually colder than surrounding lower areas).

X5.1.2.4 Specific engine design.

X5.1.2.5 Fuel system design (Recycle rate, filter location, filter capacity, filter porosity, and so forth.)

X5.1.2.6 Fuel viscosity at low temperatures

X5.1.2.7 Equipment add-ons (Engine heaters, radiator covers, fuel line and fuel filter heaters and so forth.)

X5.1.2.8 Types of operation (Extensive idling, engine shutdown, or unusual operation).

X5.1.2.9 Low temperature flow improver additives in fuel.

X5.1.2.10 Geographic area for fuel use and movement between geographical areas.

X5.1.2.11 General housekeeping (Dirt and/or water in fuel or fuel supply system).

X5.1.2.12 Impact failure for engine to start or run (Critical vs. non-critical application).

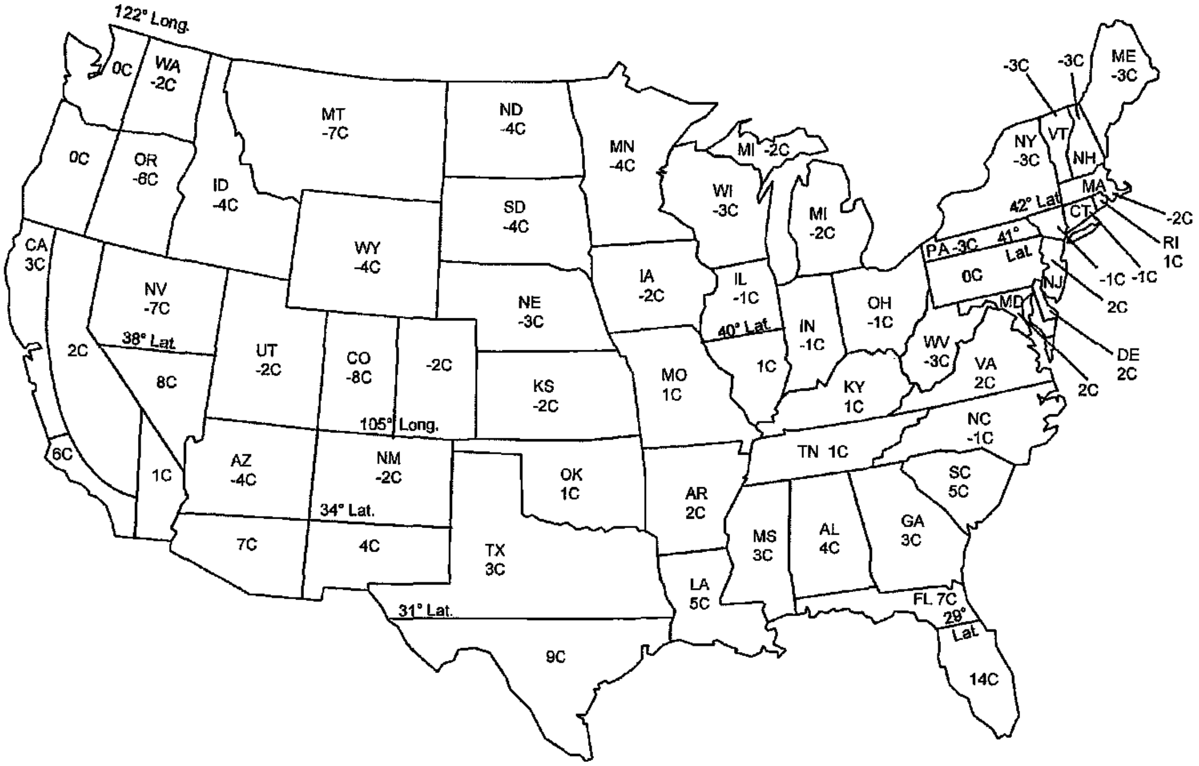


FIG. X5.1 October—10th Percentile Minimum Temperatures

X5.1.3 *Historical Background*—Three test methods have been widely used to estimate or correlate with low temperature vehicle operability. Cloud point, Test Method D 2500, is the oldest of the three and most conservative of the tests. The cloud point test indicates the earliest appearance of wax precipitation that might result in plugging of fuel filters or fuel lines under prescribed cooling conditions. Although not 100 % failsafe, it is the most appropriate test for applications that can not tolerate much risk. The Cold Filter Plugging Point (CFPP) test, Test Method D 6371, was introduced in Europe in 1965. The CFPP was designed to correlate with the majority of European vehicles. Under rapid cooling conditions, 20 cc fuel is drawn through a 45 micron screen then allowed to flow back through the screen for further cooling. This process is continued every 1°C until either the 20 cc fuel fails to be drawn through the screen in 60 s or it fails to return through the screen in 60 s. It was field tested many times in Europe[12] before being widely accepted as a European specification. Field tests have also shown CFPP results more than 10°C below the cloud point should be viewed with caution because those results did not necessarily reflect the true vehicle low temperature operability limits.[13] CFPP has been applied to many areas of the world where similar vehicle designs are used. The Low Temperature Flow Test (LTFT), Test Method D 4539, was designed to correlate with the most severe and one of the most common fuel delivery systems used in North American Heavy Duty trucks. Under prescribed slow cool conditions (1°C/h), similar to typical field conditions, several 200 cc fuel specimens in glass containers fitted with 17 µm screen assemblies are cooled. At 1°C intervals one specimen is drawn through the screen under a 20 kPa vacuum. Approximately 90 % of the fuel must come over in 60 s or less for the result to be a pass. This process is continued at lower temperatures (1°C increments) until the fuel fails to come over in the allotted 60 s. The lowest passing temperature is defined as the LTFT for that fuel. In 1981, a CRC program was conducted to evaluate the efficacy of cloud point, CFPP, pour point, and LTFT for protecting the diesel vehicle population in North America and to determine what benefit flow-improvers could provide. The field test consisted of 3 non-flow improved diesel fuels, 5 flow improved diesel fuels, 4 light-duty passenger cars, and 3 heavy-duty trucks. The field trial resulted in two documents[14,15] that provide insight into correlating laboratory tests to North

---

[12] "Low Temperature Operability of Diesels. A Report by CEC Investigation Group IGF-3," CEC P-171-82.

[13] "SFPP-A New Laboratory Test for Assessment of Low Temperature Operability of Modern Diesel Fuels," CEC/93/EF 15, 5–7, May 1993.

[14] CRC Report No. 537, "The Relationship Between Vehicle Fuel Temperature and Ambient Temperature, 1981 CRC Kapuskasing Field Test," December 1983.

[15] CRC Report No. 528, "1981 CRC Diesel Fuel Low-Temperature Operability Field Test," September 1983.

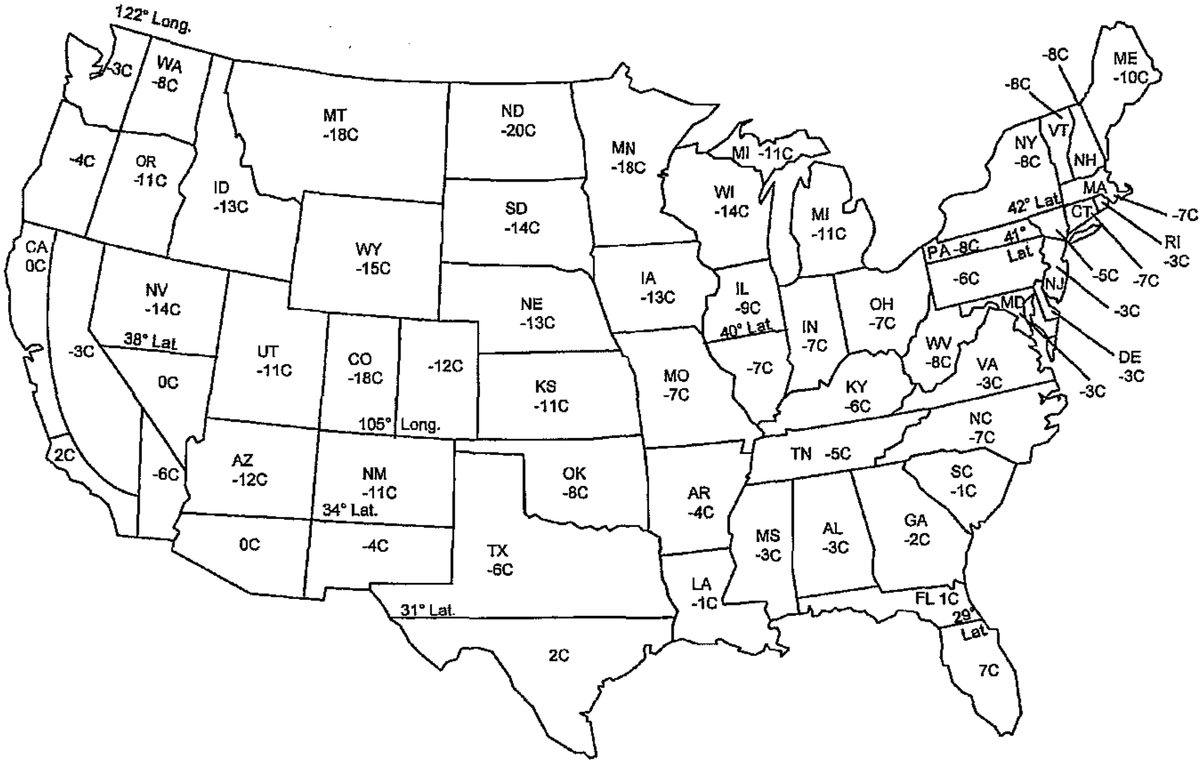


FIG. X5.2 November—10th Percentile Minimum Ambient Air Temperatures

American vehicle performance in the field. The general conclusions of the study were:

(1) In overnight cool down, 30 % of the vehicles tested had a final fuel tank temperature within 2°C of the overnight minimum ambient temperature.

(2) The use of flow-improved diesel fuel permits some vehicles to operate well below the fuel cloud point.

(3) Significant differences exist in the severity of diesel vehicles in terms of low temperature operation.

(4) No single laboratory test was found that adequately predicts the performance of all fuels in all vehicles.

(5) CFPP was a better predictor than pour point, but both methods over-predicted, minimum operating temperatures in many vehicles. For this reason, these tests were judged inadequate predictors of low-temperature performance and dismissed from further consideration.

(6) Cloud point and LTFT showed varying degrees of predictive capability, and offered distinctively different advantages. Both predicted the performance of the base fuels well, but LTFT more accurately predicted the performance of the flow-improved fuels. On the other hand, cloud point came closest to a fail-safe predictor of vehicle performance for all vehicles.

Since the 1981 field test, non-independent studies[16] using newer vehicles verified the suitability of the LTFT for North American heavy-duty trucks. Users are advised to review these and any more recent publications when establishing low temperature operability requirements and deciding upon test methods.

X5.1.3.1 *Current Practices*—It is recognized that fuel distributors, producers, and end users in the United States use cloud point, wax appearance point, CFPP, and LTFT to estimate vehicle low temperature operability limits for diesel fuel. No independent data has been published in recent years to determine test applicability for today's fuels and vehicles.

X5.2 Maps

X5.2.1 The maps in the following figures were derived from CCL Report No. 316, "A Predictive Study for Defining Limiting Temperatures and Their Application in Petroleum Product Specifications," by John P. Doner. This report was published by the U.S. Army Mobility Equipment Research and Development Center (USAMERDC), Coating and Chemical Laboratory, and it is available from the National Technical

---

[16] SAE 962197, SAE 982576, SAE 2000-01-2883.

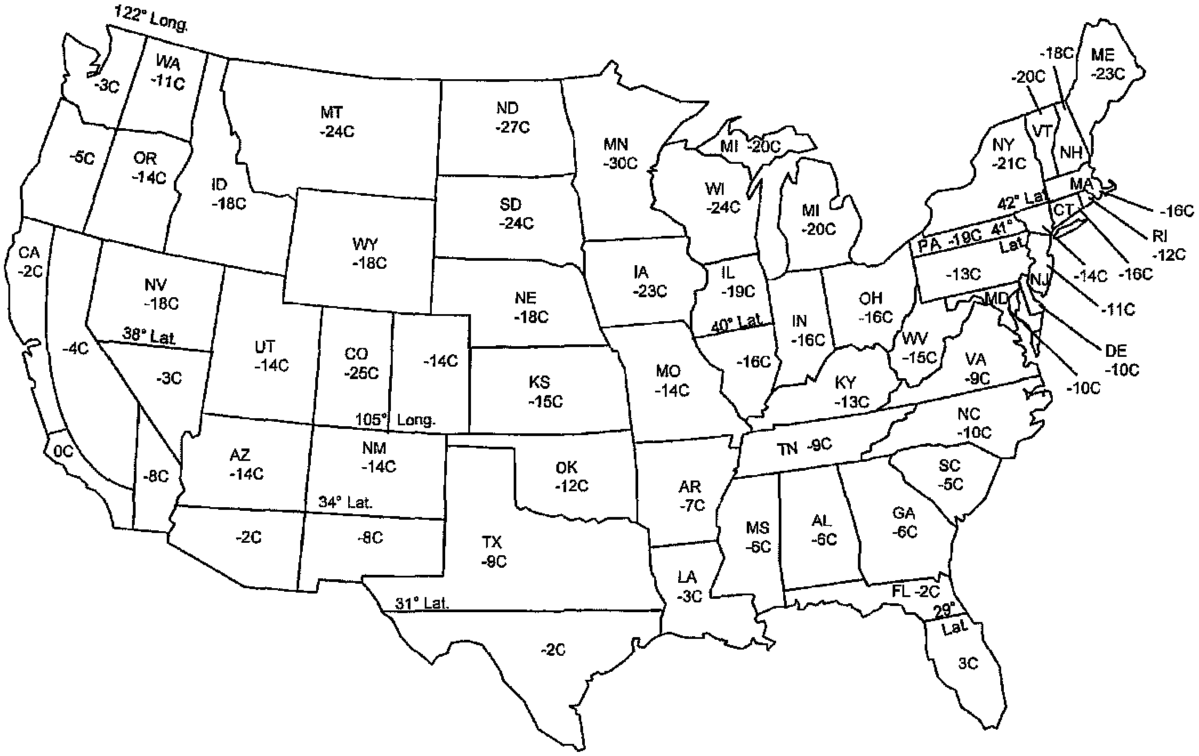


FIG. X5.3 December—10th Percentile Minimum Ambient Air Temperatures

Information Service, Springfield, VA 22151, by requesting Publication No. AD756-420.

X5.2.2 Where states are divided the divisions are noted on the maps and table with the exception of California, which is divided by counties as follows:

California, North Coast—Alameda, Contra Costa, Del Norte, Humbolt, Lake, Marin, Mendocino, Monterey, Napa, San Benito, San Francisco, San Mateo, Santa Clara, Santa Cruz, Solano, Sonoma, Trinity.

California, Interior—Lassen, Modoc, Plumas, Sierra, Siskiyou, Alpine, Amador, Butte, Calaveras, Colusa, El Dorado, Fresno, Glenn, Kern (except that portion lying east of the Los Angeles County Aqueduct), Kings, Madera, Mariposa, Merced, Placer, Sacramento, San Joaquin, Shasta, Stanislaus, Sutter, Tehama, Tulare, Tuolumne, Yolo, Yuba, Nevada.

California, South Coast—Orange, San Diego, San Luis Obispo, Santa Barbara, Ventura, Los Angeles (except that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct).

California, Southeast—Imperial, Riverside, San Bernardino, Los Angeles (that portion north of the San Gabriel Mountain range and east of the Los Angeles County Aqueduct), Mono, Inyo, Kern (that portion lying east of the Los Angeles County Aqueduct).

X5.2.3 The temperatures in CCL Report No. 316 were in degrees Fahrenheit. The degree Celsius temperatures in Appendix X5 were obtained by converting the original degree Fahrenheit temperatures.

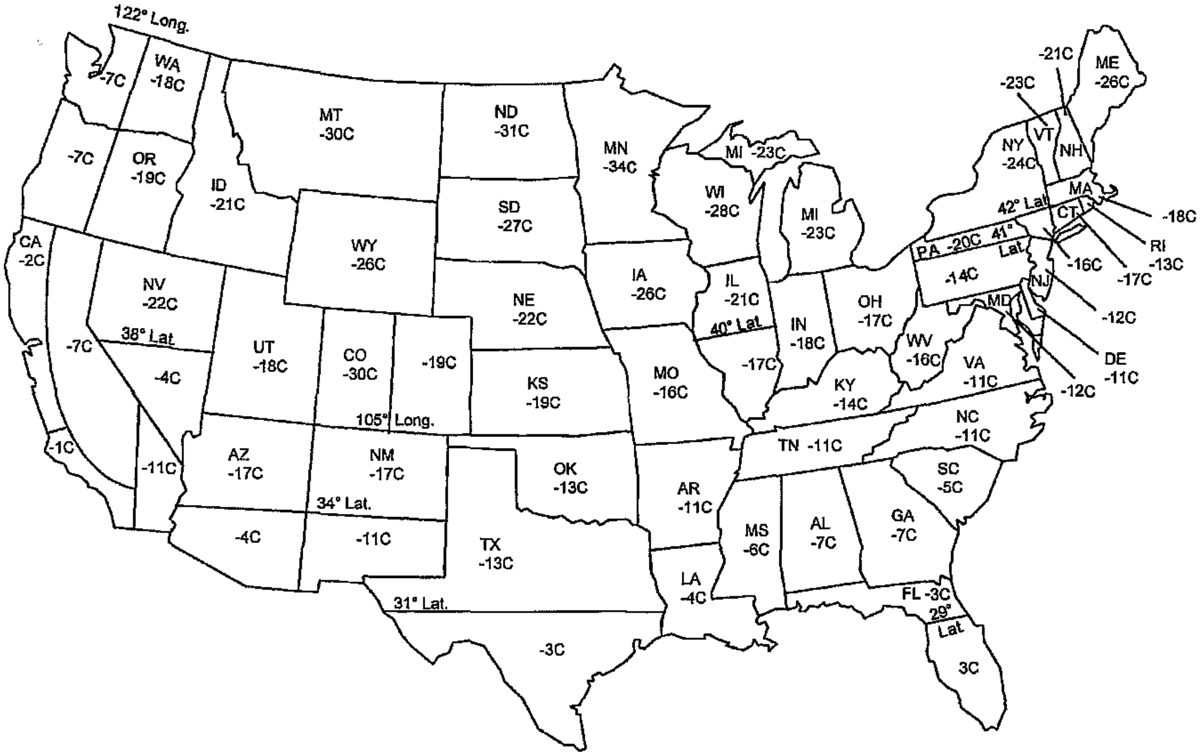


FIG. X5.4 January—10th Percentile Minimum Ambient Air Temperatures

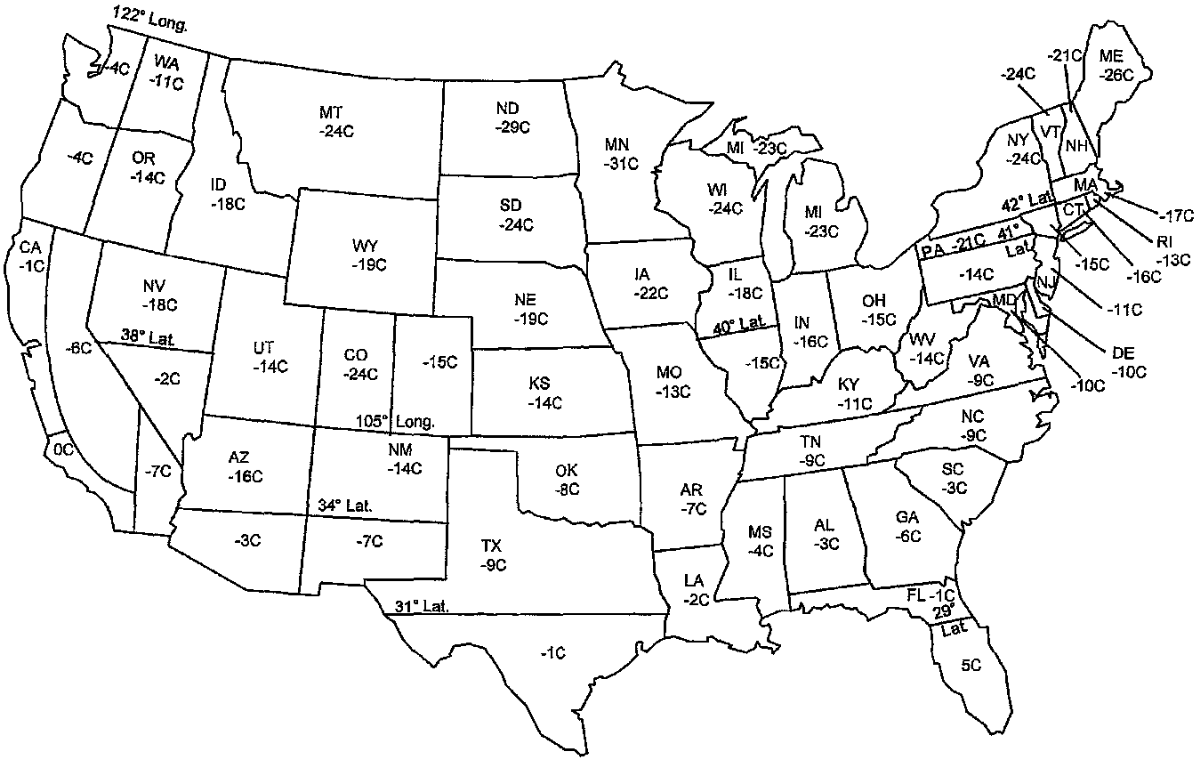


FIG. X5.5 February—10th Percentile Minimum Ambient Air Temperatures

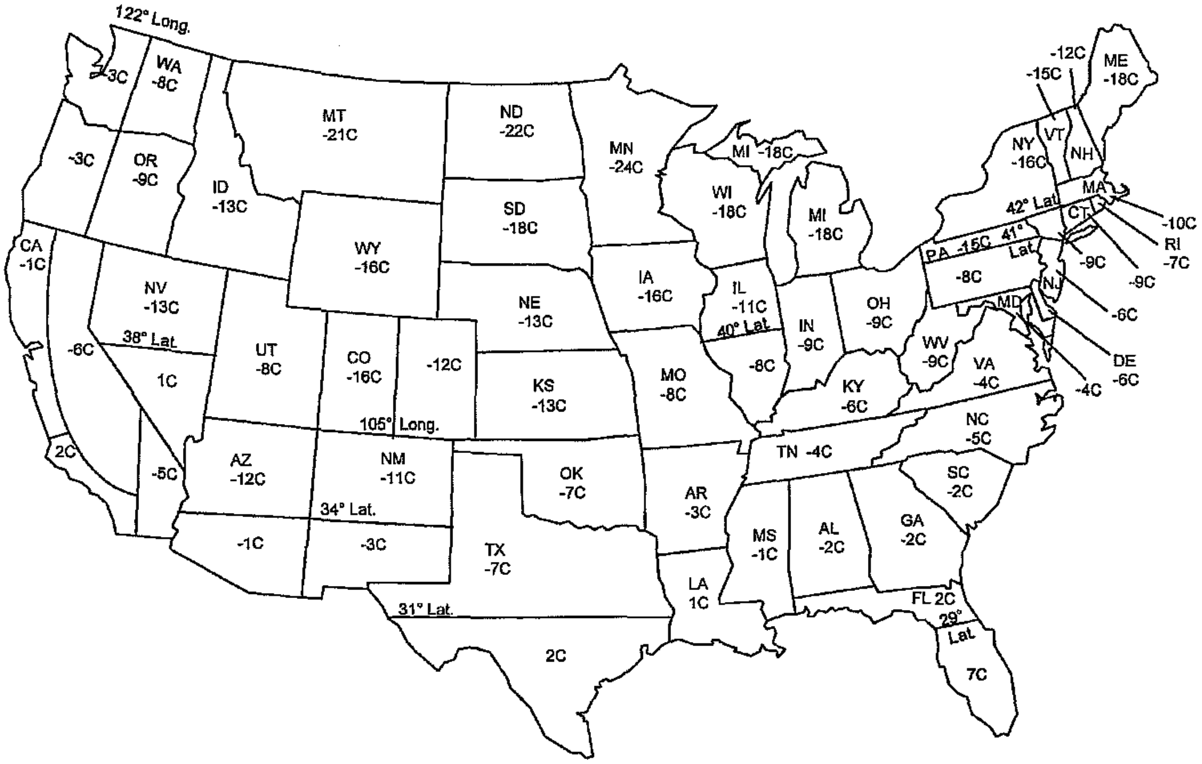


FIG. X5.6 March—10th Percentile Minimum Ambient Air Temperatures

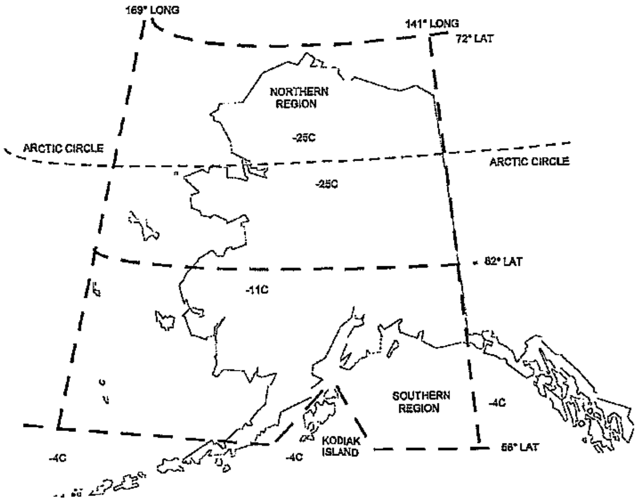


FIG. X5.7 October—10th Percentile Minimum Ambient Air Temperatures

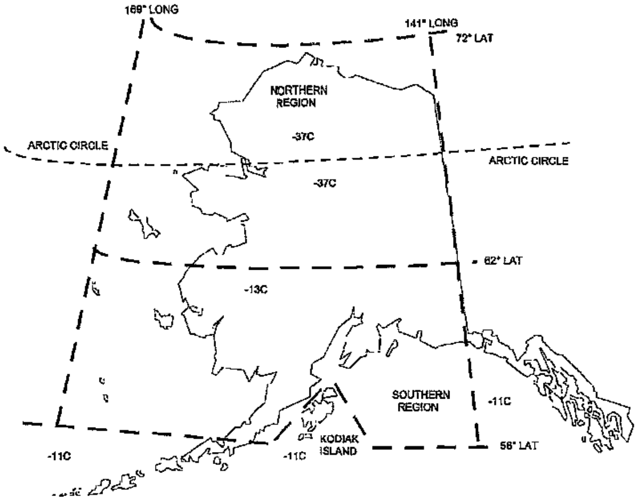


FIG. X5.8 November—10th Percentile Minimum Ambient Air Temperatures

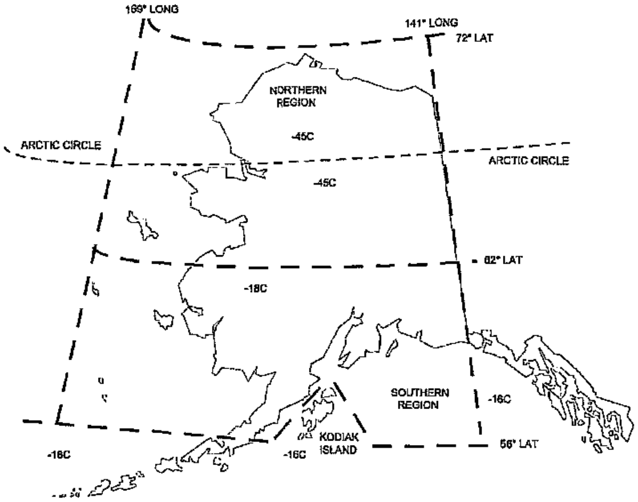


FIG. X5.9 December—10th Percentile Minimum Ambient Air Temperatures

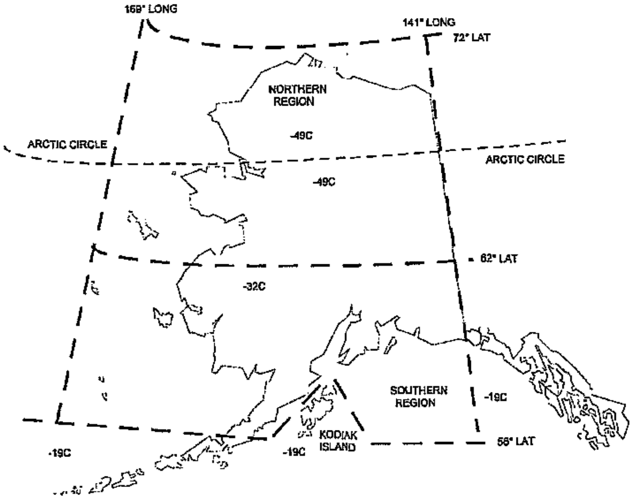


FIG. X5.10 January—10th Percentile Minimum Ambient Air Temperatures

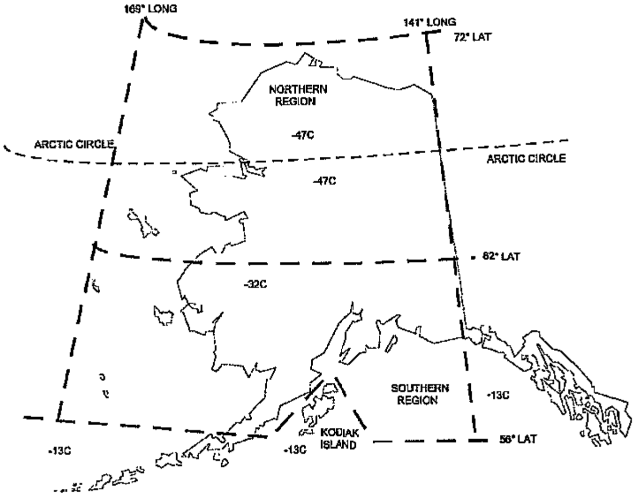


FIG. X5.11 February—10th Percentile Minimum Ambient Air Temperatures

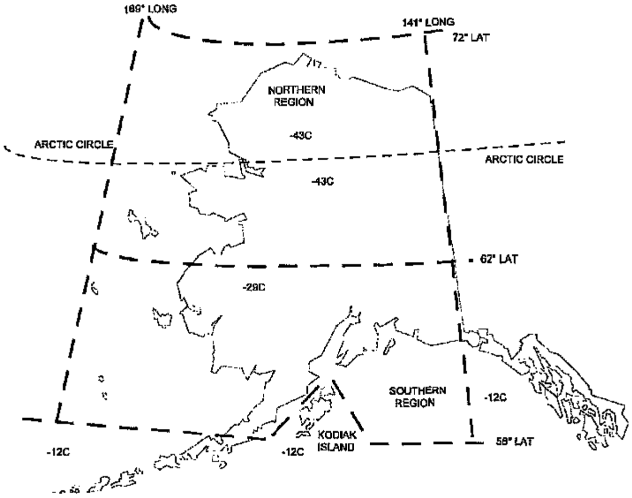


FIG. X5.12 March—10th Percentile Minimum Ambient Air Temperatures

TABLE X5.1 Tenth Percentile Minimum Ambient Air Temperatures for the United States (except Hawaii)

State		10th Percentile Temperature°C, min					
		Oct.	Nov.	Dec.	Jan.	Feb.	March
Alabama		4	−3	−6	−7	−3	−2
Alaska	Northern	−25	−37	−45	−49	−47	−43
	Southern	−11	−13	−18	−32	−32	−29
	South East	−4	−11	−16	−19	−13	−12
Arizona	North 34° latitude	−4	−12	−14	−17	−16	−12
	South 34° latitude	7	0	−2	−4	−3	−1
Arkansas		2	−4	−7	−11	−7	−3
California	North Coast	3	0	−2	−2	−1	−1
	Interior	2	−3	−4	−7	−6	−6
	South Coast	6	2	0	−1	0	2
	Southeast	1	−6	−8	−11	−7	−5
Colorado	East 105° long	−2	−12	−14	−19	−15	−12
	West 105° long	−8	−18	−25	−30	−24	−16
Connecticut		−1	−7	−16	−17	−16	−9
Delaware		2	−3	−10	−11	−10	−6
Florida	North 29° latitude	7	1	−2	−3	−1	2
	South 29° latitude	14	7	3	3	5	7
Georgia		3	−2	−6	−7	−6	−2
Idaho		−4	−13	−18	−21	−18	−13
Illinois	North 40° latitude	−1	−9	−19	−21	−18	−11
	South 40° latitude	1	−7	−16	−17	−15	−8
Indiana		−1	−7	−16	−18	−16	−9
Iowa		−2	−13	−23	−26	−22	−16
Kansas		−2	−11	−15	−19	−14	−13
Kentucky		1	−6	−13	−14	−11	−6
Louisiana		5	−1	−3	−4	−2	1
Maine		−3	−10	−23	−26	−26	−18
Maryland		2	−3	−10	−12	−10	−4
Massachusetts		−2	−7	−16	−18	−17	−10
Michigan		−2	−11	−20	−23	−23	−18
Minnesota		−4	−18	−30	−34	−31	−24
Mississippi		3	−3	−6	−6	−4	−1
Missouri		1	−7	−14	−16	−13	−8
Montana		−7	−18	−24	−30	−24	−21
Nebraska		−3	−13	−18	−22	−19	−13
Nevada	North 38° latitude	−7	−14	−18	−22	−18	−13
	South 38° latitude	8	0	−3	−4	−2	1
New Hampshire		−3	−8	−18	−21	−21	−12
New Jersey		2	−3	−11	−12	−11	−6
New Mexico	North 34° latitude	−2	−11	−14	−17	−14	−11
	South 34° latitude	4	−4	−8	−11	−7	−3
New York	North 42° latitude	−3	−8	−21	−24	−24	−16
	South 42° latitude	−1	−5	−14	−16	−15	−9
North Carolina		−1	−7	−10	−11	−9	−5
North Dakota		−4	−20	−27	−31	−29	−22
Ohio		−1	−7	−16	−17	−15	−9
Oklahoma		1	−8	−12	−13	−8	−7
Oregon	East 122° long	−6	−11	−14	−19	−14	−9
	West 122° long	0	−4	−5	−7	−4	−3
Pennsylvania	North 41° latitude	−3	−8	−19	−20	−21	−15
	South 41° latitude	0	−6	−13	−14	−14	−8
Rhode Island		1	−3	−12	−13	−13	−7
South Carolina		5	−1	−5	−5	−3	−2
South Dakota		−4	−14	−24	−27	−24	−18
Tennessee		1	−5	−9	−11	−9	−4
Texas	North 31° latitude	3	−6	−9	−13	−9	−7
	South 31° latitude	9	2	−2	−3	−1	2
Utah		−2	−11	−14	−18	−14	−8
Vermont		−3	−8	−20	−23	−24	−15
Virginia		2	−3	−9	−11	−9	−4
Washington	East 122° long	−2	−8	−11	−18	−11	−8
	West 122° long	0	−3	−3	−7	−4	−3
West Virginia		−3	−8	−15	−16	−14	−9
Wisconsin		−3	−14	−24	−28	−24	−18
Wyoming		−4	−15	−18	−26	−19	−16

SUMMARY OF CHANGES

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–06b) that may impact the use of this standard. (Approved Feb. 1, 2007.)

(*1*) Added standards to the Referenced Documents.
(2) Added Section 4.
(*3*) Added X2.2.2.

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–06a) that may impact the use of this standard. (Approved Nov. 1, 2006.)

(*1*) Revised Appendix X4.

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–06) that may impact the use of this standard. (Approved Oct. 1, 2006.)

(*1*) Added Test Method D 6890.
(2) Revised 5.1.10.

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–05) that may impact the use of this standard. (Approved May 15, 2006.)

(*1*) Deleted Test Method D 6920 from this standard.

Subcommittee D02.E0.02 has identified the location of selected changes to this standard since the last issue (D 975–04c$^{\epsilon 1}$) that may impact the use of this standard. (Approved June 1, 2005.)

(*1*) Removed footnote J from Grade No. 4–D in Table 1.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name:	ASTM D1217: Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
CFR Section(s):	40 CFR 75, Appendix D
Standards Body:	American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1217 – 93 (Reapproved 1998)

Standard Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[1]

This standard is issued under the fixed designation D 1217; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the density of pure hydrocarbons or petroleum distillates boiling between 90 and 110°C that can be handled in a normal fashion as a liquid at the specified test temperatures of 20 and 25°C.

1.2 This test method provides a calculation procedure for conversion of density to relative density (specific gravity).

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Note 1, Note 2, and Note 3.

2. Referenced Documents

2.1 *ASTM Standards:*

E 1 Specification for ASTM Thermometers[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *density*—the weight in vacuo, (that is, the mass) of a unit volume of the material at any given temperature.

3.1.2 *relative density (specific gravity)*—the ratio of the mass (weight in vacuo) of a given volume of material at a temperature, t_1, to the mass of an equal volume of water at a reference temperature, t_2; or it is the ratio of the density of the material at t_1 to the density of water at t_2. When the reference temperature is 4.00°C, the temperature at which the relative density of water is unity, relative density (specific gravity) and density are numerically equal.

4. Summary of Test Method

4.1 The liquid sample is introduced into a pycnometer, equilibrated to the desired temperature, and weighed. The relative density (specific gravity) or density is then calculated from this weight and the previously determined weight of water that is required to fill the pycnometer at the same temperature, both weights being corrected for the buoyancy of air.

5. Significance and Use

5.1 Density is a fundamental physical property which can be used in conjunction with other properties to characterize pure hydrocarbons and their mixtures.

5.2 This test method was originally developed for the determination of the density of the ASTM Knock Test Reference Fuels *n*-heptane and *iso*octane, with an accuracy of 0.00003 g/mL. Although it is no longer employed extensively for this purpose, this test method is useful whenever accurate densities of pure hydrocarbons or petroleum fractions with boiling points between 90 and 110°C are required.

6. Apparatus

6.1 *Pycnometer*, Bingham-type,[3] conforming to the dimensions given in Fig. 1, constructed of borosilicate glass and having a total weight not exceeding 30 g.

6.2 *Constant-Temperature Bath*, provided with suitable pycnometer holders or clips and means for maintaining temperatures constant to ±0.01°C in the desired range.

6.3 *Bath Thermometer*, graduated in 0.1°C subdivisions and standardized for the ice point and the range of use to the nearest 0.01°C. ASTM Saybolt Viscosity Thermometer 17C as prescribed in Specification E 1, designed for tests at 21.1°C and 25°C, is recommended. A standardized platinum resistance thermometer may also be used, and offers the best means for observing minute temperature changes in the bath. Whichever means are available, it must be realized that for most hydrocarbons the density coefficient is about 0.0008 units/°C, and therefore an error of ±0.013°C would cause an error of ±0.00001 in density.

6.4 *Hypodermic Syringe*, 30-mL capacity, of chemically resistant glass, equipped with a 152-mm (6-in.) needle made of stainless steel tubing as shown in Fig. 2.

6.5 *Draw-Off Needle*, made of stainless steel tubing as shown in Fig. 2.

---

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.04on Hydrocarbon Analysis.

Current edition approved Feb. 15, 1993. Published May 1993. Originally published as D 1217 – 52 T. Last previous edition D 1217 – 91.

[2] *Annual Book of ASTM Standards*, Vol 14.03.

[3] Pycnometer available from Reliance Glass Co., 220 Gateway Rd., Bensenville, IL 60106-0825, has been found satisfactory.

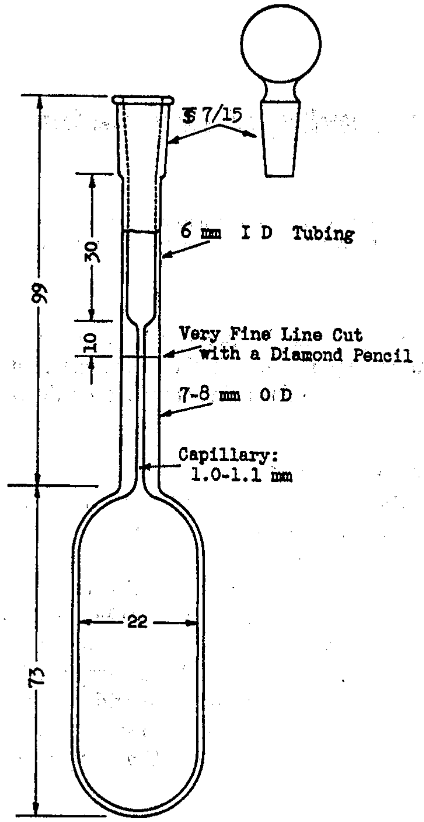


FIG. 1 Bingham-Type Pycnometer, 25 mL

6.6 *Solvent-Cleaning Assembly*, as shown in Fig. 3.

6.7 *Chromic Acid Cleaning Apparatus*, similar to that shown in Fig. 4.

6.8 *Balance*, capable of reproducing weighings within 0.1 mg. Mechanical balances should have sensitivity which causes the pointer to be deflected 2 or 3 scale divisions per 1 mg when carrying a load of 30 g or less on each pan. The balance should be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled pycnometer) do not cause a significant change in the ratio of the balance arms. Otherwise weighings shall be made by the method of substitution, in which the calibrated weights and pycnometer are alternately weighed on the same balance pan. The same balance shall be used for all related weighings.

6.9 *Weights*, whose relative values are known to the nearest 0.05 mg or better. The same set of weights shall be used for the calibration of the pycnometer and the determination of densities.

7. Reagents and Materials

7.1 *Acetone*—(**Warning**—See Note 1).

NOTE 1—**Warning:** Extremely flammable. Use adequate ventilation.

7.2 *Isopentane*—(**Warning**—See Note 2).

NOTE 2—**Warning:** Extremely flammable. Avoid buildup of vapors and remove all sources of ignition, especially non-explosion proof electrical apparatus.

7.3 *Chromic Acid (Potassium Dichromate/Conc. Sulfuric Acid)*—(**Warning**—See Note 3).

NOTE 3—**Warning:** Causes severe burns. A recognized carcinogen. Do not get in eyes, or on skin or clothing.

8. Preparation of Apparatus

8.1 Thoroughly clean the pycnometer with hot chromic acid cleaning solution by means of the assembly shown in Fig. 4 (**Warning**—See Note 3). Chromic acid solution is the most effective cleaning agent. However, surfactant cleaning fluids have also been used successfully. Mount the apparatus firmly and connect the trap to the vacuum. Warm the necessary amount of cleaning acid in the beaker, place the pycnometer on the ground joint, and evacuate by opening the stopcock to vacuum. Fill the pycnometer with acid by turning the stopcock, repeat several times or remove the filled pycnometer, and allow it to stand for several hours at 50 to 60°C. Remove the acid from the pycnometer by evacuation, empty the acid from the trap, and flush the pycnometer with water. Cleaning should be made in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pycnometer may be cleaned between determinations by washing with a suitable solvent, rinsing with pure, dry acetone, followed by isopentane, and vacuum drying.

8.2 Transfer the pycnometer to the cleaner assembly shown in Fig. 3, with vacuum line and trap attached to the side tube as indicated. Place the pycnometer on the cleaner with the upper hypodermic needle extending upward into the pycnometer, and press the edge of the ground joint on the rubber stopper until the vacuum holds it in place. Draw out all the liquid or sample. Immerse the lower end of the hypodermic tube in a suitable solvent and draw 20 to 25 mL through the pycnometer. Leaving the pycnometer in place, draw air through it until it is dry. Clean the hypodermic syringe with the same apparatus.

9. Calibration of Pycnometer

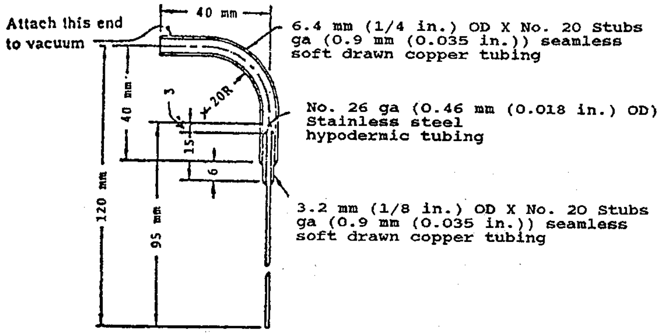
9.1 Proceeding as directed in Section 10, determine the weight of freshly-boiled and cooled distilled water (distilled from alkaline permanganate through a tin condenser) held by the pycnometer when equilibrated to volume at the bath temperature to be used in the determination. Repeat until at least three values agree to ±0.2 mg.

10. Procedure

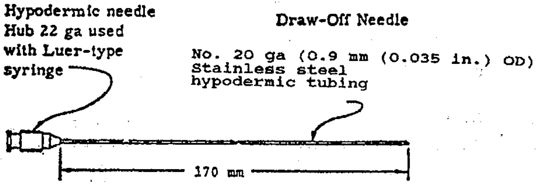
10.1 Using another 25-mL pycnometer as a tare (Note 4), weigh the clean, dry pycnometer to 0.1 mg and record the weight.

NOTE 4—It is convenient to use the lightest of a set of pycnometers as a tare. For best results the treatment and environment of both pycnometer and tare should be identical for some time prior to weighing.

10.2 Cool the sample to 5 to 10°C below the test temperature, and fill the clean 30-mL hypodermic syringe. Transfer the sample to the pycnometer through the filling needle; avoid trapping air bubbles (Note 2) in the bulb or capillary of the pycnometer. If any are present, draw them into the syringe where possible. Also remove with the syringe or draw-off



Draw-Off Needle



Filling Needle
To be used with a 30 ml Yale B-D Lok-Syringe
Becton-Dickinson and Co., Rutherford, N. J.

FIG. 2 Accessories for Bingham-Type Pycnometer

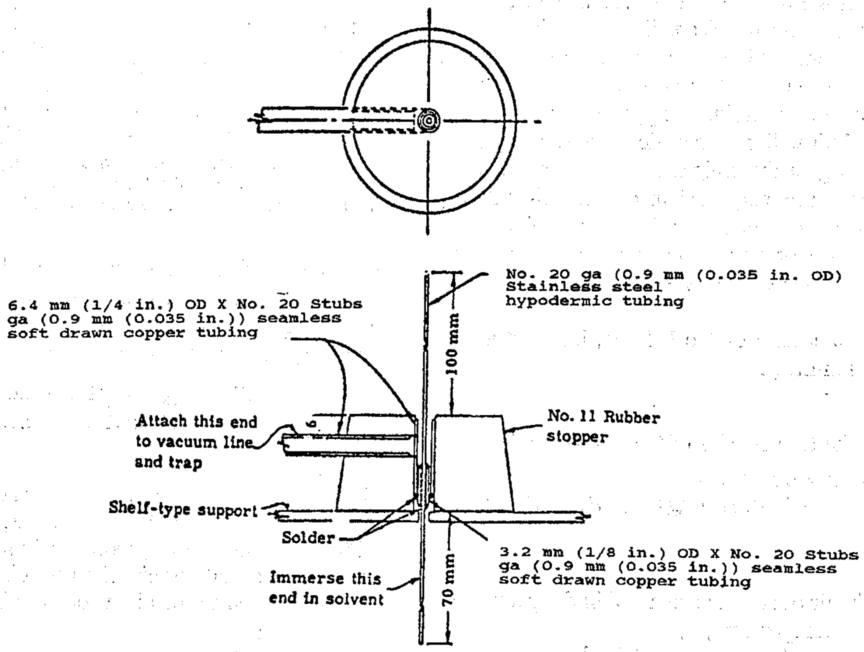


FIG. 3 Cleaner Assembly for Bingham-Type Pycnometer

needle any liquid above the calibration mark in the capillary or overflow reservoir. Dry the remainder with a cotton fiber pipe cleaner or cotton swab which has been dampened slightly with acetone.

Note 5—For work of highest accuracy on pure compounds, dissolved air may be removed from the sample by repeated freezing and remelting of the sample under vacuum in the pycnometer.

10.3 Close the pycnometer with the glass stopper and immerse it to a point above the calibration mark in the constant-temperature bath adjusted to a constancy of ±0.01°C at the desired temperature. Periodically, or before the liquid expands into the overflow chamber, remove the stopper, raise the pycnometer sufficiently to expose the calibration mark to view, and readjust the liquid level to the mark by withdrawing liquid through the steel draw-off needle until expansion has stopped, indicating that the liquid has reached the temperature of the thermostat. Do not allow the liquid to expand more than 10 mm above the calibration mark at any time, to minimize errors caused by faulty drainage. Allow the contents to equilibrate an additional 10 min and draw the level down exactly to

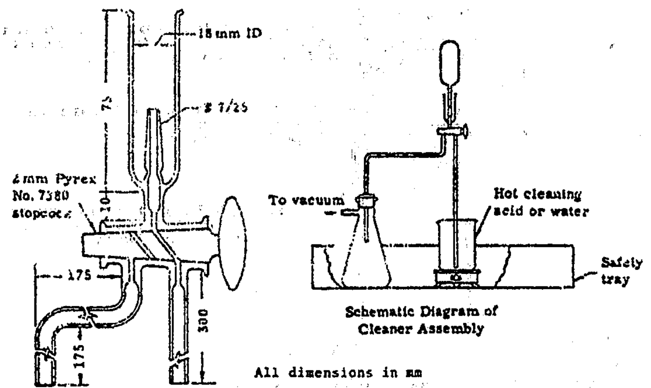


FIG. 4 All-Glass Pycnometer Cleaner Assembly for Use with Hot Chromic Acid Cleaning Solution

the calibration line, avoiding parallax and using a magnifier, if necessary, to obtain good visibility. Remove any liquid adhering to the walls above the calibration mark, with the draw-off needle or pipe cleaner, depending upon the volatility of the sample. Portions in the overflow bulb may be removed with a cotton swab moistened with acetone.

10.4 Replace the glass stopper, remove the pycnometer from the bath, wash the outside surface with acetone, and dry thoroughly with a chemically clean, lint-free, slightly damp cloth. Place the pycnometer in or near the balance case for 20 min and weigh to the nearest 0.1 mg. In atmospheres of low humidity (60 % or lower), drying the pycnometer by rubbing with a dry cotton cloth will induce static charges equivalent to a loss of about 1 mg in the weight of the pycnometer. This charge need not be completely dissipated in less than 30 min. The use of about 0.1-mg radium bromide- or polonium-coated foil in the balance case, or maintaining the relative humidity at 60 % or higher, aids in reducing weighing difficulties due to static charges.

10.5 Record temperature of the balance, barometric pressure, and relative humidity.

11. Calculation

11.1 Calculate the true density of the sample as follows:

$$\text{Density, g/mL at °C} = W_s(1 + (d_a/d_s) - (d_a/d_{wt}))d_w/W_w(1 + (d_a/d_w) - (d_a/d_{wt})) \tag{1}$$

where:

W_s = weight in air of sample contained in the pycnometer at the test temperature, g,

W_w = weight in air of the water contained in the pycnometer at the calibration temperature, g,

d_w = density of water at the calibration temperature, as obtained from Table 1,

d_a = density of air in balance case at the time of weighing, as calculated from 10.3,

d_{wt} = density of weights used in weighing the sample and water (brass = 10.4 g/mL, stainless steel = 7.75 g/mL), and

d_s = approximate density of sample or,

$$(W_s \times d)/W_w \tag{2}$$

11.2 The equation assumes that the weighings of the pycnometer empty and filled are made in such a short time interval that the air density has not changed. If significant change should occur, the calculated apparent weight of the sample, W_s, in this equation, must be corrected for the difference in air buoyancy exerted on the pycnometer as follows:

$$W_s = W^2{}_{PS} - W'_p(1 + (d'_a/2.2) - (d'_a/d_{wt}))/(1 + (d_a{}^2/2.2) - (d_a{}^2/d_{wt})) \tag{3}$$

where:

$W^2{}_{PS}$ = weight of pycnometer and contained sample under second or final air density,

W'_P = weight of pycnometer in air of first density,

d'_a = density of air when weighing empty pycnometer,

$d_a{}^2$ = density of air when weighing filled pycnometer, and

d_{wt} and 2.2 = density of weights and borosilicate glass, respectively.

Likewise, if the pycnometer, empty and filled with water for calibration, is weighed under different air densities a similar correction for different air buoyancies shall be applied.

11.3 Calculate the relative density (specific gravity) of the sample by dividing the density as obtained in 11.1 by the relative density of water at the reference temperature obtained from Table 1.

11.4 Calculate the density of air in the balance room as follows:

Air density (d_a), g/mL

TABLE 1 Density of Water[A]

Temperature,° C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0	0.999840	21	0.997991	40	0.992212
3	0.999964	22	0.997769	45	0.990208
4	0.999972	23	0.997537	50	0.988030
5	0.999964	24	0.997295	55	0.985688
10	0.999699	25	0.997043	60	0.983191
15	0.999099	26	0.996782	65	0.980546
15.56	0.999012	27	0.996511	70	0.977759
16	0.998943	28	0.996231	75	0.974837
17	0.998774	29	0.995943	80	0.971785
18	0.998595	30	0.995645	85	0.968606
19	0.998404	35	0.994029	90	0.965305
20	0.998203	37.78	0.993042	100	0.958345

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Appendix G, *Standard Methods for Analysis of Petroleum and Related Products 1991*, Institute of Petroleum, London.

$$= [(B - 0.3783\,Hp)(0.000465)]/(273 + t) \tag{4}$$

where:

B = barometric pressure, mm Hg, corrected to 0°C,
H = relative humidity, decimal fraction,
p = vapor pressure of water at temperature t, mm Hg, and
t = room temperature, °C.

NOTE 6—If this test method is to be used frequently, a considerable amount of calculation can be avoided by use of a gas density balance to determine the air density. Weigh a sealed 250-mL glass bulb at several different air densities and plot the weight against the air density. To determine the air density at some later time, weigh the bulb and read the air density from the point on the curve corresponding to the weight.

11.5 To calculate the density or relative density (specific gravity) at any test temperature, t, other than the calibration temperature, t_c(to correct for the cubical coefficient of thermal expansion of borosilicate glass), divide the value obtained in 10.1 or 10.2 by the following expression:

$$1 + 9.6 \times 10^{-6}\,(t - t_c) \tag{5}$$

12. Report

12.1 In reporting density, give the test temperature and the units (for example, density, 20°C = x.xxxxx g/mL). In reporting relative density (specific gravity), give both the test temperature and the reference temperature, but no units (for example, relative density (specific gravity), 20/4°C = x.xxxxx). Carry all calculations to one digit beyond the last significant figure, but report the final result to the fifth decimal place (0.00001).

13. Precision and Bias

13.1 *Precision*—Results, using the 25-mL Bingham-type pycnometer, should not differ from the mean by more than the following amounts:

Repeatability One Operator and Apparatus	**Reproducibility** Different Operators and Apparatus
0.00002	0.00003

NOTE 7—The precision for this method was not obtained in accordance with RR:D02-1007.

13.2 *Bias*—The difference of results from the established values when compared to pure reference materials is not expected to be more than ±0.00003 g/mL. Specific bias has not been established by cooperative testing.

14. Keywords

14.1 Density; pycnometer; relative density; specific gravity

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM D1246: Bromide - Titrimetric

CFR Section(s): 40 CFR 136.3(a) Table IB

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1246 – 95 (Reapproved 1999)

Standard Test Method for Bromide Ion in Water[1]

This standard is issued under the fixed designation D 1246; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method is applicable to the measurement of bromide ion in water, ground water, and drinking water.

1.2 Samples containing 0.5 mg/L to 1000 mg/L of bromide may be analyzed by this test method. The concentration range may be extended by the dilution of an appropriate aliquot.

1.3 The precision and bias statements were determined on natural and ground waters. It is the responsibility of the analyst to assure the validity of this test method for untested matrices.

1.4 A titrimetric and two colorimetric test methods for iodide and bromide were discontinued. Refer to Appendix X1 for historical information.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1066 Practice for Sampling Steam[2]

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[2]

D 3370 Practices for Sampling Water from Closed Conduits[2]

D 4127 Terminology Used with Ion-Selective Electrodes[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminologies D 1129 and D 4127.

4. Summary of Test Method

4.1 Bromide ion is measured potentiometrically using a bromide ion-selective electrode in conjunction with a single-junction, sleeve-type reference electrode. Potentials are read on a pH meter having an expanded millivolt scale capable of being read to the nearest 0.1 mV, or a selective ion meter having a direct concentration scale for bromide. For less precise work, a pH meter having a millivolt scale capable of being read to the nearest 1.0 mV is adequate, however, no supporting data are available.

4.2 The electrodes are calibrated in known bromide solutions and the concentrations of unknowns are determined in solutions with the same background. In most cases, addition of an ionic strength adjustor to both standards and samples is sufficient to maintain constant background ionic strength. For samples above 0.1 *M* in ionic strength, prepare standard solutions similar to the sample composition.

5. Significance and Use

5.1 By analysis for bromide in water, wastewater, and brackish waters, it is possible to evaluate the origin of the water, its potential as a source of bromide, and its condition with regard to pollution.

6. Interferences

6.1 Strongly reducing solutions and solutions containing ions which form insoluble silver salts may coat the electrode membrane. These may be removed by polishing the membrane surface. Sulfide ion and cyanide ion both poison the electrode, and should be removed (see Section 11).

6.2 Halide ions form complexes with some metals. Since the electrode responds only to free bromide ions, the presence of any complexing agents lowers the measured concentrations. Concentrations of free metal ions causing a 10 % error of a bromide concentration of 8.1 mg/L are bismuth $^{+3}$, 80 mg/L; cadmium $^{+2}$, 100 mg/L; lead $^{+2}$, 1600 mg/L; tin $^{+2}$, 2400 mg/L; and thallium $^{+3}$, 4 mg/L.

6.3 Chloride ion and hydroxide ion do not interfere when present in the concentrations of up to 400 and 30 000 times the bromide concentration, respectively. There will be no interference from ammonia when present in concentrations twice that of bromide, nor from thiosulfate 20 times as concentrated as bromide. Iodide is an interference at a concentration ratio as low as 2×10^{-4}. Mercury should be absent from samples.

7. Apparatus

7.1 *pH Meter*, capable of reading to 0.1 mV, or a selective-ion meter.

7.2 *Bromide Ion-Selective Electrode.*

---

[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved Dec. 10, 1995. Published February 1996. Originally published as D 1246 – 52. Last previous edition D 1246 – 88 (1995) $^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

7.3 *Sleeve-Type Single-Junction Reference Electrode*, filled with manufacturer's filling solution.

7.4 *Mixer*, magnetic, with a TFE fluorocarbon-coated stirring bar.

7.5 *Laboratory Glassware.*

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. [3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*— Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193, Type I. Other reagent water types may be used, provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the bias and precision of the test method. Type II water was specified at the time of round robin testing of this test method.

8.3 *Ionic Strength Adjustor* (42.5 g/100 mL $NaNO_3$)—In a 100-mL volumetric flask, dissolve 42.5 g of sodium nitrate ($NaNO_3$) in water and dilute to volume.

8.4 *Nickel Sulfate Solution, 1 M*—In a 100 mL volumetric flask dissolve 26.3 g of nickel sulfate hexahydrate ($NiSO_4 \cdot 6H_2O$) in water and dilute to volume.

8.5 *Sodium Bromide Standard Solution, 1000 mg/L*—In a 1 L volumetric flask dissolve 1.288 g dried sodium bromide in water and dilute to volume.

8.6 *Sodium Bromide Standard Solutions*, (100, 10, and 1 mg/L)—Dilute 1 volume of the 1000 mg/L bromide standard with 9 volumes of water to prepare the 100 mg/L standard. By further 1+9 serial dilutions, prepare the 10 and 1 mg/L standards.

9. Sampling

9.1 Collect the samples in accordance with Practice D 1066 and Practices D 3370, as applicable.

10. Calibration and Standardization

10.1 To 100 mL of the 1, 10, 100, and 1000 mg/L standards add 2 mL of the ionic strength adjustor (ISA). If any samples require treatment for interferences, prepare standards with the same background.

10.2 Connect and fill electrodes in accordance with manufacturer's instructions.

10.3 Transfer the 1 mg/L standard-ISA mixture to a 150 mL beaker and stir gently using the magnetic mixer. Immerse the electrodes in the solution and wait 2 min for the potential to stabilize. Record the value.

10.4 Rinse electrodes thoroughly and repeat for the 10, 100, and 1000 mg/L standard-ISA mixtures. Wait 2 min and record the potential.

10.5 The calibration curve is generated by plotting on semilogarithmic graph paper, the potential observed versus the concentration of the standard used. Note that volume corrections are incorporated into the calibration, so that samples analyzed according to Section 11 of this test method can be read directly.

11. Procedure

11.1 To any sample containing sulfide or cyanide ion, add 0.1 mL nickel sulfate solution to 100 mL sample.

NOTE 1—This concentration of nickel sulfate will react with 58 mg/L sulfide and 117 mg/L cyanide.

11.2 Transfer 100 mL sample to a 150 mL beaker and add 2 mL ionic strength adjustor. Stir thoroughly for 1 min using the magnetic mixer.

11.3 Immerse the electrodes in the sample-ISA mixture and wait 2 min for the potential to stabilize. Record the value.

11.4 Read bromide concentration of the sample, in mg/L, directly from the calibration curve. Note that volume corrections are incorporated into the calibration.

12. Precision and Bias [4]

12.1 *Precision*—The overall and single-operator precision of this test method may be expressed as follows:

12.1.1 *For Reagent Water:*

$$S_T = 0.077X + 1.10, \text{ coefficient of correlation} = 1.0$$

$$S_O = 0.067X + 0.79, \text{ coefficient of correlation} = 1.0$$

12.1.2 *For Water Matrices:*

$$S_T = 0.064X + 0.84, \text{ coefficient of correlation} = 1.0$$

$$S_O = 0.049X + 0.09, \text{ coefficient of correlation} = 1.0$$

where:

S_T = overall precision, mg/L,
S_O = single-operator precision, mg/L, and
X = concentration of bromide determined.

12.2 The selected water matrices included natural waters, ground waters, and tap water. These data on precision and bias may not apply to waters of other matrices.

12.3 *Bias*—Recoveries of known amounts of bromide from reagent water and selected water matrices were as shown in Table 1.

12.4 This information is derived from round-robin testing, in which five laboratories, including eight operators, participated. Of the eight sets of data ranked, as described in Practice D 2777, none was rejected. One operator submitted reagent water data only. One outlier data point within each set was also rejected. Four sample levels were run on three days and blanks were obtained for the water used.

12.5 This section on precision and bias conforms to Practice D 2777 – 77, which was in place at the time of collaborative testing. Under the allowances made in 1.5 of Practice

---

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[4] Supporting data for this test method have been filed at ASTM Headquarters. Request Research Report RR:D19-1078.

TABLE 1 Recoveries of Known Amounts of Bromide from Reagent Water and Selected Water Matrices

	Amount added, mg/L	Amount found, mg/L	±Bias, %	Statistically Significant (95 % Confidence Level)
Reagent Water	0.65	0.77	+ 18.5	yes
	1.00	1.19	+ 19.0	no
	92.7	96.4	+ 3.99	no
	864	854	− 1.16	no
Water	0.65	0.80	+ 23.1	yes
	1.00	1.21	+ 21.5	yes
	92.7	95.6	+ 3.2	no
	864	836	− 3.3	yes

D 2777 – 86, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D–19 test methods.

13. Keywords

13.1 bromide; ISE; ion-selective electrode

APPENDIX

(Nonmandatory Information)

X1. RATIONALE FOR DISCONTINUATION OF TEST METHODS

X1.1 *Colorimetric and Titrimetric Test Methods for Iodide and Bromide*

X1.1.1 These test methods were discontinued in 1988, and the title of the standard was changed because no iodide test methods remained in the standard. These test methods may be found in their entirety in the *1988 Annual Book of ASTM Standards*, Vol 11.01. Additional relevant data may be found in Research Report RR:D-19-57, which is filed at ASTM Headquarters.

X1.1.2 *Former Test Method A*—Colorimetric for Iodide

X1.1.2.1 This test method covers the colorimetric determination of iodide in water when concentrations up to 100 µg/L are present.

X1.1.2.2 This test method is based on the catalytic effect of traces of iodide on the rate of oxidation of arsenious ion by ceric ion in acid solution. In the presence of a small amount of iodide, the reaction follows first-order reaction rate kinetics, and, at any given temperature and reaction time, the extent of reduction of ceric ion is directly proportional to iodide concentration. Since solutions of ceric ions are yellow and those of cerous ions colorless, the extent of the reaction may be determined photometrically.

X1.1.3 *Former Test Method B*—Colorimetric for Bromide

X1.1.3.1 This test method covers colorimetric determination of bromide in water when concentrations equivalent to no more than 100 µg/L are present.

X1.1.3.2 This test method is based on the catalytic effect of traces of bromide ion on the rate of oxidation of iodine to iodate by potassium permanganate in sulfuric acid solution. Under controlled conditions of pH, temperature, and concentration of reactants, and for a given reaction time, the concentration of unreacted iodine is inversely proportional to the concentration of bromide. To determine bromide, the reaction is stopped after a given time by extraction of the unreacted iodine with carbon tetrachloride and measuring the color intensity of the exact solution at 515 nm.

X1.1.4 *Former Test Method C*—Titrimetric, for Iodide and Bromide

X1.1.4.1 This test method is recommended for samples containing appreciable amounts of iodide or bromide, or both, at concentrations greater than 5 mg/L.

X1.1.4.2 Any iodide in the sample is oxidized with bromine to the iodate in a buffered solution, the excess bromine being destroyed subsequently. Iodine equivalent to the iodate is liberated from potassium iodide and titrated with sodium thiosulfate.

X1.1.4.3 Iodide and bromide occurring together are oxidized to iodate and bromate, respectively, with hypochlorite. Iodine equivalent to the combined reaction products is liberated and measured after destroying the excess hypochlorite.

X1.1.4.4 The bromide content of the sample is calculated by difference between the iodide and combined iodide and bromide determinations.

X1.1.5 These test methods were discontinued because there were insufficient laboratories interested in participating in a collaborative study to obtain the necessary precision and bias data as required by Practice D 2777.

ASTM Logo Removed

[redacted] **D 1246**

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1253: Standard Test Method for Residual Chlorine in Water

CFR Section(s): 21 CFR 165.110(b)(4)(iii)(I)(5)(i)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1253 – 86 (Reapproved 1996)

Standard Test Method for Residual Chlorine in Water[1]

This standard is issued under the fixed designation D 1253; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of residual chlorine in water by direct amperometric titration.

1.2 Within the constraints specified in Section 6, this test method is not subject to commonly encountered interferences and is applicable to most waters. Some waters, however, can exert an iodine demand, usually because of organic material, making less iodine available for measurement by this test method. Thus, it is possible to obtain falsely low chlorine readings, even though the test method is working properly, without the user's knowledge.

1.3 Precision data for this test method were obtained on estuary, inland main stem river, fresh lake, open ocean, and fresh cooling tower blowdown water. Bias data could not be determined because of the instability of solutions of chlorine in water. It is the user's responsibility to ensure the validity of the test method for untested types of water.

1.4 In the testing by which this standard was validated, the direct and back starch-iodide titrations and the amperometric back titration, formerly part of this standard, were found to be unworkable and were discontinued in 1986. Historical information is presented in Appendix X1.

NOTE 1—Orthotolidine test methods have been omitted because of poor precision and accuracy.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]
D 1193 Specification for Reagent Water[2]
D 3370 Practices for Sampling Water from Closed Conduits[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *combined residual chlorine, n*—residual consisting of chlorine combined with ammonia nitrogen or nitrogenous compounds.

3.2.2 *free available chlorine residual, n*—residual consisting of hypochlorite ions, hypochlorous acid, or a combination thereof.

3.2.3 *total residual chlorine (chlorine residual), n*—the amount of available chlorine-induced oxidants present in water at any specified period, subsequent to the addition of chlorine.

NOTE 2—Chlorine present as chloride is neither included in these terms nor determined by this test method.

NOTE 3—Bromine, bromine combined with ammonia or nitrogenous compounds, and chlorine dioxide are not distinguished by this test method from the corresponding chlorine compounds.

4. Summary of Test Method

4.1 This is an amperometric titration test method utilizing phenylarsine oxide as the titrant. When the titrator cell is immersed in a sample containing chlorine, current is generated. As phenylarsine oxide is added, the chlorine is reduced and the generation of current ceases. When chlorine is present as a chloramine, potassium iodide is added, releasing iodine, which is titrated in a similar manner. The iodine content is calculated in terms of free chlorine.

5. Significance and Use

5.1 Chlorine is used to destroy or deactivate a variety of unwanted chemicals and microorganisms in water and wastewater.

5.2 An uncontrolled excess of chlorine in water, whether free available or combined, can adversely affect the subsequent use of the water.

6. Interferences

6.1 This test method is not subject to interferences from temperature, color, or turbidity of sample.

6.2 Values of pH above 8.0 interfere by slowing the reaction rate. Buffering the sample to pH 7.0 or less eliminates the interference.

6.3 Erratic behavior of the apparatus in the presence of cupric ions has been reported.

6.4 Cuprous and silver ions tend to poison the electrode of the titrator.

6.5 Nitrogen trichloride and some N-chloro compounds are often present as products of the chlorination of wastewaters and will titrate partially as free available chlorine and partially as combined residual chlorine. This error can be avoided only in the determination of total residual chlorine.

---

[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water, and is the direct responsibility of Subcommittee D 19.05 on Inorganic Constituents in Water.

Current edition approved Feb. 28, 1986. Published April 1986. Originally published as D 1253 – 53. Last previous edition D 1253 – 76.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

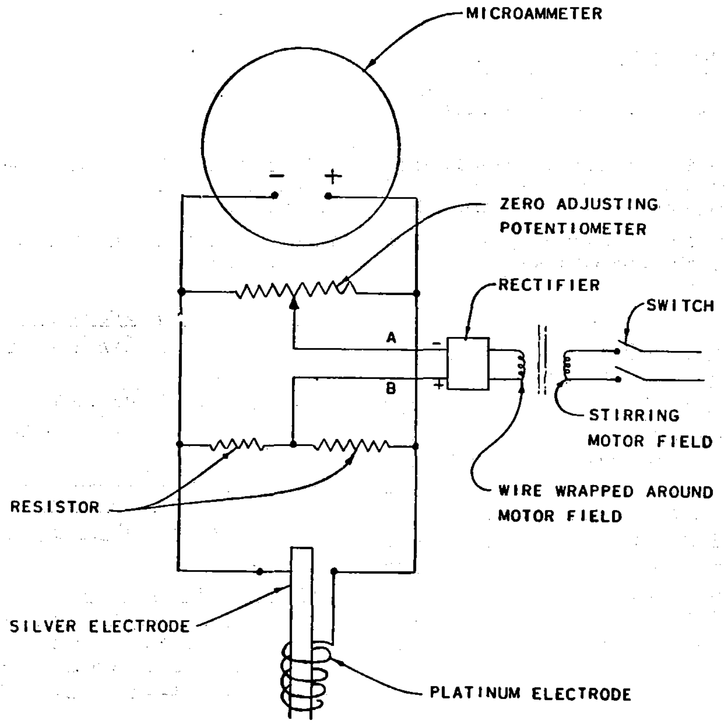


FIG. 1 Wiring Diagram of Amperometric Titrator

6.6 Exposure to high concentrations of free available chlorine causes a film-type polarization that reverses very slowly. This can be avoided by diluting the sample with water to less than 10 mg/L of free available chlorine.

6.7 If chlorine dioxide is present, an unknown portion titrates as free available chlorine. Total chlorine dioxide titrates as total residual chlorine.

6.8 Depending upon final pH, chlorination of waters containing ammonia or nitrogenous organic compounds can produce high concentrations of dichloramine. This compound produces four to five times as much current as monochloramine. The current produced by as little as 5 mg/L of dichloramine can cause the microammeter pointer to read offscale even at the end point in the titration of free available chlorine. This may be overcome by use of an opposing voltage in the apparatus' circuitry. The instrument's manufacturer should be consulted in this regard.

7. Apparatus

7.1 *Amperometric Titration Apparatus*[3,4]—Refer to Fig. 1.

NOTE 4—When the titrator has been out of service for a day or more, check the electrode for sensitivity by noting the rapidity of the pointer deflection. If the pointer responds slowly after the addition of KI solution, add a small amount of biiodate. If it responds slowly to free available chlorine, sensitize it by adding chlorine.

7.2 *Glassware*—Condition with water containing at least 10 mg/L of residual chlorine for at least 2 h prior to use and then rinse thoroughly.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type III, further treated to be free of chlorine demand. A suggested method for preparation of chlorine demand-free water is to add approximately 20 mg/L of available chlorine to Type III water, let it stand for about a week in darkness, and then expose it to sunlight until no chlorine remains.

8.3 *pH 4.0 Buffer Solution*—Dissolve 243 g of sodium acetate trihydrate and 480 g of glacial acetic acid in water and dilute to 1 L.

---

[3] *Water and Sewage Works*, May 1949, p. 171, and *Journal American Water Works Association*, Vol 34, 1942, pp. 1227–1240.

[4] Amperometric titrators are available commercially from most laboratory supply houses.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.4 *pH 7.0 Buffer Solution*—Dissolve 25.4 g of monobasic potassium phosphate and 86 g of dibasic sodium phosphate in water and dilute to 1 L.

8.5 *Biiodate, Solution Standard* (0.0282*N*)—Dissolve 0.9163 g of potassium biiodate in water and dilute to 1 L in a volumetric flask. Store in an amber glass-stoppered bottle.

8.6 *Phenylarsine Oxide, Solution Standard* (0.00564 *N*)—Dissolve 0.8 g of phenylarsine oxide in 150 mL of sodium hydroxide solution (12 g/L). After settling, decant 110 mL of this solution, add 800 mL of water, and bring to a pH of 9.0 by adding hydrochloric acid (1 + 1). This should require about 2 mL of HCl (1 + 1). Continue acidification with HCl (1 + 1) until a pH of 6 to 7 is reached, as indicated by a glass-electrode system; then dilute to a total volume of 1L. Standardize to 0.00564 *N* against 0.0282 *N* biiodate solution using the titrator (7.1) as the end-point indicator. Add 1 mL of chloroform for preservation.

8.7 *Potassium Iodide Solution* (50 g/L)—Dissolve 50 g of KI in water and dilute to 1 L. Add 1 g of sodium bicarbonate to stabilize the solution. Store in an amber bottle and avoid direct exposure to sunlight.

9. Sampling

9.1 Collect the sample in accordance with Practices D 3370. Take care that the sample is representative and keep it away from direct sunlight prior to analysis.

9.2 All tests should be made as soon as possible after collection of the sample (not more than 5 min) because the residual chlorine may diminish with time, due to the chlorine demand of the sample. Where time of contact is important, the elapsed time between the addition of chlorine and the determination of chlorine should be taken into account.

10. Procedure

10.1 For residual chlorine concentrations of 2.0 mg/L or less, use a 200-mL sample. For greater concentrations, use a 100-mL sample. It is preferable that the size of the sample be such that not more than 2 mL of titrant will be required to complete the titration.

10.2 *Determination of Total Residual Chlorine:*

10.2.1 Add 1 mL of KI solution to a 200-mL sample and immediately add 1 mL of pH 4.0 buffer solution.

10.2.2 Immerse the electrodes in the sample and start the stirrer. Adjust the microammeter pointer of the potentiometer to the right or high current side of the scale so the pointer can deflect counterclockwise during the analysis.

10.2.3 Titrate using standard phenylarsine oxide solution, adding the titrant in small increments, and noting the deflection of the microammeter pointer. Plot the progress of the titration on linear graph paper with current on the vertical axis and titrant volume on the horizontal axis. Add a small volume of titrant, wait a few seconds, and plot the current-volume point on the graph.

10.2.4 Readjust the potentiometer several times during the titration, if necessary, to bring the pointer back on scale.

10.2.5 Continue the analysis by determining at least three points spread over the downward sloping titration curve and at least three points after the equivalence or end point. The latter points will indicate practically no change in current. Points just before the end point shall be disregarded in its determination. The millilitres of titrant at the end point defined by the intersection of the two linear sections of the titration curve should be recorded.

10.3 *Determination of Free Available Chlorine Residual:*

10.3.1 Add 1 mL of pH 7.0 buffer solution to a 200-mL sample.

10.3.2 Repeat the phenylarsine oxide titration beginning with 10.2.2.

10.3.3 Note a rapid deflection of the pointer for each increment of titrant indicates the presence of free available chlorine. Slight counterclockwise movements of the pointer after addition of individual drops of titrant is a drift effect and does not indicate the presence of free available chlorine.

10.4 *Determination of Combined Available Chlorine Residual:*

10.4.1 Complete the titration for the determination of free available chlorine residual as in 10.3.

10.4.2 To the same sample, add 1 mL of KI solution and 1 mL of pH 4.0 buffer solution and repeat the titration as in 10.2.

11. Calculation

11.1 Calculate the various types of chlorine residual, in milligrams per litre, as follows:

$$\text{Chlorine residual, mg/L} = 200\,A/V$$

where:

A = phenylarsine oxide solution (0.00564 *N*) required for the titration of 10.2, 10.3, or 10.4, depending on the specific type of chlorine residual determined, mL, and
V = sample used, mL.

12. Precision and Bias[6]

12.1 The overall precision (S_t) and the single operator precision (S_o) of this test method for free available chlorine (FAC) and for total residual chlorine (TRC) were determined by eight or nine qualified cooperators each with analysis equipment and reagents at each of five sites. Each site constituted a different chlorinated cooling water matrix: estuary, inland main stem river, fresh lake, open ocean, and fresh cooling tower blowdown. Each site water was chlorinated up to nine levels. Samples were collected simultaneously and analyzed within 5 min of collection by all eight or nine cooperators. Duplicate sampling and analysis runs were made at each level.

12.2 The S_t and S_o for FAC was found to vary linearly with the mean concentration of FAC, X, in mg/L, over the range for X from 0.0 to 1.0.

12.2.1 For the pooled results from all of the matrices tested:

$$S_t = 0.025 + 0.199\,X\ (n = 37,\ r = 0.848)$$
$$S_o = 0.008 + 0.081\,X\ (n = 35,\ r = 0.638)$$

where:

n = number of runs, and
r = correlation coefficients.

12.3 The S_t and S_o for TRC was found to vary linearly

---

[6] Supporting data are available from ASTM Headquarters. Request RR: D19-1124.

with the mean concentration of TRC, Y, in mg/L, over the range for Y from 0.0 to 3.5.

12.3.1 For the pooled results from all of the matrices tested:

$$S_t = 0.022 + 0.098\,Y\ (n = 39,\ r = 0.865)$$
$$S_o = 0.012 + 0.024\,Y\ (n = 38,\ r = 0.695)$$

12.4 The bias of the test method could not be determined since the instability of solutions of chlorine in water does not permit the determination of an acceptable true value for TRC and FAC in the samples.

13. Keywords

13.1 amperometric; analysis; chlorine; water

APPENDIX

(Nonmandatory Information)

X1. RATIONALE FOR DISCONTINUATION OF TEST METHODS

X1.1 *Direct and Back Starch-Iodide Titrations and Amperometric Back Titration:*

X1.1.1 These two test methods were discontinued in 1986. These test methods may be found in *1985 Annual Book of ASTM Standards*, Vol 11.01. These test methods were originally issued in 1953.

X1.1.2 These test methods are biiodate solutions as titrating agents. Attempts to include these test methods in the round-robin testing were not successful because the reaction rate of the biiodate solution with phenylarsine oxide was slow and inconsistent. The little data obtained were widely varied, nonreproducible, and were not relatable to the values being tested.

X1.1.3 Field experience indicates that both test methods can work if iodine solution is used in place of biiodate solution as the titrating agent. Validation of these test methods through round-robin testing, however, has not been carried out.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1266: Standard Test Method for Sulfur in Petroleum Products (Lamp Method)

CFR Section(s): 40 CFR 60.106(j)(2)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1266 – 98

An American National Standard

Designation: 107/86

Standard Test Method for Sulfur in Petroleum Products (Lamp Method)[1]

This standard is issued under the fixed designation D 1266; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This test method has been adopted for use by government agencies to replace Method 5201 of Federal Test Method Standard No. 791b

1. Scope

1.1 This test method covers the determination of total sulfur in liquid petroleum products in concentrations from 0.01 to 0.4 mass % (Note 1). A special sulfate analysis procedure is described in Annex A1 that permits the determination of sulfur in concentrations as low as 5 mg/kg.

NOTE 1—The comparable lamp method for the determination of sulfur in liquefied petroleum gas is described in Test Method D 2784. For the determination of sulfur in heavier petroleum products that cannot be burned in a lamp, see the bomb method (Test Method D 129) the quartz tube method (IP 63), or the high-temperature method (Test Method D 1552).

1.2 The direct burning procedure (Section 9) is applicable to the analysis of such materials as gasoline, kerosine, naphtha, and other liquids that can be burned completely in a wick lamp. The blending procedure (Section 10) is applicable to the analysis of gas oils and distillate fuel oils, naphthenic acids, alkyl phenols, high sulfur content petroleum products, and many other materials that cannot be burned satisfactorily by the direct burning procedure.

1.3 Phosphorus compounds normally present in commercial gasoline do not interfere. A correction is given for the small amount of acid resulting from the combustion of the lead anti-knock fluids in gasolines. Appreciable concentrations of acid-forming or base-forming elements from other sources interfere when the titration procedure is employed since no correction is provided in these cases.

1.4 The preferred units are acceptable metric units.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Note 5.

2. Referenced Documents

2.1 *ASTM Standards:*

D 129 Test Method for Sulfur in Petroleum Products (General Bomb Method)[2]

D 1193 Specification for Reagent Water[3]

D 1229 Test Method for Rubber Property—Compression Set at Low Temperatures[4]

D 1552 Test Method for Sulfur in Petroleum Products (High Temperature Method)[2]

D 2784 Test Method for Sulfur in Liquefied Petroleum Gases (Oxy-Hydrogen Burner or Lamp)[5]

E 11 Specification for Wire–Cloth Sieves for Testing Purposes[6]

2.2 *Institute of Petroleum Standard:*[7]

IP 63 Sulfur Content — The Quartz Tube Method

3. Summary of Test Method

3.1 The sample is burned in a closed system, using a suitable lamp (Fig. 1) and an artificial atmosphere composed of 70 % carbon dioxide and 30 % oxygen to prevent formation of nitrogen oxides. The oxides of sulfur are absorbed and oxidized to sulfuric acid by means of hydrogen peroxide solution which is then flushed with air to remove dissolved carbon dioxide. Sulfur as sulfate in the absorbent is determined acidimetrically by titration with standard sodium hydroxide solution, or gravimetrically by precipitation as barium sulfate (see Annex A2).

3.2 Alternatively, the sample may be burned in air, the sulfur as sulfate in the absorbent being determined by precipitation as barium sulfate for weighing (see Annex A2).

NOTE 2—In the absence of acid-forming or base-forming elements, other than sulfur, results by the volumetric and gravimetric finishes described are equivalent within the limits of precision of the method.

3.3 For sulfur contents below 0.01 mass % it is necessary to

---

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

Current edition approved Feb. 10, 1998. Published April 1998. Originally published as D 1266 – 69 T. Last previous edition D 1266 – 91 (1995).

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 09.01.

[5] *Annual Book of ASTM Standards*, Vol 05.02.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available from the Institute of Petroleum, 61 New Cavendish St., London, W.I., England.

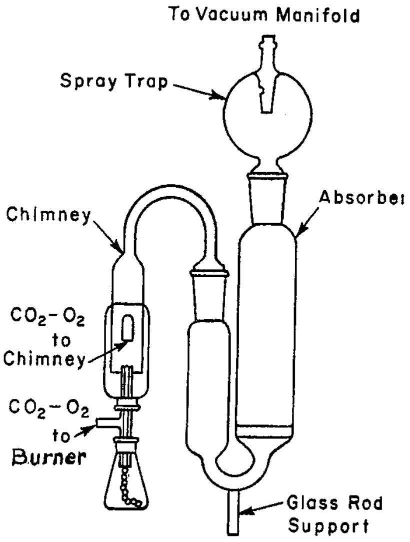


FIG. 1 Illustrative Sketch of the Assembled Lamp Unit

determine the sulfate content in the absorber solution turbidimetrically as barium sulfate (see Annex A1).

4. Significance and Use

4.1 This test method provides a means of monitoring the sulfur level of various petroleum products and additives. This knowledge can be used to predict performance, handling, or processing properties. In some cases the presence of sulfur components is beneficial to the product and monitoring the depletion of sulfur compounds provides useful information. In other cases the presence of sulfur compounds is detrimental to the processing or use of the product.

5. Apparatus

5.1 *Absorbers, Chimneys, Lamps, and Spray Traps* (Fig. 1) as required are described in detail in Annex A3. The standard flask and burner (Fig. A3.1) as shown is not suitable for burning highly aromatic mixtures without blending. The flask and burner for aromatic samples (Fig. A3.1) permits burning these samples directly without blending and may also be used to burn nonaromatic samples; with this lamp, a second port with control valve in the burner manifold is required.

5.2 *Cotton Wicking*—Clean, unused, uniform, twisted white cotton yarn of good quality.[8] For the burner to burn aromatic samples use long staple, fine-spun, commercial *fine* grade.[9]

5.3 *Manifold System* consisting of a vacuum manifold with regulating device, valves, and so forth (Fig. 2) and a dual manifold (burner and chimney) supplying a gas mixture of approximately 70 % carbon dioxide (CO_2) and 30 % oxygen (O_2) at regulated pressures. The vacuum manifold shall be connected to a pump of sufficient capacity to permit a steady gas flow of about 3 L/min through each absorber and to maintain a constant manifold pressure of approximately 40 cm of water below atmospheric. The gas mixture in the chimney manifold shall be maintained at a nearly constant pressure of 1 to 2 cm of water and the burner manifold at approximately 20 cm of water. A suitable arrangement is shown in Fig. 2 and described in Annex A3, but any other similar system can be used. Modifications of the manifold and associated equipment for burning samples in air are shown in Fig. A2.1 and described in Annex A2.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[10] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II or Type III of Specification D 1193.

6.3 *Carbon Dioxide and Oxygen*—The carbon dioxide (CO_2) and the oxygen (O_2) shall each be at least 99.5 % pure. These gases shall meet the requirements of 9.5.

6.4 *Diluent*—The diluent used shall have a sulfur content less than 0.001 mass %, be completely miscible with the sample to be analyzed, and permit burning at a moderate rate without smoking. Normal heptane, *iso*octane, and absolute ethyl alcohol have been found suitable (Note 10).

6.5 *Hydrochloric Acid (1 + 10)*—Mix 1 volume of concentrated hydrochloric acid (HCl, relative density 1.19) with 10 volumes of water.

6.6 *Hydrogen Peroxide Solution (1 + 19)*—Mix 1 volume of concentrated hydrogen peroxide (H_2O_2, 30 percent) with 19 volumes of water. Store in a dark-colored glass-stoppered bottle.

6.7 *Methyl Purple Indicator*—Aqueous solution containing approximately 0.1 % active constituent.[11] (Not methyl violet.)

6.8 *Sodium Hydroxide Solution (100 g/L)*—Dissolve 100 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

6.9 *Sodium Hydroxide, Standard Solution* (0.05 *M*)—Dilute 2.8 mL of saturated NaOH solution to 1 L (Note 3), using for this purpose the clear saturated solution decanted after standing long enough to permit any precipitate to settle out. Standardize by titration against standard acid, using the methyl purple indicator. Store in an alkali-resistant glass bottle and protect to minimize contamination by CO_2 from the air. Use only pure

---

[8] Yarn, white, 4–strand (2 to 3 mg/cm/strand), available from Koehler Instrument Co., 1595 Sycamore Ave., Bohemia, NY 11716, or the type marketed by various suppliers in the United Kingdom as 13s/14 ends, scoured, and bleached has been found suitable for this purpose.

[9] Available from Thomas Scientific, P.O. Box 99, Swedesboro, NJ 08085-0099.

[10] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[11] Fleisher Methyl Purple Indicator, U. S. Patent No. 2416619 may be obtained from Harry Fleisher Chemical Co., Benjamin Franklin Station, Washington, DC 20044, or from any chemical supply company handling Fleisher Methyl Purple.

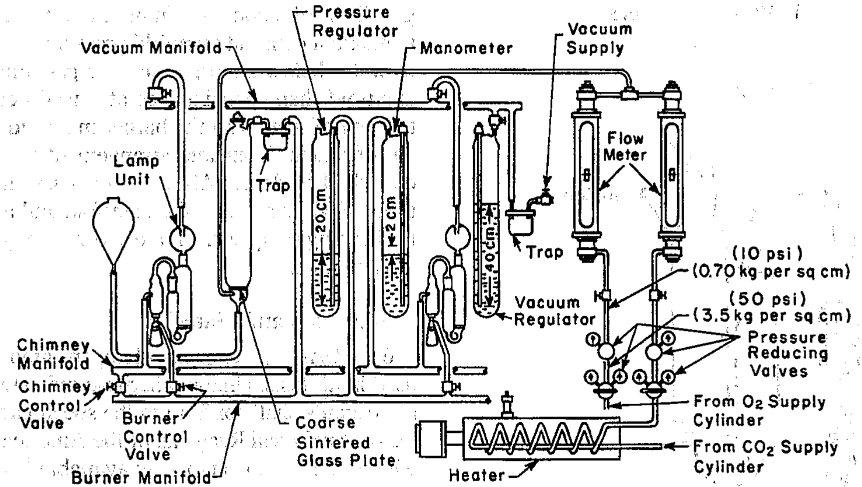


FIG. 2 Schematic Diagram of CO_2-O_2 Supply Manifold and Lamp System

gum rubber tubing for connections between the storage bottles and burets.

NOTE 3—The calculation of results can be simplified by adjusting the molarity of the NaOH solution to 0.0624 ± 0.0001. Then 1 mL of the NaOH solution will be equivalent to 0.0010 g of sulfur. In this case, the factor 16.03*M* in the calculation (see 12.1) becomes 1.000.

6.10 Quality Control (QC) Sample(s), preferably are portions of one or more liquid petroleum materials or product standards of known sulfur content that were not used in the generation of the instrument calibration curve. These (QC) samples are to be used to check the validity of the testing process as described in Section 12. An ample supply of QC sample material shall be available for the intended period of use, and must be homogeneous and stable under the anticipated storage conditions.

7. Preparation of Apparatus

7.1 When the apparatus is first assembled, charge the absorber with 30 ± 2 mL of water. Adjust the individual valves between the vacuum manifold and spray traps so that approximately 3 L of air per minute will be drawn through each absorber when the chimney outlets are open to the atmosphere, while maintaining the pressure in the vacuum manifold at approximately 40 cm of water below atmospheric. When all adjustments have been made, remove the water from the absorbers. The height of the liquids in the pressure and vacuum regulators is indicated in Fig. 2, and during operation a slow leak of gas should be maintained through them.

NOTE 4—In use, place 300 to 400 mL of H_2O_2 solution (1 + 19) in the scrubber. Since the manifold manometer also serves as a scrubber at the end of the test to remove CO_2 from the absorbent use H_2O_2 solution (1 + 19) as the manometric liquid. Replace weekly or whenever the volume becomes appreciably less than the original.

7.2 Neutralize the H_2O_2 solution (1 + 19) immediately before use. As 30 mL of the solution is needed, transfer to a beaker multiples of 30 mL sufficient for the number of absorbers to be used simultaneously. Add 1 drop of methyl purple indicator solution for each 100 mL of H_2O_2 solution and then add 0.05 *N* NaOH solution dropwise until the color changes from purple to light green.

7.3 Introduce 30 ± 2 mL of the freshly neutralized H_2O_2 solution (1 + 19) into the larger bulb of each absorber. In addition, for each set of samples burned, prepare an extra absorber for use as a control blank. Attach the spray traps and chimneys and connect them to their respective manifolds by means of sulfur-free rubber tubing. Close the chimney openings by means of corks.

7.4 With the burner control valves closed, the valve to the vacuum regulator fully open, and the pressure in the vacuum manifold adjusted to approximately 40 cm of water below atmospheric, turn on the CO_2 and O_2 supplies (**Warning**—see Note 5). Adjust the chimney manifold control valve so that, at the required rate of flow through the absorbers, only a small stream of CO_2-O_2 gas escapes at the pressure regulator, a small stream of air enters at the vacuum regulator, and the pressure in the chimney manifold is 1 to 2 cm of water. Minor adjustment of the vacuum regulator and vacuum control valve may be necessary to achieve this condition (Note 6).

NOTE 5—**Warning:** A hazardous (explosive) condition can result if the CO_2 supply is interrupted and the O_2 flow is continued while samples are being burned. The installation of suitable warning or control equipment is recommended.

NOTE 6—It is convenient to balance the gas flow system by regulating the pressure in the vacuum manifold. This is done by raising or lowering the air inlet tube in the vacuum regulator by sliding it in a rubber sleeve.

7.5 Cut the wicking to 30-cm lengths. Use the number of lengths dictated by the sample (see Section 8); fold the wicking once to give a 15-cm long bundle for threading the burners. Thread the required number of burners by inserting the looped ends into the top of the inner tube of the burner. Draw the wicking through by means of a metal hook. Trim the wick as close as possible to the top of the burner with a pair of sharp scissors. It is essential that thoroughly cleaned burners and new wicking be used for each test.

8. Control of Combustion

8.1 Most types of liquid samples burn with a luminous yellow flame, the size and shape of which is dependent on the gas flow to the burner, the volatility of the material, the tightness of the fit of the wick in the burner tube, and the position of the top of the wick relative to the top of the burner. It is preferable that the latter two variables be fixed with relation to the first before burning is started so that the flame can be controlled by variation in the rate of CO_2-O_2 flow.

8.2 Highly volatile samples require a tight-fitting wick, the top of which can need to be several millimetres below the top of the burner, and in extreme cases may have to be cooled in ice during the burning. Less volatile materials require a more loosely fitting wick and can require warming.

8.3 After trimming, draw the wick down until the trimmed edge is flush with or just a little below the top of the burner. With the burner for aromatic samples, the distance from the top of the burner to the top of the wicking should be 8 mm or more for benzene and 4 mm for toluene; a slight heating of the upper end of the burner will be helpful in starting vaporization of heavier materials.

8.4 To use the standard lamp, light the wick and then slowly admit combustion atmosphere to the burner to obtain a smoke-free flame. To use the burner for aromatic samples, introduce a small amount of combustion atmosphere into the flask to provide sufficient vapor for lighting the burner. After lighting the burner, introduce combustion atmosphere directly into the burner to prevent smoking and to adjust the flame size. If the flame is accidentally snuffed out, relight.

8.5 A short burning period (1 to 2 min is usually sufficient) at low flame height is necessary to allow combustion to reach equilibrium before the flame size can be increased without causing a smoky flame. In adjusting the standard lamp, the entire control is at the burner. For the burner for aromatic samples, first adjust the flow of gas to the flask and then reduce the flow of gas to the burner as required. In any case, it is essential that the flame burn smoothly and symmetrically and without jets in the inner cone or smoke on the outer fringes.

8.6 Satisfactory combustion of materials difficult to burn can sometimes be obtained by increasing the O_2 content of the combustion atmosphere. Never increase the O_2 content of the combustion atmosphere to more than 40 %.

8.7 Before extinguishing the flames, allow the sample to burn until the flask and wicking appear to be dry and the flame has reduced considerably in size; frequently the flame continues to burn a short time after the flask appears dry because of the sample in the wick. For example, for gasoline samples, which burn with a high flame, the flame should be extinguished when it is only 3 to 4 mm high. If the flame is permitted to burn until it goes out, partially oxidized substances (probably organic acids) are produced; as a result broad, indistinct end points are obtained. When samples are not burned until the flask is apparently dry, erratic results may be obtained. In the case of volatile samples, any unburned sample will escape from the burner during weighing. When elemental sulfur is present, it is particularly important that the sample be burned to apparent dryness and that the wick be maintained flush with the top of the burner to ensure complete combustion. With mixtures containing light and heavy hydrocarbons, the more volatile materials seem to burn first, possibly concentrating sulfur compounds in the material remaining behind.

9. Procedure for Direct Combustion of Liquid Samples (see also Annex A2)

9.1 By means of an appropriate pipet, introduce into the flask of each lamp an approximate quantity of sample as indicated in Table 1. Stopper the flasks with clean, numbered corks. Weigh each flask and its burner to the nearest 0.005 g.

NOTE 7—While the stoppered flasks and prepared burners can all be weighed separately, it is usually more convenient to place each flask and its burner on the balance pan and obtain the combined weight in a single weighing.

9.2 Handling each lamp individually, insert the burner in the flask. As soon as the sample has risen by capillary action to the top of the wick, connect the side tube of the burner to the burner manifold by means of sulfur-free rubber tubing. Light the burner with a sulfur-free flame (such as an alcohol lamp) and insert into the chimney, pinching off the connection between the chimney and the chimney manifold during the insertion if the flame tends to be blown out. At the same time, adjust the gas flow to the burner so that the flame is maintained at a point just below smoking and has a steady symmetrical appearance. Continue in this manner until all lamps have been placed in the chimneys. Make any minor adjustment of the chimney manifold control valve necessary to maintain the required pressure (see Section 7). During the burning, and particularly during the latter stages when the flame becomes small, decrease the CO_2-O_2 supply to the burners in order to prevent extinction of the flames.

NOTE 8—When incomplete combustion occurs, the absorber liquid will foam excessively.

9.3 When the burning of each sample is complete, as evidenced by the flame becoming small owing to depletion of the sample, remove the burner and flask from the chimney, extinguish the flame, shut off the CO_2-O_2 supply to the burner and stopper the chimney opening. Immediately reweigh the flask, burner, and numbered cork. When all combustions have been completed, turn off the CO_2 and the O_2 supplies, close the chimney control valve, and close the connection to the vacuum regulator; this will cause air to be drawn into the chimney manifold through the manometer. Allow air to be drawn through the absorbers in this manner for 5 min to remove dissolved CO_2 from the absorbent; then close the vacuum control valve.

NOTE 9—If it is desired to conserve the combustion atmosphere, the gas flow through each individual absorber can be turned off upon completion of the burning period. To accomplish this, pinch off the rubber tubing connecting the spray trap to the vacuum manifold, reduce the flow of mixed gases at the rotameters proportionately, and readjust the vacuum control valve and the chimney control valve. When the burning of all samples has been completed, it is necessary to remove the pinch clamps and readjust the vacuum control valve in order to draw air at the required rate through the absorbers for removal of dissolved CO_2.

TABLE 1 Sample Size for Direct Combustion of Liquid Samples

Sulfur Content, mass percent	Sample Size g	Sample Size mL
Under 0.05	10 to 15	20
0.05 to 0.4	5 to 10	10

9.4 Rinse the chimneys and spray traps three times, using about 10 mL of water each time. When the sample contains lead anti-knock fluids, use hot water to rinse the chimneys. Add the rinsing to the absorbers, and titrate as directed in Section 11.

9.5 *Blank*—Leave the chimney of the blank absorber (see 7.3) stoppered, and allow the CO_2-O_2 stream to pass through that absorber until all samples started at one time have finished burning. Turn off the CO_2 and the O_2 supplies and aerate the blank absorber in the same manner as the sample absorbers (see 9.3). Titrate the absorber liquid as directed in Section 11. Normally, the combustion atmosphere blank will be small, but if the titration requires more than 0.1 mL of 0.05 *N* NaOH solution discard the determination and replace the CO_2 cylinder.

10. Procedure for Blending and Combustion of Liquid Samples

10.1 Add 6 mL of sulfur-free diluent to each flask. Stopper the flasks with numbered corks and weigh to the nearest 0.005 g. By means of a pipet, introduce into the flask of each burner an approximate quantity of sample as indicated in Table 2; swirl to mix thoroughly, and reweigh.

NOTE 10—Alternatively, make a quantitative 40 % blend of the sample in sulfur-free diluent and proceed as described in Section 9.

10.2 Insert the burner and burn as described in 9.2. Remove each lamp from its chimney as the flame nears extinction and extinguish the flame. Add 2 mL of diluent, allowing the diluent to rinse down the walls of the flask. Burn the additional diluent and repeat the addition of diluent and burning one more time so that a total of 10 mL of diluent has been burned.

NOTE 11—In this case, it is desirable that a 10-mL diluent blank be run; the titration of the absorber solution from this blank, shall not exceed 0.1 mL of 0.05 *M* NaOH solution.

10.3 After all lamps have completed burning, turn off the CO_2 and O_2 supplies, close the connection to the vacuum regulator, draw air through the absorbers for 5 min, and finally close the vacuum control valve. Rinse the chimneys and spray traps three times, using about 10 mL of water each time. Add the rinsings to the absorbers, and titrate as directed in Section 11.

11. Titration of Absorbent Solution

11.1 Add 3 to 4 drops of methyl purple indicator solution to the liquid in each absorber. Titrate the absorbent solution by introducing 0.05 *N* NaOH solution from a buret into the smaller bulb of the absorber. Use a 10-mL microburet if less than 10 mg of sulfur is expected to be present in the absorber. Stir during the titration by applying suction intermittently to the top of the larger bulb.

NOTE 12—When incomplete combustion of the sample occurs, the air drawn through the absorber during the titration will have a characteristic taste or odor and the end point will be broad. In these cases, discard the determination.

TABLE 2 Sample Size for Testing Blended Liquid Samples

Sulfur Content, mass percent	Sample Size g	mL
0.4 and under	3 to 4	5

12. Calculations

12.1 Calculate the sulfur content of liquid samples as follows:

$$\text{Sulfur content, mass percent} = 16.03\, M \times (A/10\, W) \qquad (1)$$

where:

A = millilitres of NaOH solution required to titrate the acid in the absorbent solution from the burned sample,
M = molarity of the NaOH solution (see Note 3), and
W = grams of sample burned.

12.2 When it is required by specifications to correct the sulfur content (Note 13) for lead antiknock fluids, calculate the corrected values as follows:

$$\text{Corrected sulfur content, mass percent} = S - LF \qquad (2)$$

where:

F = 0.0015 if the sample contains aviation lead antiknock fluid or 0.0035 if the sample contains tetraethyllead, tetramethyllead, or the mixed lead alkyl antiknock fluid,
L = lead content, g/U.S. gal, and
S = sulfur content, mass %.

NOTE 13—These corrections are based on experiments of burning fuels blended with antiknock fluid containing tetraethyllead and ethylene halide in commonly-used combinations. Tetramethyllead and the mixed lead alkyl antiknock fluids contain the same ethylene halide combination as the tetraethyllead fluid.

NOTE 14—To convert grams of lead per Imperial gallon into grams per U.S. gallon, multiply by 0.8326. Multiply by 3.7853 to convert grams of lead per litre into grams per U.S. gallon.

13. Report

13.1 Report the results of the test to the nearest 0.01 % for sulfur at a level of 0.05 % and higher, and the specific test procedure used.

14. Quality Control

14.1 Confirm the performance of the apparatus or the procedure, or both, each day it is in use by analyzing a QC sample (6.10) that is representative of samples typically analyzed. Increase the analysis frequency of the QC sample if a large number of samples are analyzed. Analysis of the result(s) from the QC sample(s) can be carried out using control charts[12], or other statistically equivalent techniques, to ascertain the control status of the total testing process. Any out of control data should trigger investigation for root cause. The QC sample precision shall be checked against the ASTM method precision to ensure data quality.

15. Precision and Bias

15.1 The precision of this test is not known to have been obtained in accordance with currently accepted guidelines (for example, in Committee D-2 Research Report RR-D-2-1007, "Manual on Determining Precision Data for ASTM Methods

---

[12] ASTM Manual 7, *Manual on Presentation of Data and Control Chart Analysis*, 6th edition, available from ASTM Headquarters.

on Petroleum Products and Lubricants").[13]

15.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Repeatability	0.005

15.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Reproducibility	0.010 + 0.025S

where:

S = the total sulfur content, mass percent, of the sample.

15.2 *Bias*—It is not practicable to specify bias of Test Method D 1226 for measuring sulfur becuase the responsible subcommittee, after diligent search, was unable to attract volunteers for an interlaboratory study.

16. Keywords

16.1 lamp; sulfur

ANNEXES

(Mandatory Information)

A1. TEST METHOD OF TEST FOR TRACE QUANTITIES OF SULFUR

A1.1 Scope

A1.1.1 This annex describes a procedure for extending the lamp method of test for sulfur to the analysis of samples having sulfur contents as low as 5 ppm (Note A1.1): The procedure is not applicable for the determination of less than 300 mg/kg of sulfur in liquids containing lead antiknock compounds.

A1.1.1.1 Only by the exercise of the most scrupulous care and attention to details can reliable results be obtained by this method. Before placing new glassware into use and thereafter as required, wash the glassware with concentrated nitric acid. Rinse three times with tap water, followed by three rinsings with deionized distilled water. Reserve units of glassware for use in this method alone.

A1.2 Summary of Test Method

A1.2.1 A sample of suitable size is burned as described in Section 9. Sulfate ion in the absorber solution is determined by precipitation as barium sulfate and measurement of the turbidity of a suspension of the precipitate. The suspension is stabilized by the addition of alcohol and glycerin, and its turbidity is measured by use of a spectrophotometer or filter photometer.

A1.3 Additional Apparatus

A1.3.1 *Photometer*— Preferably a spectrophotometer having an effective band width of about 50 nm and equipped with a blue-sensitive phototube for use at 450 nm, or alternatively a filter photometer equipped with a color filter having a maximum transmission at approximately 450 nm.

A1.3.2 *Absorption Cells* having optical path lengths of 5 cm are preferred. With use, the cells may become coated with a film. To remove this film, wash the cells with a detergent using a soft brush. Rinse thoroughly with deionized water following cleaning.

NOTE A1.1—The procedure as written assumes an absorbance change of about 0.100 for each 0.1 mg of sulfur in 50 mL of solution measured in a 5-cm cell. Photometers employing cells of shorter optical paths give proportionately poorer precision.

A1.3.3 *Scoop*, capable of dispensing 0.30 ± 0.01 g of barium chloride dihydrate as specified in A1.4.2.

A1.3.4 *Magnetic Stirrer*, equipped with tetrafluoroethylene covered stirring bars about 32 mm (1¼in.) long.

A1.3.5 *Lamp Assembly*, as described in Annex A3. Reserve complete units consisting of flask, burner, chimney, absorber, and spray trap for use in this procedure only.

A1.4 Additional Reagents[14]

A1.4.1 *Alcohol-Glycerin Mixture*—Mix 2 volumes of denatured ethyl alcohol conforming to Formula No. 3A of the U.S. Bureau of Internal Revenue or ethyl alcohol (95 % by volume) with 1 volume of glycerin.

A1.4.2 *Barium Chloride Dihydrate* ($BaCl_2 \cdot 2H_2O$)—Crystals passing an ASTM E 11 20-mesh sieve or a BS 18-mesh sieve and retained on an ASTM E11 30-mesh sieve or a BS 30-mesh (See Specification E 11).

NOTE A1.2—The crystal size of the $BaCl_2 \cdot 2H_2O$ is an important variable that affects the development of turbidity.

A1.4.3 *Hydrochloric Acid* (*1 + 12*)—Add 77 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to a 1-L volumetric flask and dilute to the mark with deionized water.

A1.4.4 *Hydrochloric Acid* (*1 + 215*)—Add 60 mL of 1 + 12 HCl to a 1-L volumetric flask and dilute to the mark with deionized water.

A1.4.5 *Sulfuric Acid* (*1 mL = 0.100 mg S*)—Dilute 6.24± 0.01 mL of 1 *N* sulfuric acid (H_2SO_4) to exactly 1 L with deionized water. Check the dilution by titration against standard NaOH solution of about the same normality and adjust the concentration, if necessary, so that each millilitre of this solution is equivalent to 0.100 mg of S.

---

[13] *Annual Book of ASTM Standards*, Vol 05.03.

[14] For Purity of Reagents, see 6.1.

A1.4.6 *Water, Deionized Distilled*—Percolate water through a column of mixed anion and cation exchange resins.

NOTE A1.3—A means for determining when to replace the exchange resins should be supplied. Use of a simple electrical conductivity meter has been found satisfactory for this purpose.

A1.5 Calibration

A1.5.1 Into 50-mL volumetric flasks introduce, by means of a buret, 0.25, 0.50, 0.75, 1.00, 1.50, 2.00, 3.00, and 5.00 mL of H_2SO_4 (1 ml = 0.100 mg S). Add 3.0 mL of HCl (1 + 12) to each flask, dilute to volume with water, and mix thoroughly. Prepare a reagent blank standard in a similar way, omitting the H_2SO_4.

A1.5.2 Pour the entire contents of each flask into a 100-mL beaker, add by means of a pipet 10 ± 0.1 mL of alcohol-glycerin mixture, and mix for 3 min on the magnetic stirrer. Select a stirring speed just below that which might cause loss of sample through splashing. Maintain this speed throughout the entire procedure.

A1.5.3 Allow the solution to stand undisturbed for 4 min. Transfer to an absorption cell and measure the initial absorbance, using water as reference.

A1.5.4 Return the solution to the beaker and add 0.30± 0.01 g of $BaCl_2 \cdot 2H_2O$ crystals, either by weighing this amount or by use of the scoop. Stir with the magnetic stirrer for exactly 3 min. Allow to stand for an additional 4 min, transfer to the cell, and again measure the absorbance relative to water.

A1.5.5 Following steps described in A1.5.2-A1.5.4, obtain a reagent blank reading by subtracting the initial absorbance of the reagent blank standard from that obtained after addition of $BaCl_2 \cdot 2H_2O$. This reading should not exceed 0.005.

A1.5.6 Obtain the net absorbance for each standard by subtracting the initial absorbance and reagent blank reading from the absorbance obtained in accordance with A1.5.4. Plot the net absorbance of each standard against milligrams of sulfur contained in 50 mL of solution, and draw a smooth curve through the points.

A1.5.7 Check the calibration curve daily by making single determinations to detect possible shifts.

A1.6 Procedure for Combustion of Samples

A1.6.1 Prepare the combustion apparatus and burn between 5 and 30 g of sample depending on the expected sulfur level (Note A1.5). Follow the general procedures described in Sections 7, 8, and 9 of the main method. The requirements for initial neutralization of the H_2O_2 solution (see 7.2) and for final removal of dissolved CO_2 from this solution (see 9.3, and 10.3), may be omitted. Draw combustion atmosphere through one absorber of a set to serve as a blank on the purity of this atmosphere. Reserve all glassware exclusively for use with this trace procedure to avoid any possible contamination from other sources. Transfer the absorber solution, containing rinsings from the spray trap and chimney (see 9.4), to a 250-mL beaker, rinse the absorber two or three times with 10-mL portions of water, and add the rinsings to the solution in the beaker.

A1.6.1.1 A sample size that will yield between 0.15 and 2.5 mg of sulfur in the absorber must be selected. This will then allow subsequent direct application of the procedures described in A1.6.3 and A1.7 and will avoid the necessity for using less than a one-fifth aliquot of the absorber solution for analysis. When the sulfur level of the sample is about 15 mg/kg or less, at least 30 g of sample must be burned. To accommodate the large sample sizes, a burner flask of suitable size must be fabricated to replace the standard 25-mL flask. In recognition of the larger size of the flask, it is preferable to use 18 cm of wicking rather than the 15 cm specified in 7.5. To avoid excessive depletion of absorber liquid caused by the longer burning time for larger samples, it is preferable to charge the absorbers with 50 mL of the hydrogen peroxide solution instead of the 30 mL specified in 7.2.

A1.6.2 Reduce the volume of the absorber solution to about 20 mL by evaporation on a hot plate. Quantitatively transfer the resulting solution to a 50-mL volumetric flask, rinsing the beaker with several small portions of water. Add 3 mL of HCl (1 + 12) to the flask, make up to volume with water, and mix thoroughly.

A1.6.3 If the sulfur content of the absorber solution is known to be less than 0.5 mg, use the entire contents of the volumetric flask for analysis. If the approximate sulfur content is unknown or is expected to exceed 0.5 mg, transfer a 10-mL aliquot to a second 50-mL volumetric flask and dilute the solution in both flasks to volume with HCl (1 + 215). Use the more dilute solution first and, if less than 0.05 mg of sulfur is found, then use the more concentrated solution. Prepare a dilution of the combustion atmosphere blank similar to the solution used for analysis. Analyze the solutions as described in A1.7.

A1.7 Procedure for Analysis of Solutions

A1.7.1 Pour the entire contents of the 50-mL volumetric flask containing the solution to be analyzed into a 100-mL beaker and proceed as directed in A1.5.2-A1.5.4. Treat the combustion atmosphere blank in the same way and obtain a combustion atmosphere-reagent blank reading by subtracting its initial absorbance from that obtained after addition of $BaCl_2 \cdot 2H_2O$.

NOTE A1.4—Should the blank reading exceed 0.020, the precision obtainable will be impaired. In this event, make an analysis of the reagents alone to determine whether the atmosphere or reagents are at fault. Place 30 mL of the H_2O_2(1:5 percent) in the 50-mL volumetric flask, dilute to the mark with HCl (1 + 215), and proceed as described in A1.5.5. If this reagent blank reading exceeds 0.010, results should not be considerable reliable.

A1.7.2 Obtain the net absorbance of the analysis solution by subtracting the initial absorbance and the combustion atmosphere-reagent blank reading from that obtained after addition of $BaCl_2 \cdot 2H_2O$.

A1.7.3 Convert net absorbance to milligrams of sulfur by using the calibration curve.

A1.8 Calculation

A1.8.1 Calculate the amount of sulfur in the sample as follows:

$$\text{Sulfur content, mg/kg} = (A/WF) \times 1000 \tag{A1.1}$$

where:

A = milligrams of sulfur read from the calibration curve,

W = grams of sample burned, and
F = aliquot fraction of the sample solution used for analysis.

A1.9 Precision and Bias

A1.9.1 The following criteria should be used for judging the acceptability of results (95 % confidence):

A1.9.1.1 *Repeatability*— Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Sulfur Content, mg/kg	Repeatability
5 to 80	0.116 × mg/kg S
Over 80 to 280	(0.01 × mg/kg S) + 8.5

A1.9.1.2 *Reproducibility*— The results submitted by each of two laboratories should be considered suspect if the two results differ by more than the following amounts:

Sulfur Content, mg/kg	Reproducibility
5 to 125	0.145 × mg/kg S
Over 125 to 280	(0.508 × mg/kg S) − 45.4

NOTE A1.5—For the determination of trace quantities of sulfur by a rapid burning method see Test Method D 2785.

A1.9.2 *Bias*—It is not practicable to specify the bias of Test Method D 1266, Annex A1 for measuring trace quantities of sulfur because the responsible subcommittee, after diligent search, was unable to attract volunteers for an interlaboratory study.

A2. AIR BURNING OF SAMPLE, GRAVIMETRIC FINISH

A2.1 Scope

A2.1.1 This procedure is recommended only for analyzing liquid petroleum samples that can be burned with a wick lamp.

A2.2 Apparatus

A2.2.1 The mainfold system described in 5.3 may be used with only a slight modification. Substitute filtered air for the CO_2-O_2 supply train and add a second sintered-plate scrubber to the incoming air line as shown in Fig. A2.1.

A2.3 Additional Reagents

A2.3.1 *Barium Chloride Solution (100 g/L)*—Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in water and dilute to 1 litre.

A2.3.2 *Hydrochloric Acid (relative density 1.19)*—Concentrated hydrochloric acid (HCl).

A2.3.3 *Hydrogen Peroxide Solution (30 %)*—Concentrated hydrogen peroxide (H_2O_2).

A2.3.4 *Sodium Hydroxide Solution (100 g/L)*—Dissolve 100 g of technical grade sodium hydroxide (NaOH) pellets in water and dilute to 1 L.

A2.3.5 *Sulfuric Acid (1 + 16)*—Mix 60 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) with 960 mL of water.

A2.4 Preparation of Apparatus

A2.4.1 Place 300 to 400 mL of NaOH solution in the first scrubber (Fig. A2.1) and the same amount of H_2O_2-H_2SO_4 solution (300 mL of H_2O, 30 mL of H_2SO_4 (1 + 16), and 30 mL of H_2O_2(30 %)) in the second scrubber. For apparatus in daily use, replace these solutions two times each week or whenever the volume becomes less than two thirds of the original.

A2.4.2 Make other preparations as described in Section 7, except that the H_2O_2 solution (1.5 %) need not be neutralized.

A2.5 Procedure for Combustion

A2.5.1 Burn the sample as described in Section 9, controlling combustion as described in Section 8. Use a sample size as prescribed in Table A2.1. Analyze the absorber solutions from the samples and blank as described in A2.6.1.

A2.6 Procedure for Analysis of Absorber Solution

A2.6.1 Transfer the absorber liquid to a 400-mL beaker. Rinse the absorber and chimney thoroughly with water and add the rinsings to the beaker. Filter the solution to remove any foreign material, receiving the filtrate in a 400-mL beaker having a mark to indicate 75 mL. Add 2 mL of HCl, heat to boiling, and add 10 mL of $BaCl_2$ solution, either in a fine

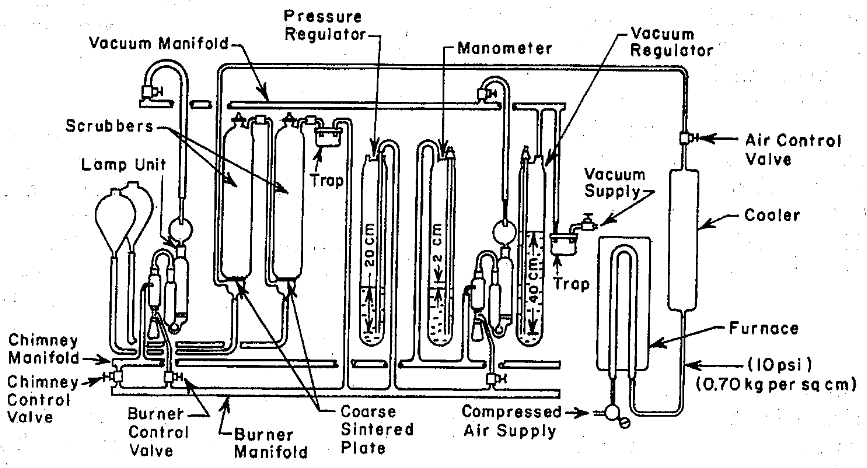


FIG. A2.1 Schematic Diagram of Purified Air Supply Manifold and Lamp System

TABLE A2.1 Sample Size for Air Burning of Liquid Samples

Sulfur Content, weight percent	Sample Size	
	g	mL
0.5 and under	5 to 10	10
Over 0.5	3 to 5	5

stream or dropwise. Stir the solution during the addition and for 2 min thereafter.

A2.6.2 Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume of approximately 75 mL, as indicated by the mark on the beaker. Remove the beaker from the hot plate (or other source of heat) and allow to cool 1 h before filtering.

A2.6.3 Filter the supernatant liquid through a close-texture, ashless filter paper. Wash the precipitate with water, first by decantation and then on the filter paper, until free of chloride ion. Transfer the paper and precipitate to a suitable weighed crucible, and dry at low heat until the moisture hasevaporated. Char the paper completely without igniting it, and finally ignite at a bright red heat until the precipitate is burned white (Note A2.1). After ignition is complete, allow the crucible to cool to room temperature and weigh.

NOTE A2.1—A satisfactory means of accomplishing these operations is to place the uncovered crucible containing the wet filter paper in a cold electric muffle furnace and turn on the current. Drying, charring, and ignition usually occur at the desired rate.

A2.7 Calculation

A2.7.1 Calculate the sulfur content of the sample as follows:

$$\text{Sulfur content, mass percent} = [(w - b) \times 13.73]/W \quad \text{(A2.1)}$$

where:

w = grams of barium sulfate ($BaSO_4$) precipitate in the absorber solution from the burned sample,

b = grams of $BaSO_4$ precipitate from the corresponding blank absorber solution (Note A2.2), and

W = grams of sample burned.

NOTE A2.2—The determination should be discarded if the blank correction used in the calculation exceeds 1.5 mg of $BaSO_4$. Frequently, impure reagents are the cause of this difficulty.

A2.8 Precision

A2.8.1 See Section 15 for recommended data.

A3. APPARATUS DETAIL

A3.1 Flask and Burner for Nonaromatic Samples

A3.1.1 A lamp of chemically resistant glass, consisting of a 25-mL Erlenmeyer flask and a burner that conforms to the dimensions shown in Fig. A2.1, shall be used. The burner consists of two concentric glass tubes, the external tube having a sidearm and standard-taper glass joints for connection with the flask and the chimney. The upper ends of both burner tubes shall be polished and shall have plane surfaces that are in the same horizontal plane. The burner shall have a 1-mm opening near its base to allow equalization of pressure between the chimney and the flask. When connected with the chimney, the lamp shall be held in position by rubber bands or metal springs stretched between glass hooks on the flask and chimney.

A3.2 Flask and Burner for Aromatic Samples

A3.2.1 A lamp of chemically resistant glass, consisting of a 25-mL Erlenmeyer flask with a side-arm and a burner that conforms to the dimension shown in Fig. A2.1, shall be used. The burner consists of two concentric glass tubes, the external tube having a sidearm and standard-taper glass joints for connecting the burner with the flask and the chimney. The upper ends of both burner tubes shall be polished and shall have plane surfaces that are in the same horizontal plane. When connected with the chimney, the lamps shall be held in position by rubber bands or metal springs stretched between glass hooks on the flask and chimney.

A3.3 Chimney

A3.3.1 A chimney of chemically resistant glass, conforming to the dimensions shown in Fig. A2.1 and provided with standard-taper glass joints for connection with the burner and absorber, shall be used.

A3.4 Absorber

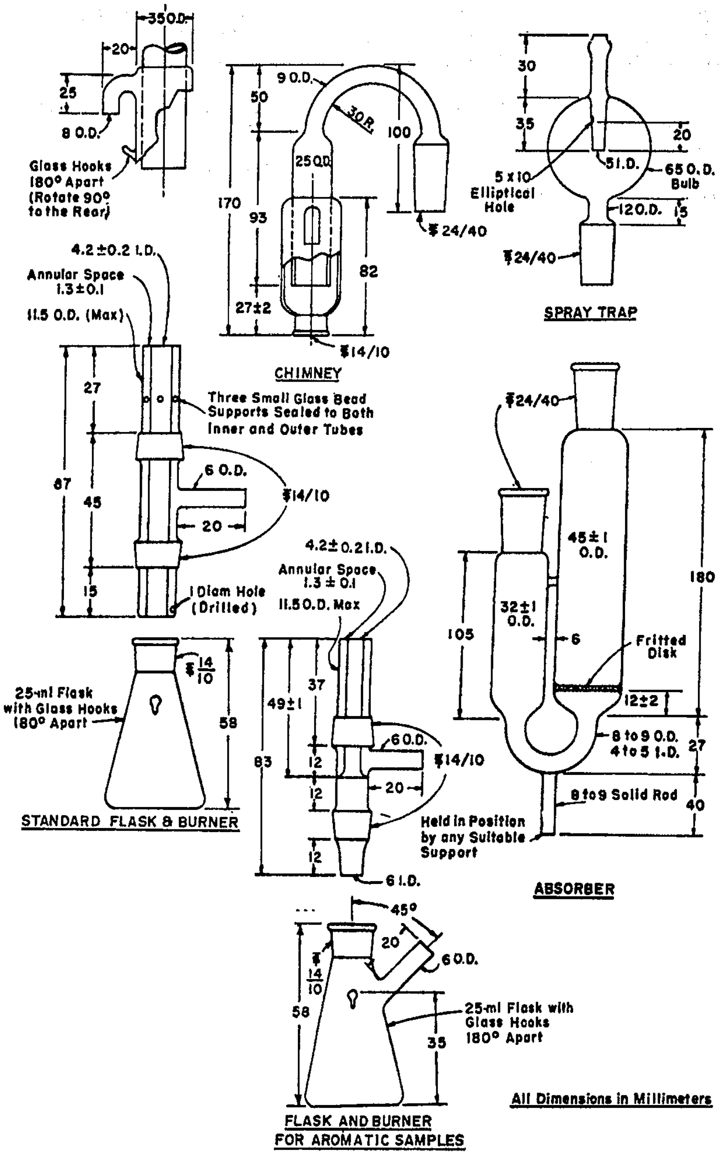
A3.4.1 An absorber of chemically resistant glass conforming to the dimensions shown in Fig. A3.1 and provided with standard-taper glass joints for connection with the chimney and spray trap, shall be used. A fritted disk with average pore diameter from 150 to 200 µm shall be sealed in the larger of two bulbs of the absorber. The fritted disk should be of such a porosity that, when 50 mL of water is placed in the absorber and air is passed through at the rate of 3.0 L/min in the forward direction, the pressure differential between the two sides of the absorber is between 15 and 23 cm of water and the air is dispersed uniformly.

A3.5 Spray Trap

A3.5.1 A spray trap of chemically resistant glass conforming to the dimensions shown in Fig. A2.1 and provided with a standard-taper glass joint for connection with the absorber, shall be used.

A3.6 Manifold System

A3.6.1 A satisfactory vacuum and combustion atmosphere manifold and supply system for supplying the required CO_2-O_2 mixture to the lamp assemblies is shown diagrammatically in Fig. 2. The gases are supplied from commercial cylinders, the pressure of each gas being adjusted to 10 ± 2 psig (0.70 ± 0.14 kg/cm ^{2}) by means of two single-stage regulating valves to ensure constant pressure at the flow-regulating needle valves. It is necessary to pass the CO_2 through a heat exchanger installed ahead of the regulating valves to prevent freezing of the valves. The gases are passed through a metering system consisting of two calibrated rotameter flow meters to indicate the proportion of the two gases mixed in the surge tank. Any number of lamp assemblies can be operated as a unit, the throughput of the flow



NOTE 1—Standard tapers 14/10, 24/40, or equivalent.

NOTE 2—The fritted disk shown in the drawing of the absorber shall be of such a porosity that, when 50 mL of water is placed in the absorber and air is passed through at the rate of 3.0 litres/min in the forward direction, the pressure differential between the two sides of the absorber is between 15 and 23 cm of water and the air is dispersed uniformly.

FIG. A3.1 Detailed Drawing of Combustion and Absorption Apparatus

meters being chosen accordingly. The tubing that connects the chimney manifold to the chimneys should have an internal diameter not smaller than 6.4 mm (¼ in.) in order to prevent unnecessary restriction in gas flow. The scrubber should have a capacity of about 1 litre.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1298: Standard Practice for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products

CFR Section(s): 40 CFR 75, Appendix D, Section 2.2.6

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1298 – 99

An American National Standard

MPMS Chapter 9.1

160/99

Standard Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method[1]

This standard is issued under the fixed designation D 1298; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the laboratory determination using a glass hydrometer, of the density, relative density (specific gravity), or API gravity of crude petroleum, petroleum products, or mixtures of petroleum and nonpetroleum products normally handled as liquids, and having a Reid vapor pressure of 101.325 kPa (14.696 psi) or less.

1.2 Values are measured on a hydrometer at either the reference temperature or at another convenient temperature, and readings corrected to the reference temperature by means of the Petroleum Measurement Tables; values obtained at other than the reference temperature being hydrometer readings and not density measurements.

1.3 Values determined as density, relative density, or API gravity can be converted to equivalent values in the other units at alternate reference temperatures by means of the Petroleum Measurement Tables.

1.4 Annex A1 contains a procedure for verifying or certifying the equipment for this test method.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 97 Test Method for Pour Point of Petroleum Products[2]
D 323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)[2]
D 1250 Guide for Petroleum Measurement Tables[2]
D 2500 Test Method for Cloud Point of Petroleum Oils[2]
D 3117 Test Method for Wax Appearance Point of Distillate Fuels[3]
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]
D 5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products[4]
E 1 Specification for ASTM Thermometers[5]
E 100 Specification for ASTM Hydrometers[5]

2.2 *Institute of Petroleum Standards*[6]

IP 389 Determination of wax appearance temperature (WAT) of middle distillate fuels by differential thermal analysis (DTA) or differential scanning calorimetry (DSC)
IP Standard Methods Book, Appendix A, Specifications – IP Standard Thermometers

2.3 *ISO Standards*[7]

ISO 649-1 Laboratory glassware – Density hydrometers for general purpose – Part 1: Specification

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *density, n*—the mass of liquid per unit volume at 15°C and 101.325 kPa with the standard unit of measurement being kilograms per cubic metre.

3.1.1.1 *Discussion*—Other reference temperatures, such as 20°C may be used for some products or in some locations. Less

---

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.02 on Static Petroleum Measurement.

Current edition approved June 10, 1999. Published August 1999. Originally published as D 1298 – 53. Last previous edition D 1298 – 85 (1990)$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] *Annual Book of ASTM Standards*, Vol 05.03.

[5] *Annual Book of ASTM Standards*, Vol 14.03.

[6] Available from Institute of Petroleum, 61 New Cavendish St., London, W1M 8AR, UK.

[7] Available from American National Standards Institute, 11 W 42nd St., 13th floor, New York, NY 10036.

preferred units of measurement; for example, kg/L or g/mL are still in use.

3.1.2 *relative density (specific gravity), n*—the ratio of the mass of a given volume of liquid at a specific temperature to the mass of an equal volume of pure water at the same or different temperature. Both reference temperatures shall be explicitly stated.

3.1.2.1 *Discussion*—Common reference temperatures include 60/60°F, 20/20°C, 20/4°C. The historic deprecated term specific gravity may still be found.

3.1.3 *API gravity, n*—a special function of relative density (specific gravity) 60/60°F, represented by:

$$° \text{API} = 141.5/(\text{sp gr } 60/60°\text{F}) - 131.5 \qquad (1)$$

3.1.3.1 *Discussion*—No statement of reference temperature is required, as 60°F is included in the definition.

3.1.4 *observed values, n*—values observed at temperatures other than the specified reference temperature. These values are only hydrometer readings and not density, relative density (specific gravity), or API gravity at that other temperature.

3.1.5 *cloud point, n*—temperature at which a cloud of wax crystals first appears in a liquid when it is cooled under specific conditions.

3.1.6 *pour point, n*—lowest temperature at which a test portion of crude petroleum or petroleum product will continue to flow when it is cooled under specified conditions.

3.1.7 *wax appearance temperature (WAT), n*—temperature at which waxy solids form when a crude petroleum or petroleum product is cooled under specified conditions.

4. Summary of Test Method

4.1 The sample is brought to a specified temperature and a test portion is transferred to a hydrometer cylinder that has been brought to approximately the same temperature. The appropriate hydrometer, also at a similar temperature, is lowered into the test portion and allowed to settle. After temperature equilibrium has been reached, the hydrometer scale is read, and the temperature of the test portion is taken. The observed hydrometer reading is reduced to the reference temperature by means of the Petroleum Measurement Tables. If necessary, the hydrometer cylinder and its contents are placed in a constant temperature bath to avoid excessive temperature variation during the test.

5. Significance and Use

5.1 Accurate determination of the density, relative density (specific gravity), or API gravity of petroleum and its products is necessary for the conversion of measured volumes to volumes or masses, or both, at the standard reference temperatures during custody transfer.

5.2 This test method is most suitable for determining the density, relative density (specific gravity), or API gravity of low viscosity transparent liquids. This test method can also be used for viscous liquids by allowing sufficient time for the hydrometer to reach equilibrium, and for opaque liquids by employing a suitable meniscus correction.

5.3 When used in connection with bulk oil measurements, volume correction errors are minimized by observing the hydrometer reading at a temperature close to that of the bulk oil temperature.

5.4 Density, relative density (specific gravity), or API gravity is a factor governing the quality and pricing of crude petroleum. However, this property of petroleum is an uncertain indication of its quality unless correlated with other properties.

5.5 Density is an important quality indicator for automotive, aviation and marine fuels, where it affects storage, handling and combustion.

6. Apparatus

6.1 *Hydrometers*, of glass, graduated in units of density, relative density, or API gravity as required, conforming to Specification E 100 or ISO 649-1, and the requirements given in Table 1.

6.1.1 The user should ascertain that the instruments used for this test conform to the requirements set out above with respect to materials, dimensions, and scale errors. In cases where the instrument is provided with a calibration certificate issued by a recognized standardizing body, the instrument is classed as certified and the appropriate corrections listed shall be applied to the observed readings. Instruments that satisfy the requirements of this test method, but are not provided with a recognized calibration certificate, are classed as uncertified.

6.2 *Thermometers*, having range, graduation intervals and maximum permitted scale error shown in Table 2 and conforming to Specification E 1 or IP Appendix A.

6.2.1 Alternate measuring devices or systems may be used, provided that the total uncertainty of the calibrated system is no greater than when using liquid-in-glass thermometers.

6.3 *Hydrometer Cylinder*, clear glass, plastic (see 6.3.1), or metal. The inside diameter of the cylinder shall be at least 25 mm greater than the outside diameter of the hydrometer and the height shall be such that the appropriate hydrometer floats in the test portion with at least 25 mm clearance between the bottom of the hydrometer and the bottom of the cylinder.

6.3.1 Hydrometer cylinders constructed of plastic materials shall be resistant to discoloration or attack by oil samples and shall not affect the material being tested. They shall not become opaque under prolonged exposure to sunlight.

6.4 *Constant-Temperature Bath*, if required, of dimensions such that it can accommodate the hydrometer cylinder with the test portion fully immersed below the test portion liquid surface, and a temperature control system capable of maintaining the bath temperature within 0.25°C of the test temperature throughout the duration of the test.

6.5 *Stirring Rod*, optional, of glass or plastic, approximately 400 mm in length.

TABLE 1 Recommended Hydrometers

Units	Range		Scale		Meniscus
	Total	Each Unit	Interval	Error	Correction
Density, kg/m³ at 15°C	600 - 1100	20	0.2	± 0.2	+0.3
	600 - 1100	50	0.5	± 0.3	+0.7
	600 - 1100	50	1.0	± 0.6	+1.4
Relative density (specific gravity) 60/60°F	0.600 - 1.100	0.020	0.0002	± 0.0002	+0.0003
	0.600 - 1.100	0.050	0.0005	± 0.0003	+0.0007
	0.600 - 1.100	0.050	0.001	± 0.0006	+0.0014
Relative density (specific gravity), 60/60°F	0.650 - 1.100	0.050	0.0005	±0.0005	
API	−1 - +101	12	0.1	± 0.1	

TABLE 2 Recommended Thermometers

Scale	Range	Graduation Interval	Scale Error
°C	−1 - +38	0.1	± 0.1
°C	−20 - +102	0.2	± 0.15
°F	−5 - +215	0.5	± 0.25

7. Sampling

7.1 Unless otherwise specified, samples of non-volatile petroleum and petroleum products shall be taken by the procedures described in Practices D 4057 and D 4177.

7.2 Samples of volatile crude petroleum or petroleum products are preferably taken by Practice D 4177, using a variable volume (floating piston) sample receiver to minimize any loss of light components which may affect the accuracy of the density measurement. In the absence of this facility, extreme care shall be taken to minimize these losses, including the transfer of the sample to a chilled container immediately after sampling.

7.3 *Sample Mixing*—may be necessary to obtain a test portion representative of the bulk sample to be tested, but precautions shall be taken to maintain the integrity of the sample during this operation. Mixing of volatile crude petroleum or petroleum products containing water or sediments, or both, or the heating of waxy volatile crude petroleum or petroleum products may result in the loss of light components. The following sections (7.3.1 to 7.3.4) will give some guidance on sample integrity maintenance.

7.3.1 *Volatile Crude Petroleum and Petroleum Products Having an RVP Greater than 50 kPa*—Mix the sample in its original closed container in order to minimize the loss of light components.

NOTE 1—Mixing volatile samples in open containers will lead to loss of light components and consequently affect the value of the density obtained.

7.3.2 *Waxy Crude Petroleum*—If the petroleum has a pour point above 10°C, or a cloud point or WAT above 15°C, warm the sample to 9°C above the pour point, or 3°C above the cloud point or WAT, prior to mixing. Whenever possible, mix the sample in its original closed container in order to minimize the loss of light components.

7.3.3 *Waxy Distillate*—Warm the sample to 3°C above its cloud point or WAT prior to mixing.

7.3.4 *Residual Fuel Oils*—Heat the sample to the test temperature prior to mixing (see 8.1.1 and Note 4).

7.4 Additional information on the mixing and handling of liquid samples will be found in Practice D 5854.

8. Procedure

8.1 *Temperature of Test*:

8.1.1 Bring the sample to the test temperature which shall be such that the sample is sufficiently fluid but not so high as to cause the loss of light components, nor so low as to result in the appearance of wax in the test portion.

NOTE 2—The density, relative density or API gravity determined by the hydrometer is most accurate at or near the reference temperature.

NOTE 3—The volume and density, the relative density, and the API corrections in the Petroleum Measurement Tables are based on the average expansions of a number of typical materials. Since the same coefficients were used in compiling each set of tables, corrections made over the same temperature interval minimize errors arising from possible differences between the coefficient of the material under test and the standard coefficients. This effect becomes more important as temperatures diverge from the reference temperature.

NOTE 4—The hydrometer reading is obtained at a temperature appropriate to the physico-chemical characteristics of the material under test. This temperature is preferably close to the reference temperature, or when the value is used in conjunction with bulk oil measurements, within 3°C of the bulk temperature (see 5.3).

8.1.2 For crude petroleum, bring the sample close to the reference temperature or, if wax is present, to 9°C above its pour point or 3°C above its cloud point or WAT, whichever is higher.

NOTE 5—For crude petroleum an indication of the WAT can be found using IP 389, with the modification of using 50 µL ± 5 µL of sample. The precision of WAT for crude petroleum using this technique has not been determined.

9. Apparatus Verification or Certification

9.1 Hydrometers and thermometers shall be verified in accordance with the procedures in Annex A1.

10. Procedure

10.1 Bring the hydrometer cylinder and thermometer to within approximately 5°C of the test temperature.

10.2 Transfer the sample to the clean, temperature-stabilized hydrometer cylinder without splashing, to avoid the formation of air bubbles, and minimize evaporation of the lower boiling constituents of more volatile samples.

NOTE 6—**Warning:** Extremely flammable. Vapors may cause flash fire!

10.3 Transfer highly volatile samples by siphoning or water displacement.

NOTE 7—**Warning:** Siphoning by mouth could result in ingestion of sample!

10.3.1 Samples containing alcohol or other water-soluble materials should be placed into the cylinder by siphoning.

10.4 Remove any air bubbles formed after they have collected on the surface of the test portion, by touching them with a piece of clean filter paper before inserting the hydrometer.

10.5 Place the cylinder containing the test portion in a vertical position in a location free from air currents and where the temperature of the surrounding medium does not change more than 2°C during the time taken to complete the test. When the temperature of the test portion differs by more than 2°C from ambient, use a constant temperature bath to maintain an even temperature throughout the test duration.

10.6 Insert the appropriate thermometer or temperature measurement device and stir the test portion with a stirring rod, using a combination of vertical and rotational motions to ensure uniform temperature and density throughout the hydrometer cylinder. Record the temperature of the sample to the nearest 0.1°C and remove the thermometer/temperature measuring device and stirring rod from the hydrometer cylinder.

NOTE 8—If a liquid-in-glass thermometer is used, this is commonly used as the stirring rod.

10.7 Lower the appropriate hydrometer into the liquid and release when in a position of equilibrium, taking care to avoid wetting the stem above the level at which it floats freely. For low viscosity transparent or translucent liquids observe the meniscus shape when the hydrometer is pressed below the point of equilibrium about 1 to 2 mm and allowed to return to equilibrium. If the meniscus changes, clean the hydrometer stem and repeat until the meniscus shape remains constant.

10.8 For opaque viscous liquids, allow the hydrometer to settle slowly into the liquid.

10.9 For low viscosity transparent or translucent liquids depress the hydrometer about two scale divisions into the liquid, and then release it, imparting a slight spin to the hydrometer on release to assist in bringing it to rest floating freely from the walls of the hydrometer cylinder. Ensure that the remainder of the hydrometer stem, which is above the liquid level, is not wetted as liquid on the stem affects the reading obtained.

10.10 Allow sufficient time for the hydrometer to come to rest, and for all air bubbles to come to the surface. Remove any air bubbles before taking a reading (see 10.4).

10.11 If the hydrometer cylinder is made of plastic, dissipate any static charges by wiping the outside with a damp cloth.

NOTE 9—**Caution:** Static charges often build up on plastic cylinders and may prevent the hydrometer from floating freely.

10.12 When the hydrometer has come to rest floating freely away from the walls of the cylinder, read the hydrometer scale reading to the nearest one-fifth of a full scale division in accordance with 10.12.1 or 10.12.2.

10.12.1 For transparent liquids, record the hydrometer reading as the point on the hydrometer scale at which the principal surface of the liquid cuts the scale by placing the eye slightly below the level of the liquid and slowly raising it until the surface, first seen as a distorted ellipse, appears to become a straight line cutting the hydrometer scale (see Fig. 1).

10.12.2 For opaque liquids record the hydrometer reading at the point on the hydrometer scale to which the sample rises, by observing with the eye slightly above the plane of the surface of the liquid (see Fig. 2).

NOTE 10—When testing opaque liquids using a metal hydrometer cylinder, accurate readings of the hydrometer scale can only be ensured if the liquid surface is within 5 mm of the top of the cylinder.

10.13 Immediately after recording the hydrometer scale reading, carefully lift the hydrometer out of the liquid, insert the thermometer or temperature measurement device and stir the test portion vertically with the stirring rod. Record the temperature of the test portion to the nearest 0.1°C. If this temperature differs from the previous reading (10.6) by more than 0.5°C, repeat the hydrometer observations and thermometer observations until the temperature becomes stable within 0.5°C. If a stable temperature cannot be obtained, place the hydrometer cylinder in a constant temperature bath and repeat the procedure from 10.5.

10.14 If the test temperature is higher than 38°C, allow all hydrometers of the lead shot-in-wax type to drain and cool in a vertical position.

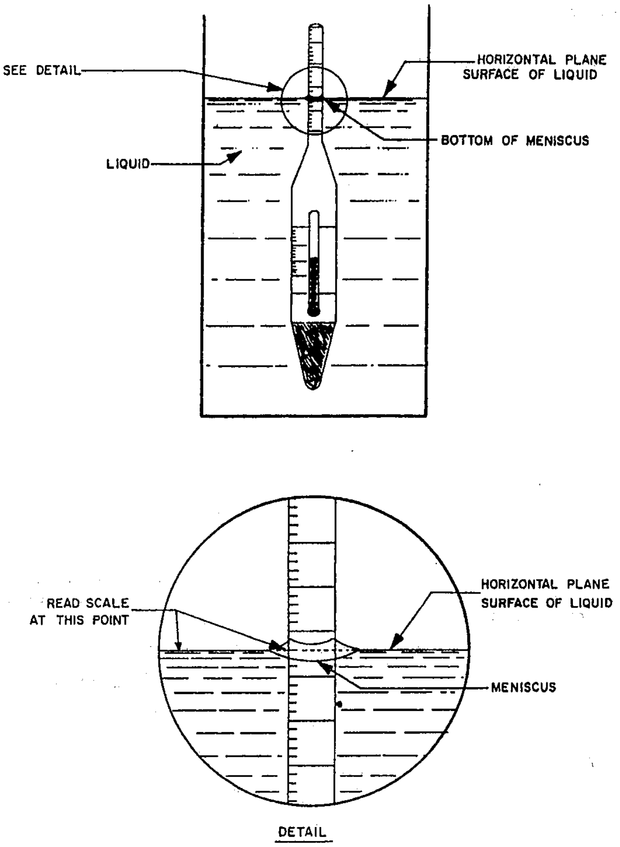


FIG. 1 Hydrometer Scale Reading for Transparent Liquids

11. Calculation

11.1 Apply any relevant thermometer corrections to the temperature reading observed in 10.6 and 10.13 and record the average of those two temperatures to the nearest 0.1°C.

11.2 For opaque samples, apply the relevant meniscus correction given in Table 1 to the observed hydrometer reading (10.12.2) as hydrometers are calibrated to be read at the principal surface of the liquid.

NOTE 11—The meniscus correction for a particular hydrometer in use is determined by observing the maximum height above the principal surface of the liquid to which liquid rises on the hydrometer scale when the hydrometer in question is immersed in a transparent liquid having a surface tension similar to that of the sample under test. For hydrometers specified in this test method, the corrections in Table 1 are approximate.

11.3 Apply any hydrometer correction to the observed reading and record the corrected hydrometer scale reading to the nearest 0.1 kg/m^3 in density, 0.0001 g/mL, kg/L or relative density, or 0.1° API.

11.4 If the hydrometer has been calibrated at a temperature other than the reference temperature, use the equation below to correct the hydrometer scale reading:

$$\rho r = \frac{\rho t}{1 - [23 \times 10^{-6}\ (t - r) - 2 \times 10^{-8}\ (t - r)^2]} \qquad (2)$$

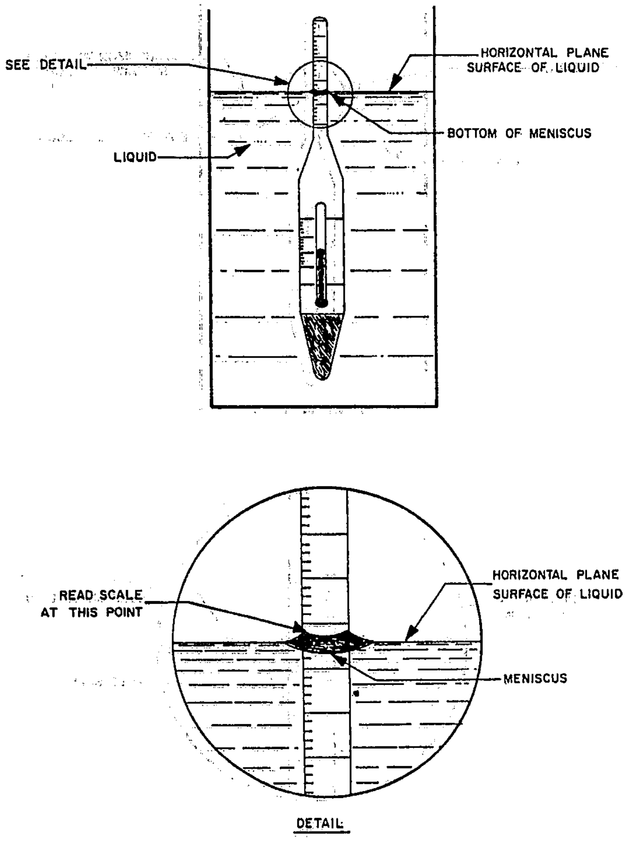


FIG. 2 Hydrometer Scale Reading for Opaque Fluids

where:

ρ_r = hydrometer reading at the reference temperature, r °C, and

ρ_t = hydrometer reading on the hydrometer scale whose reference temperature is t °C.

11.5 Convert the corrected hydrometer scale reading to density, relative density or API gravity using the appropriate parts of the Petroleum Measurement Tables in Guide D 1250 according to the nature of the materials under test. Table 3 gives some examples of relevant table numbers in the Petroleum Measurement Tables.

11.5.1 The strictly correct procedure for the conversion is to use the computer implementation procedures contained in the Petroleum Measurement Tables and not the printed tables. If the printed tables are used, ensure that all errata discovered since original publication have been included in the version used. The tables include corrections for soda-lime glass expansion and contraction of the hydrometer over the temperature range, and thus the observed hydrometer reading is added directly after correction (11.2-11.4) as necessary.

TABLE 3 Example PMT Table Numbers

Material	Density at 15°C kg/m³	Density at 20°C kg/m³	Relative Density at 60/60°F	°API
Crude petroleum	53A	59A	23A	5A
Petroleum products	53B	59B	23B	5B
Lubricating oils	53D	59D	–	5D

11.5.2 To convert densities expressed in kg/m^3 to densities expressed in g/mL or kg/L, divide by 10^3.

11.5.3 To convert hydrometer readings from one unit to another, Tables 51 (density at 15°C), 21 (relative density at 60/60°F) or 3 (API gravity), contained in Guide D 1250, are appropriate.

12. Report

12.1 Report the final value as density, in kilograms per cubic metre, at the reference temperature, to the nearest 0.1 kg/m^3.

12.2 Report the final value as density, in kilograms per litre or grams per millilitre at the reference temperature, to the nearest 0.0001.

12.3 Report the final value as relative density, with no dimensions, at the two reference temperatures, to the nearest 0.0001.

12.4 Report the final value as API gravity to the nearest 0.1° API.

13. Precision and Bias

13.1 *Precision*—The precision of the method as determined by statistical examination of interlaboratory results is as follows:

13.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values in Table 4 only in one case in twenty.

13.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty.

13.2 *Bias*—Bias for this test method has not been determined. However, there should be no bias from absolute measurements, if the calibration of the hydrometer and the thermometer is traceable to International Standards, such as supplied by the National Institute of Standards and Technology.

14. Keywords

14.1 API gravity; crude petroleum; density; hydrometer; Petroleum Measurement Tables; petroleum products; relative density; specific gravity

TABLE 4 Precision Values

Product	Parameter	Temperature Range, °C (°F)	Units	Repeatability	Reproducibility
Transparent Low-viscosity Liquids	Density	−2 - 24.5 (29 - 76)	kg/m³	0.5	1.2
			kg/L or g/mL	0.0005	0.0012
	Relative density		none	0.0005	0.0012
	API gravity	(42 - 78)	°API	0.1	0.3
Opaque liquids	Density	−2 - 24.5 (29 - 76)	kg/m³	0.6	1.5
			kg/L or g/mL	0.0006	0.0015
	Relative density		none	0.0006	0.0015
	API gravity	(42 - 78)	°API	0.2	0.5

ANNEX

(Mandatory Information)

A1. APPARATUS

A1.1 Apparatus Verification and Certification

A1.1.1 *Hydrometers*, shall either be certified or verified. Verification shall be either by comparison with a certified hydrometer (see 6.1.1) or by the use of a certified reference material (CRM) specific to the reference temperature used.

A1.1.1.1 The hydrometer scale shall be correctly located within the hydrometer stem by reference to the datum mark. If the scale has moved, reject the hydrometer.

A1.1.2 *Thermometers*, shall be verified at intervals of no more than six months for conformance with specifications. Either comparison with a referenced temperature measurement system traceable to an international standard, or a determination of ice point, is suitable.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1335: Standard Test Method for Tuft Bind of Pile Floor Coverings

CFR Section(s): 24 CFR 200.945(a)(1)(ii)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1335 – 67
(Reapproved 1972)

American National Standard L14.221-1973 (R-1968)
Reaffirmed Aug. 10, 1973
By American National Standards Institute

Standard Method of Test for
TUFT BIND OF PILE FLOOR COVERINGS[1]

1. Scope

1.1 This method covers the determination of the force required to pull a tuft completely out of a cut pile floor covering or to pull one or both legs of a loop free from the backing of looped pile floor covering.

NOTE 1—For the determination of other properties of pile floor coverings refer to Methods D 418.

2. Applicable Documents

2.1 *ASTM Standards:*

D 76 Tensile Testing Machines for Textile Materials[2]

D 123 Definitions of Terms Relating to Textile Materials[2]

D 418 Testing Woven and Tufted Pile Floor Coverings[3]

D 1776 Conditioning Textiles and Textile Products for Testing[2]

3. Definitions

3.1 *tuft, n.*—the cut or uncut loops forming the face of a tufted or woven pile floor covering.

3.2 *tuft bind, n.*—the force required to pull a tuft from a cut pile floor covering or to pull free one leg of a loop from a looped pile floor covering.

3.2.1 The tuft bind force is generally expressed in pounds-force but see also 4.1.

3.3 *pile floor covering, n.*—a pile fabric intended for use as a floor covering. The pile may be in the form of cut loops or loops, or both. Both the cut loops and the loops may vary in height.

3.4 *looped pile floor covering, n.*—a pile floor covering in which the pile is composed of uncut loops only.

3.5 *cut pile floor covering, n.*—a pile floor covering in which the pile is composed of tufts in the form of cut loops.

3.6 For definitions of other textile terms used in this method, refer to Definitions D 123.

4. Summary of Method

4.1 The force required to pull a cut loop from a cut pile floor covering or to pull one or both legs of a loop from a looped pile floor covering is determined by means of a tensile testing machine. The required load or force is reported in pounds-force (lbf) or kilograms-force (kgf) or newtons (N).

5. Uses and Significance

5.1 The satisfactory performance of a pile floor covering depends to a considerable extent on the maintenance of its original appearance. In a cut pile floor covering an inadequate tuft bind may result in complete loss of pile in areas exposed to severe wear. In a looped pile floor covering with inadequate tuft bind the pile loops may be pulled out to form unsightly long tufts or occasionally hazardous loops.

5.2 The wide range of capacity of tensile testing machines is specified to cover the application of the method to intermediate products (without back coating) which might have a tuft bind of less than 1 lb and also to cover

---

[1] This method is under the jurisdiction of ASTM Committee D-13 on Textiles, and is the direct responsibility of Subcommittee D13.21 on Pile Floor Coverings.

Current edition effective Sept. 8, 1967. Originally issued 1954. Replaces D 1335 – 60 T.

[2] *1974 Annual Book of ASTM Standards*, Parts 32 and 33.

[3] *1974 Annual Book of ASTM Standards*, Part 32.

the use of the method when it is desired to determine that the tuft bind exceeds a specified value of 3 to 4 lb, for example, without taking the time to pull the tuft out completely.

5.3 In cases when a floor covering contains both cut and uncut pile, an equal number of each type of pile should be tested.

6. Apparatus

6.1 *Tensile Testing Machine*, conforming to Specifications D 76, with a capacity selected such that the force required to complete the test falls within 20 to 80 percent of full scale. Full-scale loads ranging from 1 to 25 lb (0.5 to 11 kg) are generally adequate. The testing machines must be operable at the specified rates; for constant-rate-of-traverse (CRT) and constant-rate-of-extension (CRE) types—12 ± 0.5 in. (305 ± 10 mm)/min. For constant-rate-of-load (CRL) type, the full load of the tester shall be applied in 20 s.

NOTE 2—The level of test results obtained with different types of testing machines is not always the same.

6.2 *Cylindrical Specimen Holder, Cut-Away Type*, consisting of a 6-in. (152-mm) length of 1.5-in. (38-mm) outside diameter tubing with a section 2 in. (51 mm) long having half of the tubing cut away. See Fig. 1. This specimen holder should be constructed in a manner that will permit clamping the test specimen in the nonmeasuring, pulling clamp of the tensile testing machine or replacement of the nonmeasuring clamp by the specimen holder.

6.3 *Tuft Clamp*, for use only with cut pile floor coverings, consisting of a tweezer-like clamp that can be used to grip a single tuft tightly enough to assure removal of the whole tuft from the fabric without slippage of the tuft in the clamp. Alternatively, a hemostat[4] can be used.

6.4 *Loop Hook*, for use only with looped pile floor coverings, consisting of a hook which can be readily passed through the loop and hooked under the top of the loop. The hook should be made of wire having a diameter of at least $\frac{1}{32}$ in. (0.8 mm) and should be so constructed that it will not cut the loop during the normal test procedure. The shank of the hook should be so constructed that it can be clamped in the measuring clamp or can replace the measuring clamp of the test machine.

NOTE 3—Because the tuft clamp or loop hook is attached to, or replaces, the usual measuring clamp of the test machine, it is necessary to compensate for the effect of the altered weight of the clamp to retain the previous calibration of the testing machine.

7. Sampling

7.1 Take a lot sample and a laboratory sample as directed in the applicable material specification or as agreed upon by the purchaser and the seller. In the absence of such a specification or prior agreement, select a sample representative of the roll or piece to be tested and of sufficient size so that five specimens each about 6 in. (150 mm) wide and about 8 in. (200 mm) long can be cut from it.

NOTE 4—If the pile floor covering is back coated, exercise care in handling the sample in order that the back coating is not broken or otherwise disturbed.

8. Conditioning

8.1 Bring the specimens to moisture equilibrium for testing in the standard atmosphere for testing textiles approaching equilibrium from the dry side. Determine that moisture equilibrium for testing has been attained as directed in Method D 1776.

9. Preparation of Apparatus

9.1 If required, replace the nonmeasuring clamp of the test machine with the specimen holder described in 6.2.

9.2 Replace the measuring clamp of the test machine with, or attach to the measuring clamp of the test machine, the tuft clamp described in 6.3 or the loop-hook described in 6.4, depending upon which is required for the type of pile floor covering under test (Note 3).

10. Procedure

10.1 Test the conditioned specimens in the standard atmosphere for testing textiles.

10.2 Adjust the constant-rate-of-traverse (CRT) or the constant-rate-of-extension (CRE) type testing machine to operate at a rate of 12 ± 0.5 in. (305 ± 10 mm)/min. Adjust the constant-rate-of-load (CRL) type tester so that the full load is applied in 20 s.

---

[4] Hemostats suitable for this purpose can be obtained from the Fisher Scientific Co., Catalog 65, Catalog No. 8-907, Forceps, Kelly Hemostatic.

10.3 *Cut Pile Floor Coverings:*

10.3.1 Mount the test specimen on the specimen holder with the rows of tufts at a right angle to the long axis of the holder in such a position that the tuft to be tested is approximately centered over the cut-away portion of the specimen holder. Adjust the tension so that the specimen presents an undistorted cylindrical surface over the cut-away section of the specimen holder. Locate the tuft to be pulled directly below the center of the pulling clamp or hook.

10.3.2 Select only one tuft for testing from any one row of tufts and allow at least 1 in. (26 mm) between any tuft tested and the edge of the specimen.

10.3.3 Using the tuft clamp grip one leg (side) of the original loop. Make certain that all fibers forming the tuft are securely gripped by the tuft clamp. Take care not to pinch, "break the back," or otherwise deform the carpet in the selection of, and attachment of the clamp to, the tuft under test.

10.3.4 Determine tuft bind in pounds-force or kilograms-force to the nearest 0.1 lbf or 50 gf required to pull out the tuft from the pile floor covering.

10.3.5 Repeat this procedure on two additional tufts from different rows of tufts.

10.4 *Looped Pile Floor Covering:*

10.4.1 Mount the test specimen on the specimen holder as described in 10.3.1.

10.4.2 Select a location where three adjacent loops are formed by the same end. Cut completely through the first and third loops and test the center loop (Note 5). See Fig. 2.

NOTE 5—If this procedure is not followed, a spurious value may be obtained if one or both ends of the loop under test is buried in the back construction for a number of construction unit repeats.

10.4.3 Insert the loop hook in the loop to be tested. Determine the pounds-force or kilograms-force to the nearest 0.1 lbf or 50 gf required to pull at least one leg of the loop from the pile floor covering.

10.4.4 Repeat this procedure on two additional loops from other rows of loops.

11. Calculations

11.1 Calculate the average force required for all specimens tested for a sample to the nearest 0.1 lbf (50 gf).

12. Report

12.1 State that the tests were performed as directed in ASTM Method D 1335. Describe the product tested and the method of sampling used.

12.2 Report the following information:

12.2.1 Average tuft bind in pounds-force or kilograms-force to the nearest 0.1 lbf or 50 gf value of the test results,

12.2.2 Number of specimens tested,

12.2.3 Type of floor covering tested, and

12.2.4 Type of tensile testing machine on which the tests were performed.

13. Precision and Accuracy

13.1 *Within-Laboratory Precision*—The within-laboratory precision, at the 95 percent probability level, of the average of three replicates is expected to be within ±7 percent of the average tuft bind.

13.2 *Between-Laboratory Precision*—The precision, at the 95 percent probability level, of the difference between two laboratories, each making three measurements, is expected to be within ±15 percent of the average tuft bind.

NOTE 6—These values of precision are based on interlaboratory testing by three laboratories on a series of samples that represent most styles of currently produced carpets.

13.3 *Accuracy*—No justifiable statement on the accuracy of Method D 1335 for the measurement of the tuft bind of pile floor coverings can be made since the true value of the property cannot be determined by an accepted referee method.

ASTM Logo Removed

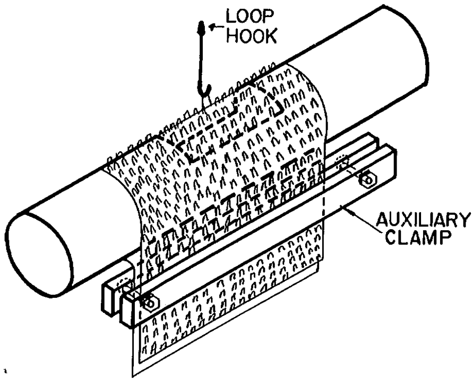


FIG. 1 Specimen Holder, Cut-Away Type.

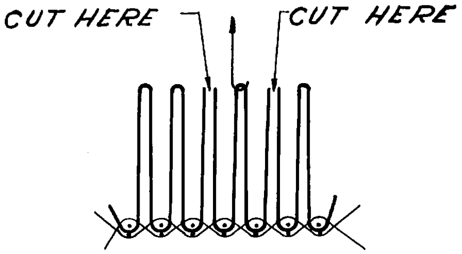


FIG. 2 Arrangement of Hook and Loops for Testing.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1412: Standard Test Method for Equilibrium Moisture of Coal at 96 to 97 Percent Relative Humidity and 30 Degrees Celsius

CFR Section(s): 30 CFR 870.19

Standards Body: American Society for Testing and Materials

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1412 – 93 (Reapproved 1997)

Standard Test Method for Equilibrium Moisture of Coal at 96 to 97 Percent Relative Humidity and 30°C[1]

This standard is issued under the fixed designation D 1412; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method[2] covers determination of the equilibrium moisture of coal in an atmosphere over a saturated solution of potassium sulfate at 30°C.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 121 Terminology of Coal and Coke[3]
D 388 Classification of Coals by Rank[3]
D 2013 Method of Preparing Coal Samples for Analysis[3]
D 2234 Test Methods for Collection of a Gross Sample of Coal[3]
D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke[3]
D 3302 Test Method for Total Moisture in Coal[3]
D 4596 Practice for Collection of Channel Samples of Coal in the Mine[3]

3. Significance and Use

3.1 This test method affords a means of estimating the bed moisture of either coal that is wet and shows visible surface moisture, or coal that may have lost some moisture. It may be used for estimating the surface, or extraneous moisture of wet coal, such moisture being the difference between the total moisture as determined by Test Method D 3302 and the equilibrium moisture.

3.2 When samples are collected in conformity with Classification D 388, the equilibrium moisture is considered to be equal to bed moisture with the exception of some low rank coals that yield equilibrium moisture values below bed moisture.

4. Apparatus

4.1 *Water Bath or Insulated Air Cabinet*—The bath or cabinet shall be of sufficient size to accommodate several vacuum-type desiccators, and shall be provided with a temperature regulator to maintain a uniform temperature of 30.0 ± 0.2°C.

4.2 *Moisture Oven*—The oven shall be so constructed as to have a uniform temperature in all parts and a minimum of air space. It may be of the type shown in the Apparatus section of Test Method D 3173. Provision shall be made for renewing the air (or, if desired, dry oxygen-free nitrogen for subbituminous and lignitic coals) in the oven at the rate of two times per minute, with the air dried by passing it through H_2SO_4 (sp gr 1.84).

4.3 *Mechanical Vacuum Pump.*

4.4 *Crusher,* laboratory, coffee-mill type.

4.5 *Sieve,* 8-in. (203-mm) diameter, with 1.18-mm (No. 16) openings.

4.6 *Shaking Machine.*

4.7 *Desiccator*—Small vacuum-type desiccator, 160 mm in diameter (see Fig. 1).

4.8 *Weighing Bottles,* glass, low-form, flat-bottom, cylindrical, 70 mm in diameter, with well-fitting covers.

4.9 *Filter Pump,* aspirator.

4.10 *Buchner-Type Funnel,* approximately 2½ in. (64 mm) in diameter.

5. Technical Hazards

5.1 In collecting, containing, handling, reducing, and dividing the gross moisture sample, all operations must be done expeditiously and in a manner which attempts to preserve the original sample moisture integrity.

5.2 If the gross sample is too wet to allow reduction and division, spread sample in a thin layer and expose to the air of the laboratory. Dry no more than necessary to enable satisfactory reduction and division of sample.

5.3 Take particular care not to overdry low rank coals, especially lignites. Drying will accelerate oxidation and can also result in shrinkage of pore size and volume which will affect the moisture holding capacity.

6. Collection of Gross Samples

6.1 Samples shall not be taken from outcrop, weathered, or oxidized coal.

---

[1] This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.21 on Methods of Analysis.

Current edition approved July 15, 1993. Published September 1993. Originally published as D 1412 – 56 T. Last previous edition D 1412 –89.

[2] For information concerning the experimental work on which this test method is based, see the following papers:

Stansfield, Edgar and Gilbart, K. C., "Moisture Determination for Coal Classification," *Transactions,* American Inst. of Mining and Metallurgical Engineers, Coal Division, TAMCA, Vol. 101, 1932, pp. 125–43.

Rees, O. W., Reed, F. H., and Land, G. W., "A Study of the Equilibration Method of Determining Moisture in Coal for Classification by Rank," *Report of Investigations No. 58,* Illinois State Geological Survey, ILGIA, 1939, pp. 34.

Krumin, Peter, "The Determination of Forms of Moisture in Coal," No. 195, Ohio State University, p 92, 1963.

Kreulen, D. J. W., "The Adsorption Water of Coal," *Chemische en Pharmaceutische Techniek (Dordrecht),* CHPHA, Vol. 7, 1951, pp. 23–4.

Selvig, W. A., and Ode, W. H., "Determination of Moisture-Holding Capacity (Bed Moisture) of Coal for Classification by Rank," *Report of Investigations No. 4968,* U.S. Bureau of Mines, XMBUA 1953.

[3] *Annual Book of ASTM Standards,* Vol 05.05.

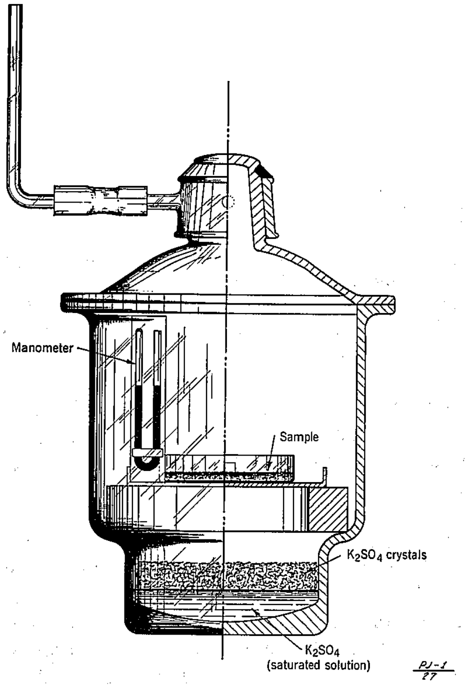


FIG. 1 Vacuum-Type Desiccator

6.1.1 *Mine Samples*—Take mine samples in accordance with Practice D 4596.

6.1.2 *Tipple or Shipment Samples*—Collect a representative gross sample of coal in accordance with Test Methods D 2234. If only the equilibrium moisture is desired, use the General Purpose Sampling Procedure. If the surface moisture of wet coal is to be determined, use the procedure for sampling the special total moisture subsample described in Test Methods D 2234.

7. Preparation of Laboratory Samples

7.1 Crush the gross sample to No. 4 (4.75-mm) sieve size in accordance with Method D 2013; however, it is important to also observe the technical hazards stated in Section 5 of this test method.

7.1.1 Divide sample in accordance with Method D 2013.

7.1.2 Rapidly stage-crush the divided sample to pass a No. 16 (1.18-mm) sieve by means of a coffee-mill type crusher. This stage crushing produces a minimum amount of fine material; however, it increases segregation so the crushed sample shall be thoroughly mixed.

7.1.3 Divide out the equilibration moisture subsample to be used for testing.

8. Procedure

8.1 Place 20 to 25 g of the crushed coal into a 250-mL Erlenmeyer flask and add 100 mL of recently boiled, cooled, distilled water (Note 1). Shake the flask mechanically for 30 min, and then place it in the constant-temperature bath for 3 h at 30°C. At the end of the wetting period, remove the excess water from the coal by filtering on a Büchner-type funnel approximately 2½ in. (64 mm) in diameter, using suction supplied by a water filter pump. Use a minimum amount of water to transfer the coal to the filter. After transfer of the coal, close the funnel with a rubber stopper fitted with a glass tube through which air saturated with water vapor is passed to prevent drying of the coal. Thoroughly mix the wet coal in the funnel with a spoon and place about 5.0 g in a uniform layer in a weighing bottle of known weight. Place the uncovered weighing bottle in the small vacuum-type desiccator containing a saturated solution of K_2SO_4 for maintaining the relative humidity of 96 to 97 %. An excess of crystalline K_2SO_4 shall extend above the solution level. Evacuate the desiccator to an absolute pressure equivalent to about 30 mm Hg by means of a mechanical vacuum pump, and then totally immerse in a constant-temperature water bath or place in an insulated air cabinet, maintained at 30 ± 0.2°C for 48 h for all coals higher in rank than lignite. Lignite will require 72 h to reach equilibrium for practical purposes.

NOTE 1—Mine samples and certain coals that deteriorate when treated with water may be equilibrated directly without wetting, provided the samples are collected and prepared with a minimum loss of moisture. Unwetted coals should be equilibrated for varying periods of time, in units of 24 h, in order that equilibrium may be attained.

8.2 After equilibration of the coal, restore the pressure in the desiccator to atmospheric, with the desiccator still in the bath, by slowly admitting dry air for a period of not less than 15 min. Admit the air to the inlet tube of the desiccator after passing it through a train consisting first of a bubbler containing H_2SO_4 (sp gr 1.84), then a capillary tube with one end drawn out to a tip having a suitable bore for regulating the rate of air flow, and finally a coiled copper tube placed in the constant-temperature bath. Remove the desiccator from the bath and open immediately. Quickly close the weighing bottle, and weigh to the nearest 0.2 mg. Uncover the weighing bottle, place it in the moisture oven preheated to 105°C, and heat for 1½ h. Then remove the weighing bottle from the oven, cover, cool 30 min over H_2SO_4 (sp gr 1.84) in a desiccator, and weigh.

9. Report

9.1 Report the equilibrium or bed moisture to the nearest 0.1 % as the percentage loss in weight of the equilibrated coal.

10. Precision and Bias

10.1 *Reproducibility*—The permissible differences between two or more determinations shall not exceed the following values:

Equilibrium Moisture, %	Permissible Same Laboratory	Differences Different Laboratories
Under 5	0.3	0.5
5 to 15	0.5	1.0
Over 15	1.0	1.5

10.2 *Bias*—Certified standards or absolute methods are not available for this test, therefore bias of results cannot be determined.

APPENDIX

(Nonmandatory Information)

X1. PRACTICE FOR COMPARING THE RELATIONSHIP BETWEEN INHERENT AND EQUILIBRIUM MOISTURE

X1.1 The purpose of the equilibrium moisture test is to provide an estimate of the inherent (bed) moisture. However, evidence has shown that equilibrium moisture results on many low rank coals, including most lignite coal, are often lower than inherent moisture. The procedure described in this appendix can be used where there is a question about the applicability of the equilibrium moisture result as an estimator of inherent moisture. The method is straightforward, and has proven effective in many situations for examining this moisture relationship.

X1.2 Special coal samples, collected at their inherent moisture level, are analyzed for both total (inherent) and equilibrium moisture. The results are then compared to see if differences exist between the two moisture parameters, and the end user(s) can then determine whether such differences have any practical significance. This procedure does not directly yield inherent moisture values for an entire coal seam or mine, because the samples are not necessarily representative of the full seam as would be the case for face channel samples (Practice D 4596). Nevertheless, the procedure does provide a tool for evaluating the relationship between inherent and equilibrium moisture for a given area.[4]

X1.3 The most critical step in evaluating this relationship is the collection of samples containing their full complement of inherent moisture. Occasionally, it may be difficult to obtain a channel sample that contains no surface moisture. Also, obtaining a channel sample from thick coal seams, such as those in the Western U.S., is generally impractical, especially from a safety standpoint. However, the collection of fresh, unfractured pieces of coal without visible surface moisture is usually feasible. Such samples are considered to contain only inherent moisture (Terminology D 121, Classification D 388).

NOTE X1—The collection of coal at its inherent moisture levels requires some degree of judgement and the sampler should have the necessary experience. For increased confidence, multiple comparisons are recommended to define the variability of the data.

X1.4 *Characteristics and Conditions of Sampling Locality*—Samples should be obtained from freshly exposed, unweathered mine faces. Avoid coal that exhibits any signs of moisture loss or weathering. There is no single test to determine the degree of weathering of coal under field conditions. However, when an obvious indication of weathering is observed, the sample should be obtained from a different locality or sampling postponed until suitable, fresh coal is available. Collecting a substandard sample simply because it was the best material available will not yield valid results.

NOTE X2—Obvious indicators of weathering include, but are not limited to: (*1*) any discoloration of broken coal surfaces or cleats, (*2*) presence of sulfate minerals resulting from the oxidation of pyrite, (*3*) presence of gypsum ($CaSO_4$) crystals, and (*4*) presence of dust, dried, crazed, or fragmented condition of the coal blocks resulting from moisture loss from the coal.

X1.5 Use heavy equipment such as a backhoe, front end loader, or continuous miner (with spray turned off) to expose a fresh, unweathered coal seam face. Immediately after exposure, collect pieces of coal either by picking from the face or from coal pulled from the face by the machine. The pieces must be solid and unfractured and must exhibit no visible surface moisture. The nominal size of the pieces should be 8 cm (3 in.) to 25 cm (10 in.). Larger pieces minimize any effects of surface drying that could reduce the inherent moisture. If there is any doubt that the coal contains its full complement of inherent moisture, select larger pieces (1 foot or more in diameter), and collect the sample material from the center portion of the larger pieces. Each sample should be comprised of multiple pieces totaling a minimum mass of 8 kg (17.6 lb). Where practical, collect pieces from various positions in the seam rather than concentrating on a particular horizon. Avoid layers or pieces that are excessively high in mineral matter content, especially clay.

X1.6 An alternative procedure for collecting the pieces is to obtain them from freshly shot coal at the toe of a coal face as the coal is being loaded out. Be especially alert to obtain pieces that were not lying on the surface of the pile of shot coal, and do try to obtain pieces that were well covered by other coal before outloading.

X1.7 Break each piece with a hammer to inspect for any internal moisture-filled fractures. Discard any pieces with visible surface moisture. Remove any fine particles adhering to the coal chunks by wiping or brushing the surfaces.

X1.8 Promptly put the pieces in a polyethylene bag at least 0.2 mm (4 mil) thick. Perform the operation in a manner that minimizes drying of the pieces. Samples should not be left sitting in direct sunlight during collection or transporting. It is recommended that the sample be double-bagged for added protection. Promptly ship the samples to the laboratory for analysis. Freezing conditions can affect the pore structure of the coal and therefore, samples should be protected from freezing during shipment to the laboratory.

X1.9 The samples should be processed immediately upon receipt by the laboratory. Inspect the sample bag for punctures occurring during transit which could cause moisture loss. Discard any samples where this has occurred. Record the weight of the coal and the bag.

X1.10 Most freshly-collected samples will exhibit visible moisture which has desorbed from the coal and condensed on the coal surfaces and the inside of the bag. To account for this desorbed inherent moisture, weigh the bag containing

---

[4] Luppens, J. A., and Hoeft, A. P., "Relationship Between Inherent and Equilibrium Moisture Contents in Coal by Rank," Journal Coal Quality, Vol 10, No. 4, 1991, pp. 133–144.

the sample. Then, open the bag and allow the coal and bag to air dry at room temperature for 15 minutes or just until all visible moisture has evaporated. Use caution to prevent overdrying, as that can result in shrinkage of the pore structure, thereby reducing the moisture holding capacity. Reweigh the coal and bag, and record this initial air-drying loss. Also, clean and weigh the bag(s) separately so that the air-drying loss can be calculated as a percentage of the coal weight.

X1.11 Following the reweighing, immediately reduce the sample to minus 4.75 mm (No. 4) using an enclosed crusher. Work rapidly to minimize moisture loss during this and subsequent handling steps. Use an enclosed riffle to divide the sample into at least two splits (A and B) with a minimum mass of 4000 g (8.8 lb) each.

X1.12 Analyze split A for total moisture using Test Method D 3302. Be certain to include the initial air-drying loss (section X1.8) in the calculation of the total moisture.

NOTE X3—Although only total moisture is required for comparison with equilibrium moisture, a proximate analysis (Test Method D 3172), a sulfur content analysis, and a calorific value analysis should also be performed. This will allow comparison of the quality of the sample to the quality of the coal from the entire seam or typically shipped from a mine.

X1.13 Analyze split B for equilibrium moisture using Test Method D 1412.

NOTE X4—To ensure that a true equilibrium condition has been reached, it is recommended that, at least for the first few comparisons, portions of split B be equilibrated for longer than the 2 to 3 days as specified in Test Method D 1412.

NOTE X5—As a quality assurance tool, it has been found useful to measure the moisture in the sample just prior to the start of equilibration.[4] To determine this "zero-day" moisture, prepare an extra weighing bottle (equilibration dish) containing 5 g of the sample and immediately measure the moisture using the same procedure as for the equilibrated sample. The zero-day result for wetted samples provides insight as to the amount of excess moisture remaining in the samples after the washing and filtration procedures. This can give an indication of the time required to reach equilibration or detect samples that have been overly dried during filtration that can lead to anomalously low equilibrium moisture values. For non-wetted samples, the zero-day result is useful in monitoring moisture loss during sample preparation as well as identifying suspect inherent (total) and equilibrium moisture data.

X1.14 The difference between inherent (total) moisture and equilibrium moisture (if any) is obtained by subtracting the total moisture value obtained in X1.10 from the equilibrium moisture value obtained in X1.11. The acceptable range of the difference at which equilibrium moisture may still be considered equivalent to inherent moisture depends on the specific purpose and circumstances. This final decision is left to the user(s) to agree upon.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1480: Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer

CFR Section(s): 40 CFR 75, Appendix D

Standards Body: American Society for Testing and Materials

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1480 – 93 (Reapproved 1997)

An American National Standard

Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer[1]

This standard is issued under the fixed designation D 1480; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes two procedures for the measurement of the density of materials which are fluid at the desired test temperature. Its application is restricted to liquids of vapor pressures below 600 mm Hg (80 kPa) and viscosities below 40 000 cSt (mm^2/s) at the test temperature. The method is designed for use at any temperature between 20 and 100°C. It can be used at higher temperatures; however, in this case the precision section does not apply.

NOTE 1—For the determination of density of materials which are fluid at normal temperatures, see Test Method D 941 or where greater precision is desired see Test Method D 1217.

1.2 This test method provides a calculation procedure for converting density to specific gravity.

1.3 The values stated in acceptable SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Note 1, Note 2, and Note 3.

2. Referenced Documents

2.1 *ASTM Standards:*

D 941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer[2]

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[2]

E 1 Specification for ASTM Thermometers[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *density*—the weight in a vacuum (that is, the mass) of a unit volume of the material at any given temperature.

3.1.2 *relative density (specific gravity)*—the ratio of the mass (weight in a vacuum) of a given volume of material at a temperature, t_1, to the mass of an equal volume of water at a reference temperature, t_2; or it is the ratio of the density of the material at t_1 to the density of water at t_2. When the reference temperature is 4°C (the temperature at which the relative density of water is unity), relative density (specific gravity) and density are numerically equal.

4. Summary of Test Method

4.1 The liquid sample is introduced into the pycnometer, equilibrated to the desired temperature, and weighed. The density or specific gravity is then calculated from this weight and the previously determined calibration factor, and a correction is applied for the buoyancy of air.

5. Significance and Use

5.1 Density is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and to assess the quality of crude oils.

5.2 Determination of the density or relative density of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperatures of 15°C.

5.3 The determination of densities at the elevated temperatures of 40 and 100°C is particularly useful in providing the data needed for the conversion of kinematic viscosities in centistokes (mm^2/s) to the corresponding dynamic viscosities in centipoises (mPa·s).

6. Apparatus

6.1 *Pycnometer,*[4] Bingham-type of 10-mL capacity (as shown in Fig. 1), constructed of heat-resistant[5] glass.

NOTE 2—Pycnometers having capacities of 2 to 25 mL are available but have not been cooperatively evaluated.

6.2 *Constant-Temperature Bath,* provided with suitable pycnometer holders and means for maintaining temperatures constant to ±0.01°C in the desired range. Water-glycerin mixtures can be used for temperatures up to 100°C.

---

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.04on Hydrocarbon Analysis.

Current edition approved Feb. 15, 1993. Published May 1993. Originally published as D 1480 – 57 T. Last previous edition D 1480 – 91.

In 1962, this test method was adopted as standard without revision.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] Available from Reliance Glass Co., 220 Gateway Rd., Bensonville, IL 60106-0825.

[5] Borosilicate glass has been found satisfactory for this purpose.

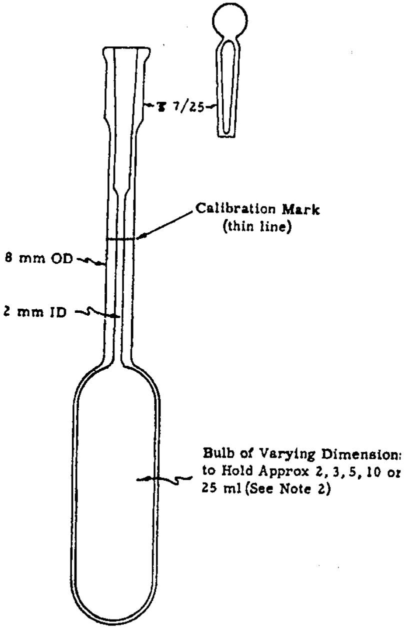
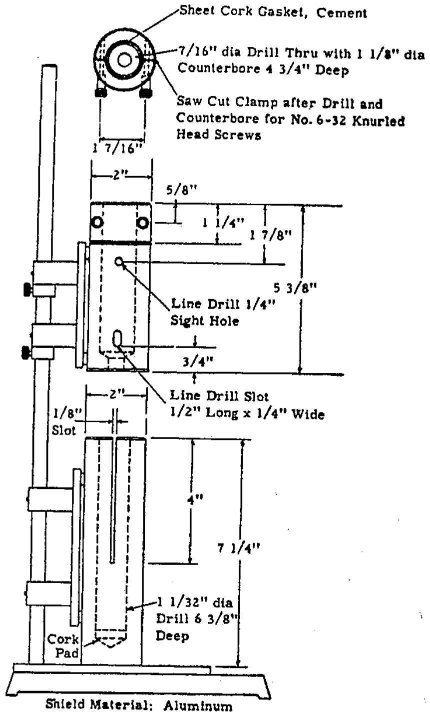


FIG. 1 Bingham-Type Pycnometer



Metric Equivalents

in.	mm	in.	mm	in.	mm	in.	mm
⅛	3.2	⅝	15.9	1¼	31.8	4	102
¼	6.4	¾	19.1	1 7⁄16	36.5	4¾	121
7⁄16	11.1	1 1⁄32	26.2	1⅞	47.6	5⅜	136
½	12.7	1⅛	28.6	2	50.8	6⅜	162
						7¼	184

NOTE 1—Cover shields with mica or insulating cement. Wind with No. 26 gage Chromel "A" wire: Upper block 60 in. (1.52 m) (25.4Ω), lower block (85 in. (2.16 m) (35.0Ω) wound vertically. Cover with insulating tape or insulating cement and connect heaters in series. Insulate shields from stand with ¼-in. Transite.

FIG. 2 Details of Thermal Shields for 30-mL Syringe and 25-mL Pycnometer

6.3 *Bath Thermometer*, graduated in 0.1°C subdivisions and standardized for the range of use to the nearest 0.01°C (ASTM Saybolt Viscosity Thermometers 17C to 22C, conforming to the requirements in Specification E 1, are recommended). For most hydrocarbons the density coefficient is about 0.0008 units/°C, and therefore an error of ±0.013°C would cause an error of ±0.00001 in density.

6.4 *Thermal Shields*, as shown in Fig. 2 , to hold the pycnometer and syringe during the filling procedure, constructed of two aluminum shells with suitably spaced viewing ports, the upper bored to hold a 30-mL hypodermic syringe and the lower bored to hold a 25-mL Bingham pycnometer. A winding of No. 26 Chromel" A" wire, insulated from the shields with mica, covered with insulating tape, and having resistances connected in series of 25 Ω on the upper shield and 35 Ω on the lower produces controlled heat to the shields by means of a variable transformer. A stand is necessary to support the shields in such a manner that the center of the wells may be aligned, and the upper shield raised 180 to 200 mm and swung through 45°.

6.5 *Hypodermic Syringes*, 2 to 30-mL capacity, of chemically resistant glass, equipped with a 170-mm, 16-gage (0.065 in.) filling needle made from stainless-steel tubing, as shown in Fig. 3 .

6.6 *Draw-off Needle*, made of stainless-steel tubing, as shown in Fig. 3.

6.7 *Solvent Cleaning Assembly*, as shown in Fig. 4 .

6.8 *Chromic Acid Cleaning Apparatus*, similar to that shown in Fig. 5 .

6.9 *Balance*, capable of reproducing weighings within 0.1 mg when carrying a load of 30 g. The balance shall be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled pycnometer) do not cause a significant change in the ratio of the balance arms. The same balance shall be used for all related weighings.

6.10 *Weights*, whose relative values are known to the nearest 0.05 mg or better. Use the same set of weights for the calibration of the pycnometer and the determination of densities.

7. Reagents and Materials

7.1 *Acetone*—(**Warning**—See Note 3).

NOTE 3—**Warning:** Extremely flammable. Use adequate ventilation.

7.2 *Isopentane*—(**Warning**—See Note 4).

NOTE 4—**Warning:** Extremely flammable. Avoid build up of vapors and remove all sources of ignition, especially non-explosion proof electrical apparatus.

7.3 *Chromic Acid (Potassium Dichromate/Conc. Sulfuric Acid)*—(**Warning**—See Note 5).

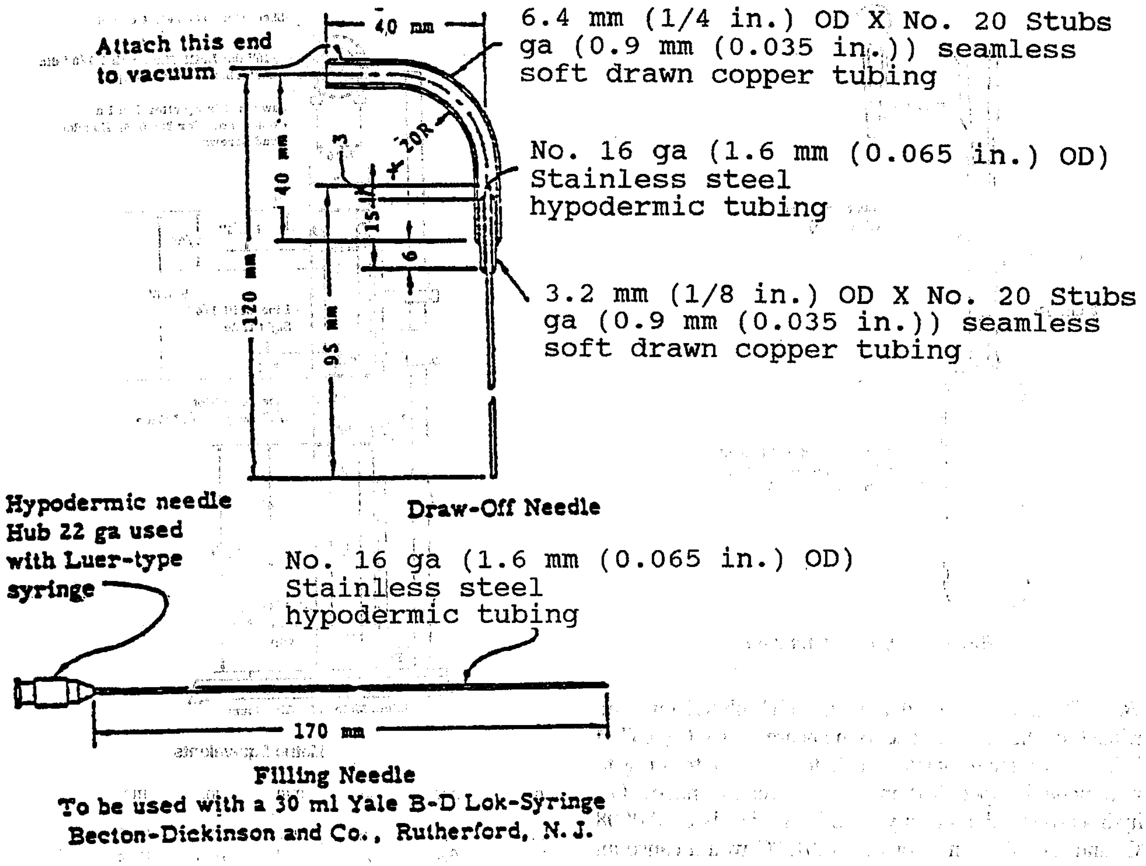


FIG. 3 Accessories for Bingham-Type Pycnometer

NOTE 5—**Warning:** Causes severe burns. A recognized carcinogen. Do not get in eyes, on skin or clothing.

8. Preparation of Apparatus

8.1 Clean the pycnometer thoroughly with hot chromic acid cleaning solution by means of the assembly shown in Fig. 5 (**Warning**—See Note 5). Chromic acid solution is the most effective cleansing agent. However, surfactant cleansing fluids have also been used successfully. Mount the apparatus firmly and connect the trap to the vacuum. Warm the necessary amount of cleaning acid in the beaker, place the pycnometer on the ground joint, and evacuate by opening the stopcock to vacuum. Fill the pycnometer with acid by turning the stopcock, and either repeat several times, or remove the filled pycnometer and allow it to stand for several hours at 50 to 60°C. Remove the acid from the pycnometer by evacuation, empty the acid from the trap, and flush the pycnometer with distilled water. Clean in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pycnometer may be cleaned between determinations by washing with a suitable solvent, rinsing with pure, dry acetone, followed by isopentane, and vacuum drying. (**Warning**—See Note 3 and Note 4.)

8.2 Transfer the pycnometer to the cleaner assembly shown in Fig. 4, with vacuum line and trap attached to the side tube as indicated. Place the pycnometer on the cleaner with the upper hypodermic needle extending upward into the pycnometer, and press the edge of the ground joint on the rubber stopper until the vacuum holds it in place. Draw out all the liquid or sample. Immerse the lower end of the hypodermic tube in a suitable solvent and draw 20 to 25 mL through the pycnometer. Leaving the pycnometer in place, draw air through it until it is dry. Clean the hypodermic syringe with the same apparatus.

9. Calibration of Pycnometers

9.1 Weigh the clean, dry pycnometer to 0.1 mg and record the weight.

NOTE 6—It is convenient to use the lightest of a set of pycnometers as a tare. For best results the treatment and environment of both pycnometer and tare should be identical for some time prior to weighing.

9.2 With a syringe of suitable size, transfer freshly boiled and cooled distilled water to the pycnometer through the filling needle (Note 9). Avoid trapping air bubbles in the bulb or capillary of the pycnometer, removing bubbles, as they form, with the syringe, when possible. Also remove any water above the calibration mark and dry the overflow chamber and capillary with a cotton-fiber pipe cleaner or cotton swab which has been moistened slightly with acetone. Do not touch the plunger of the syringe or hypodermic needle with fingers as

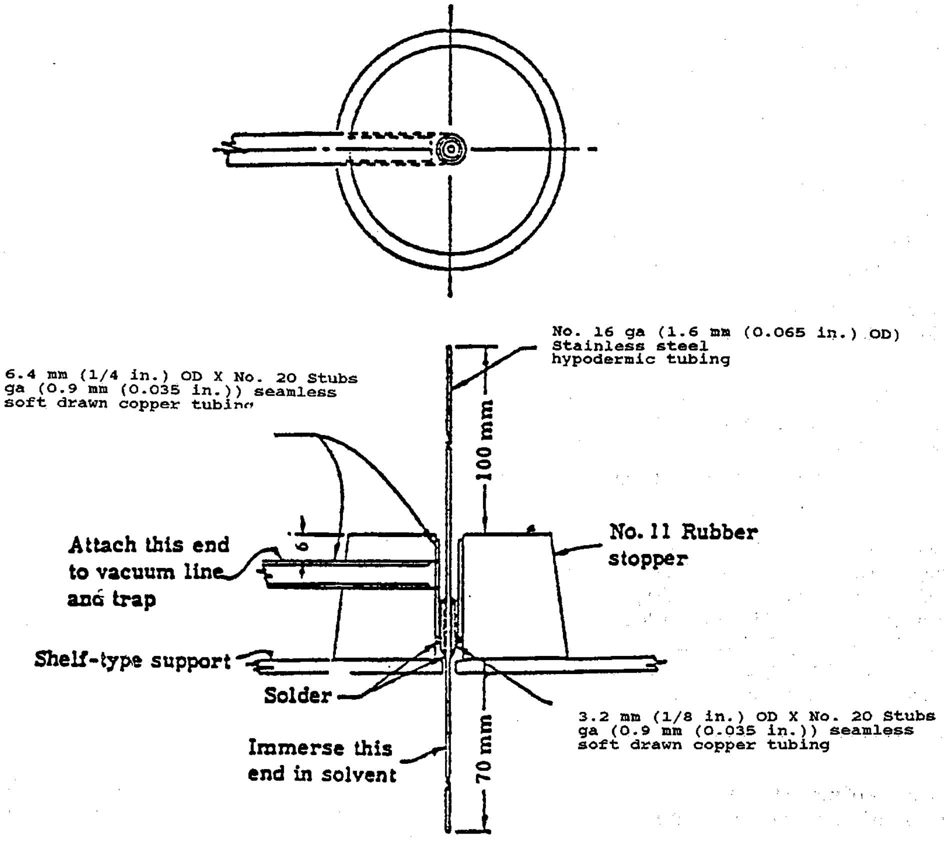


FIG. 4 Cleaner Assembly for Bingham-Type Pycnometer

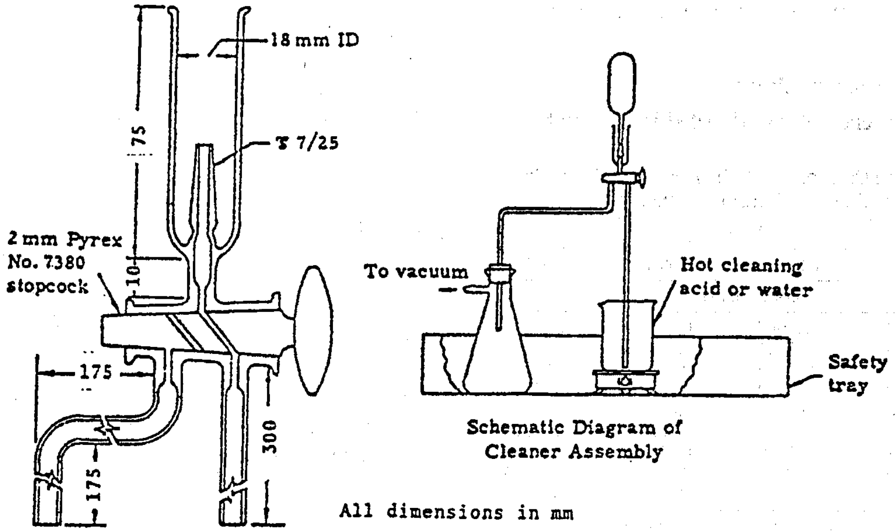


FIG. 5 All-Glass Pycnometer Cleaner Assembly for Use with Hot Chromic Acid Cleaning Solution

minute quantities of oil transferred this way would cause faulty drainage in the capillary neck of the pycnometer.

9.3 Close the pycnometer with the glass stopper and immerse it to a point above the calibration mark in the constant-temperature bath adjusted to a constancy of ±0.01°C at the desired temperature (Note 7). Periodically, or before the liquid expands into the overflow chamber, remove the stopper, raise the pycnometer sufficiently to expose the calibration mark to view, and readjust the liquid level to the mark by withdrawing liquid through the steel draw-off needle until expansion has stopped, indicating that the liquid has reached the temperature of the thermostat. To minimize errors caused by faulty drainage, do not allow the liquid to expand more than 10 mm above the calibration mark at any time. Allow the contents to equilibrate an additional 10 min and draw the level down exactly to the calibration line, avoiding parallax and using a magnifier, if necessary, to obtain good visibility. Remove any liquid adhering to the walls above the calibration mark, with the draw-off needle or pipe cleaner, depending upon the volatility of the sample. Portions in the overflow bulb can be removed with a cotton swab moistened with acetone.

NOTE 7—For temperatures above 80°C calculate the volume from the coefficient of expansion of the glass observed from calibrations made at 60, 70, and 80°C.

9.4 Replace the glass stopper, remove the pycnometer from the bath, wash the outside surface with acetone, and dry thoroughly with a chemically clean, lint-free, slightly damp cloth. Place the pycnometer in or near the balance case for 20 min and weigh to the nearest 0.1 mg.

NOTE 8—In atmospheres of low humidity (60 % or lower), drying the pycnometer by rubbing with a dry cotton cloth will induce static charges equivalent to a loss of about 1 mg in the weight of the pycnometer. This charge may not be completely dissipated in less than 30 min. The use of about 0.1 mg of radium bromide- or polonium-coated foil in the balance case, or maintaining the relative humidity at 60 percent or higher, aids in reducing weighing difficulties due to static charges.

9.5 Calculate the pycnometer calibration factor, F_t, from the equation:

$$F_t = (\text{density of water at } t°\text{C})/(\text{weight of water in pycnometer at } t°\text{C}) \quad (1)$$

See Table 2 for the density of water between 0 and 100°C.

9.6 Duplicate determinations should not show a variation greater than ±0.2 mg in the net weight of the water in the pycnometer.

10. Procedure for Viscous Liquids

10.1 Weigh the pycnometer as directed in Section 8.

TABLE 1 Vacuum Corrections to be Applied to Densities Observed in Air of Various Densities

Observed Density	Air Density			
	0.00116	0.00118	0.00120	0.00122
	Corrections to be Added			
0.60	0.00046	0.00047	0.00048	0.00049
0.65	0.00040	0.00041	0.00042	0.00042
0.70	0.00034	0.00035	0.00036	0.00036
0.75	0.00029	0.00029	0.00030	0.00030
0.80	0.00023	0.00024	0.00024	0.00024
0.85	0.00017	0.00018	0.00018	0.00018
0.90	0.00011	0.00012	0.00012	0.00012
0.95	0.00005	0.00006	0.00006	0.00006
1.00	0	0	0	0
	Corrections to be Subtracted			
1.05	0.00005	0.00006	0.00006	0.00006
1.10	0.00011	0.00012	0.00012	0.00012
1.15	0.00017	0.00018	0.00018	0.00018
1.20	0.00023	0.00024	0.00024	0.00024

NOTE 1—Interpolate linearly for intermediate sample densities.

NOTE 2—For air densities outside this table the vacuum correction shall be calculated from the equation $C = d_a[1 - (F_t W_t)]$, d_a being the density of the air in the balance case in grams per millilitre. See Section 10 of Test Method D 1217 for calculating the air density.

TABLE 2 Density of Water[A]

Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0	0.999840	21	0.997991	40	0.992212
3	0.999964	22	0.997769	45	0.990208
4	0.999972	23	0.997537	50	0.988030
5	0.999964	24	0.997295	55	0.985688
10	0.999699	25	0.997043	60	0.983191
15	0.999099	26	0.996782	65	0.980546
15.56	0.999012	27	0.996511	70	0.977759
16	0.998943	28	0.996231	75	0.974837
17	0.998774	29	0.995943	80	0.971785
18	0.998595	30	0.995645	85	0.968606
19	0.998404	35	0.994029	90	0.965305
20	0.998203	37.78	0.993042	100	0.958345

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Appendix G, *Standard Methods for Analysis of Petroleum and Related Products 1991*, Institute of Petroleum, London.

10.2 Warm, in an oven or convenient warming chamber, the pycnometer, syringe with needle, and sample to a convenient working temperature consistent with the fluidity and volatility of sample. Draw the requisite amount of sample into the syringe and immediately fill the warmed pycnometer taking care to avoid occluding air bubbles in the pycnometer bulb or capillary. Continue the addition of sample, withdrawing the filling needle gradually so that the tip remains immersed in the sample, until the sample has been added to a depth of 10 or 20 mm in the expansion chamber above the capillary, depending upon the amount of contraction expected.

10.3 Immerse the pycnometer bulb in the constant-temperature bath. As the sample contracts continue sample addition before the level recedes into the capillary or until a sufficient amount has been added to maintain the meniscus slightly above the calibration mark at the reference temperature. Allow to equilibrate to reference temperature.

NOTE 9—Equilibration time depends upon the viscosity and temperature of the sample at the time of filling. Usually this is three to four times that required for a fluid sample. A safe criterion is to allow 15 min more equilibration time after the meniscus remains stationary.

10.4 Remove excess sample with the 16-gage needle attached to a vacuum line, warming the needle if necessary. Swab the capillary above the calibration mark and the overflow chamber several times with a pipe cleaner or small cotton swab slightly moistened with a suitable solvent. Follow with a dry swab. Final adjustment to the mark may be done by picking out sample with a small probe, splinter, or wire.

10.5 Remove the pycnometer from the bath, wash the outer surface with a suitable solvent followed by acetone and dry thoroughly with a clean, lint-free, slightly damp cloth. Observe the same cleaning procedure as used in calibrating the pycnometer in the bath. Allow the pycnometer to come to room temperature and weigh to the nearest 0.1 mg.

11. Procedure for Melted Solids at High Temperature

11.1 Place the sample in a heat-resistant container and bring to a temperature 8 to 12°C above its melting point in an explosion-proof oven.

11.2 Insert the pycnometer, previously weighed to the nearest 0.1 mg, in the lower chamber of the thermal shield and lightly clamp the syringe in the upper chamber so that the filling needle is inside the pycnometer. Apply power to the shields until the temperature is 2 to 3°C above the melting point of the sample, then reduce the voltage until the shield temperature increases less than 0.5°C/min.

NOTE 10—In the absence of a thermal shield, an oven can be fitted with a rack to support the pycnometer and hypodermic, and the whole operation of charging the syringe and filling the pycnometer performed in the oven. Weights applied to the syringe plunger reduce the filling time. An internal light and glass door for the oven are aids in this procedure.

11.3 After thermal equilibrium of sample, pycnometer, and syringe has been established, raise the upper shield, swing to one side, and quickly charge the syringe.

11.4 Quickly wipe the needle, swing the syringe over, and lower into the pycnometer. Fill the pycnometer in the usual manner, as given in 10.2. Remove the syringe and needle and place the pycnometer in the bath for temperature equilibration. Remove excess sample with a thin strip of filter paper or heated draw-off needle, taking care not to remove sample below the calibration mark.

11.5 Close the pycnometer with the glass stopper and immerse it to a point above the calibration mark in the constant-temperature bath, adjusted to the desired temperature within ±0.01°C. Periodically, or before the liquid expands into the overflow chamber, remove the stopper, raise the pycnometer sufficiently to expose the calibration mark to view, and readjust the liquid level to the mark by withdrawing liquid with thin strips of filter paper. Continue in this manner until expansion has stopped, indicating that the liquid has reached the temperature of the bath. To minimize errors caused by faulty drainage, do not allow the liquid to expand more than 10 mm above the calibration mark at any time. Allow the contents to equilibrate an additional 10 min and draw the level down exactly to the top of the calibration line, avoiding parallax and using a magnifier, if necessary, to obtain good visibility. Remove any liquid adhering to the walls above the calibration mark with filter paper or a pipe cleaner, barely moistened with a suitable solvent if necessary.

11.6 Replace the glass stopper, and proceed as directed in 10.5.

12. Calculation

12.1 Calculate the density of the sample, corrected to vacuum, by the following equation:

$$\text{Density in vacuum } (d_t),\ \text{g/mL} = (F_t)(W_t) + C \qquad (2)$$

where:

F_t = calibration factor of the pycnometer at t°C,
W_t = weight of sample, g, in pycnometer at t°C, and
C = vacuum correction, obtained from Table 1.

12.2 Calculate the relative density (specific gravity) of the sample by dividing the density, as obtained in 12.1, by the density of water at the reference temperature obtained from Table 2.

13. Precision and Bias

13.1 The precision of the test method as obtained by statistical examination of interlaboratory test results is as follows:

13.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Pycnometer Volume, mL	Repeatability, g/mL
10	0.00005

13.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators, working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Pycnometer Volume, mL	Reproducibility, g/mL
10	0.00014

NOTE 11—If pycnometers of other than 10 mL in volume are used, or if the temperature of test exceeds 100°C, this precision statement may not apply.

13.1.3 *Bias*—The difference of results from the established value when compared to pure reference materials is not expected to be more than ±0.00014 g/mL. Specific bias has not been established by cooperative testing.

14. Keywords

14.1 density; gravity; pycnometer; relative density; specific gravity

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1481: Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer

CFR Section(s): 40 CFR 136.3(a) Table IC

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1481 – 93 (Reapproved 1997)

An American National Standard

Standard Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Lipkin Bicapillary Pycnometer[1]

This standard is issued under the fixed designation D 1481; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the density of oils more viscous than 15 cSt at 20°C (mm^2/s), and of viscous oils and melted waxes at elevated temperatures, but not at temperatures at which the sample would have a vapor pressure of 100 mm Hg (13 kPa) or above.

NOTE 1—To determine the densities of less viscous liquids at 20 or 25°C use Test Method D 941 or Test Method D 1217.

1.2 This test method provides a calculation procedure for converting density to relative density (specific gravity).

1.3 The values stated in acceptable SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer[2]

D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[2]

D 1250 Guide for Petroleum Measurement Tables[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *density*—the weight in a vacuum (that is, the mass) of a unit volume of the material at any given temperature.

3.1.2 *relative density (specific gravity)*—the ratio of the mass (weight in a vacuum) of a given volume of material at a temperature, t_1, to the mass of an equal volume of water at a reference temperature, t_2; or the ratio of the density of the material at t_1 to the density of water at t_2.

4. Summary of Test Method[3]

4.1 The liquid is drawn into the bicapillary pycnometer through the removable siphon arm and adjusted to volume at the temperature of test, in such a manner that there is practically no drainage in the unfilled tubing. After equilibration at the test temperature, liquid levels are read, and the pycnometer is removed from the thermostated bath, cooled to room temperature, and weighed.

4.2 Density or relative density (specific gravity), as desired, is then calculated from the volume at the test temperature and the weight of the sample. The effect of air buoyancy is included in the calculations.

5. Significance and Use

5.1 Density is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and to access the quality of crude oils.

5.2 Determination of the density or relative density of petroleum and its products is necessary for the conversion of measured volumes to volumes at the standard temperatures of 15°C.

5.3 The determination of densities at the elevated temperatures of 40 and 100°C is particularly useful in providing the data needed for the conversion of kinematic viscosities in centistokes (mm^2/s) to the corresponding dynamic viscosities in centipoises (mPa·s).

6. Apparatus

6.1 *Pycnometer*[4]—A side-arm type of pycnometer conforming to the dimensions given in Fig. 1 and made of borosilicate glass. The weight shall not exceed 35 g without the side arm.

6.2 *Rack*—A rack to use in filling the pycnometer (see Fig. 2).

6.3 *Constant-Temperature Oven*—An oven for use in filling the pycnometer. Any oven capable of holding the filling rack, and of maintaining a temperature of approximately 100°C, can be used.

6.4 *Constant-Temperature Bath*—A mixture of water and

---

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.04on Hydrocarbon Analysis.

Current edition approved Feb. 15, 1993. Published June 1993. Originally published as D 1481 – 57 T. Last previous edition D 1481 – 91.

[2] *Annual book of ASTM Standards*, Vol 05.01.

[3] For a more complete discussion of this procedure see Lipkin, M. R., Mills, I. W., Martin, C. C., and Harvey, W. T., *Analytical Chemistry*, ANCHA, Vol 21, 1949, p. 504.

[4] Pycnometers available from Reliance Glass Co., 220 Gateway Rd., Bensenville, IL 60106-0825 have been found satisfactory.

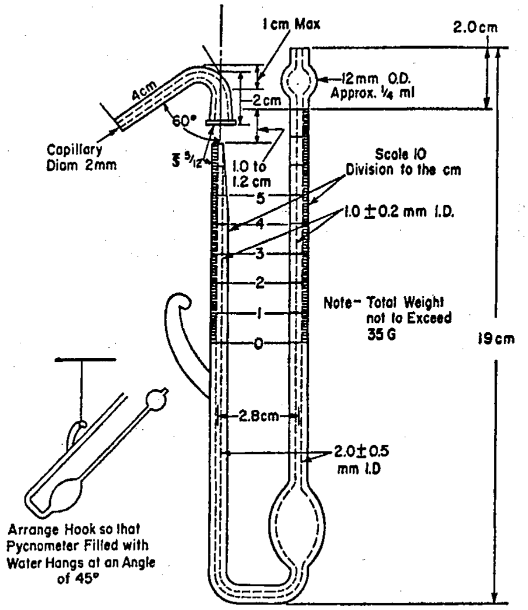


FIG. 1 Pycnometer

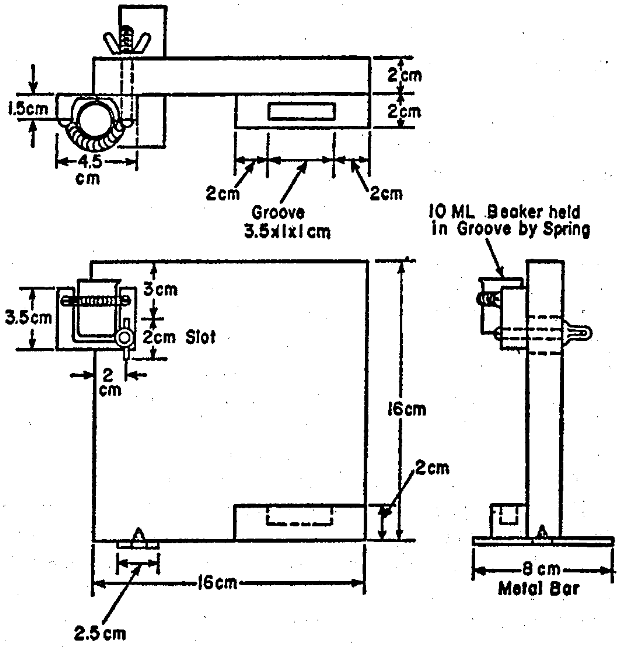


FIG. 2 Rack for Filling Pycnometer

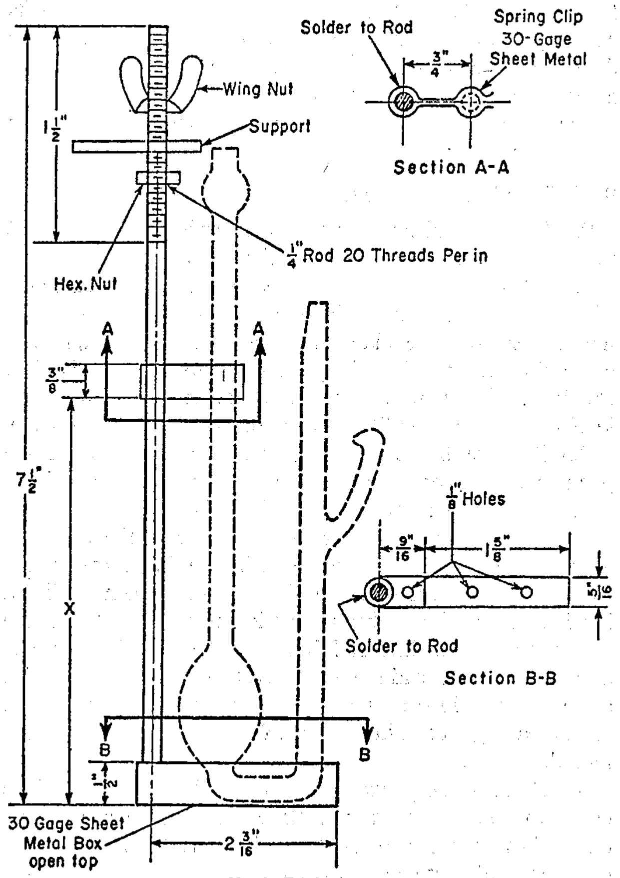
glycerin, or oil bath having a depth of at least 305 mm (12 in.) and provided with heating, stirring, and thermostating devices adequate to maintain desired temperatures in the range from 20 to 100°C with an accuracy of ±0.01°C.

6.5 *Bath Thermometers*—Thermometers graduated in 0.1°C subdivisions and standardized for the range of use to the nearest 0.01°C (ASTM Saybolt Viscosity Thermometers 17C to 22C are recommended). For most hydrocarbons, the density coefficient is about 0.0008 units/°C, and therefore a temperature error of ±0.013°C would cause an error of ±0.000 01 in density.

6.6 *Pycnometer Holder*—A holder, as shown in Fig. 3 , is recommended for supporting the pycnometer in the bath. A single clamp device may be used.

6.7 *Balance*—A balance able to reproduce weighings within 0.1 mg when carrying a load of 35 g or less on each pan. The balance shall be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled pycnometer) do not cause a significant change in the ratio of the balance arms. Otherwise, weighings shall be made by the substitution method in which the calibrated weights and pycnometer are alternatively weighed on the same balance pan. The same balance shall be used for all related weighings.

6.8 *Weights*—Weights shall be used whose relative values are known to the nearest 0.05 mg or better. The same set of weights shall be used for the calibration of the pycnometer and the determination of the densities, or the sets of weights shall be calibrated relative to each other.



Metric Equivalents

in.	mm	in.	mm	in.	mm
½	3.2	½	12.7	1⅝	41.3
¼	6.4	9⁄16	14.3	2 3⁄16	55.7
5⁄16	7.9	¾	19.1	7½	191
⅜	9.5	1½	38.1		

FIG. 3 Pycnometer Holder

7. Reagents and Materials

7.1 *Acetone*—**Warning**—Extremely flammable. Use adequate ventilation.

7.2 *Isopentane*—**Warning**—Extremely flammable. Avoid buildup of vapors and remove all sources of ignition, especially nonexplosion-proof electrical apparatus.

7.3 *Chromic Acid (Potassium Dichromate/Conc. Sulfuric Acid)*—**Warning**—Causes severe burns. A recognized carcinogen. Do not get in eyes, on skin or clothing.

7.4 *Benzene*—**Warning**—Poison. Known carcinogen. Extremely flammable. Avoid contact with skin and eyes.

8. Preparation of Apparatus

8.1 Thoroughly clean the pycnometer and side arm with hot chromic acid cleaning solution (**Warning**—See 7.4.) Chromic acid solution is the most effective cleaning agent. However, surfactant cleaning fluids have also been used successfully. Rinse well with distilled water, and dry at 105 to 110°C for at least 1 h, preferably with a slow current of filtered air passing through the pycnometer. Cleaning shall be done in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pycnometer may be cleaned between determinations by washing with a suitable solvent, such as isopentane or benzene, and vacuum drying. If acetone is used as the wash liquid, the pycnometer should then be rinsed with isopentane or benzene.

9. Calibration of Pycnometer

9.1 Weigh the clean, dry pycnometer (without the side arm) to the nearest 0.1 mg, and record the weight.

9.2 Fill the pycnometer with freshly boiled distilled water. This may be conveniently done by placing the pycnometer in the holder with the side arm dipping into a sample cup containing water. Allow the pycnometer to fill by siphoning. Break the siphon by removing the side arm when the liquid level in the bulb arm of the pycnometer reaches 6 on the scale.

9.3 Remove the side arm which was used to fill the pycnometer and remove excess liquid from the capillary tip by wiping with a small piece of absorbent paper.

9.4 Place the pycnometer in the holder in the constant-temperature bath at temperature *t* with the liquid level in the capillaries below the liquid level in the bath. When the liquid level has reached equilibrium (not less than 15 min), read the scale to the nearest 0.2 small division at the liquid level in each arm. After 5 min, read the liquid level again. If the sum of the scale readings in each reading differs by more than ±0.04, repeat readings at 5-min intervals. When readings are constant, record.

9.5 Remove the pycnometer from the bath and allow it to come to room temperature. Rinse the outer surface with distilled water, with acetone, then with redistilled benzene, and dry thoroughly with a chemically clean lint-free cloth, slightly damp with water. Allow to stand a few minutes, and then weigh to nearest 0.1 mg.

NOTE 2—In atmospheres of low humidity (60 % or lower), drying the pycnometer by rubbing with dry cotton cloth will induce static charges equivalent to a loss of about 1 mg or more in the weight of the pycnometer. This charge may not be completely dissipated in less than ½ h and can be detected by touching the pycnometer to the wire hook on the balance and then drawing it away slowly. If the pycnometer exhibits an attraction for the wire hook, it may be considered to have a static charge.

9.6 Repeat the above, but break the siphon when water has reached the 3 mark in the bulb arm, and in the next experiment, at the 0 mark in the bulb arm. Obtain the apparent volume for each filling by dividing the weight of water held by the pycnometer in each experiment by the density of water at the calibration temperature *t*. Calibration shall be made at 20, 40, and 50°C. Prepare a calibration curve for 20°C by plotting the sum of the two scale readings *versus* the apparent volume at 20°C. If the curve is not a straight line, and future checks do not correct it, discard the pycnometer. The line shall not be more than 0.0002 mL/unit from any one determined point.

9.7 Corresponding calibration curves shall be made for 40 and 50°C. These calibration curves are checked using the following equation:

$$V_2 = V_1(1 + ct) \tag{1}$$

where:

V_2 = apparent volume at test temperature,
V_1 = apparent volume at 20°C, and
c = cubical coefficient of expansion of borosilicate glass (9.9×10^{-6}/°C).

The calculated and determined curves at 40 and 50°C should check to within ±0.0002 mL/unit at all points. The calibration curves for higher temperatures shall be obtained by calculation.

10. Procedure

10.1 Weigh the clean, dry pycnometer, without the side arm, to 0.1 mg and record the weight.

10.2 Place a 10-mL sample beaker in the wooden rack (Fig. 2). Before attaching the side arm to the pycnometer, drain a few drops of sample through the side arm to wet the inside surface and reduce the chance of trapping air bubbles in the capillary during the filling operation. Place the side arm on the pycnometer, and place the assembly on the rack with the side arm dipping into the sample beaker as shown in Fig. 4.

10.3 In filling the pycnometer with very viscous oils or high-melting waxes, place the whole filling assembly in a hot-air oven to facilitate filling. An oven at approximately 100°C is usually hot enough for this purpose.

10.4 Apply gentle suction to the bulb arm of the pycnometer to start the siphoning action. The suction must be gentle to avoid the formation of bubbles. After siphoning is started, allow filling by siphoning to continue until the liquid level in the bulb arm ceases to rise. Then remove the pycnometer from the rack and place in the thermostated bath, in the same tilted position, until the oil ceases to contract. At this point, place the pycnometer in an upright position, and allow the liquid level in the bulb arm to reach the upper portion of the calibrated capillary, but not above 6.4. Stop siphoning by removing the side arm.

NOTE 3—With viscous oils, it will reduce drainage errors to fill to the 6.0 to 6.4 mark, and it may be necessary to apply a little suction to the long arm during cooling to prevent the meniscus in the bulb arm from falling. Maintain the meniscus at about the same level in the long arm throughout the whole determination.

10.5 After removing the side-arm cap from the short arm of

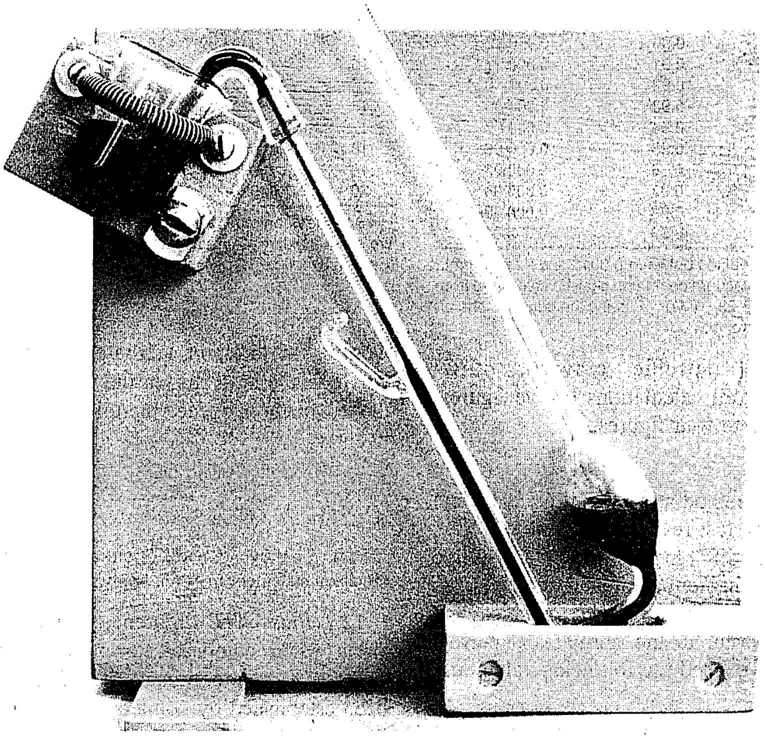


FIG. 4 Pycnometer Filling Assembly

the pycnometer, wipe the tip and ground joint of the pycnometer, and adjust it to an upright position in the thermostated bath. The bath liquid level shall be above the 6 mark on the pycnometer and below the ground glass tip of the pycnometer.

10.6 Allow 15 min for equilibrium to be obtained. After the stated 15-min time for coming to equilibrium, read the meniscus levels in both arms of the pycnometer to the nearest 0.2 of the smallest scale division. Wait 5 min and check readings. If the sum of the readings at the two different times do not agree to within ±0.04, repeat at 5-min intervals until checks are obtained. Record the sum of these readings and also record the corresponding apparent volume from the calibration curve for the same temperature.

NOTE 4—The final level of oil in the pycnometer should not be more than 5 mm below the tip of the ground glass end of the pycnometer, and the level in the long (bulb) side of the pycnometer should be no lower than it has been at any time during the procedure. With these precautions, drainage error (which is important with very viscous samples) is entirely eliminated.

10.7 Remove the pycnometer from the bath and tilt it so that the liquid moves down in the short arm and up in the bulb arm. Clean and dry the outside of the pycnometer as described in the calibration procedure (Section 9). Allow to come to balance room temperature. Weigh to the nearest 0.1 mg. Subtract the weight of empty pycnometer, without the side arm, to get the weight of sample.

11. Calculation

11.1 Calculate the density of the sample, corrected to vacuum, by the following equation:

$$\text{Density in vacuum, } d_t\text{, g/mL} = (W/V) + C \qquad (2)$$

where:

W = weight of sample in air, g;

V = apparent volume, mL; and

C = vacuum correction, obtained from Table 1.

11.2 Calculate the relative density (specific gravity) of the sample at t_1/t_2 by dividing the density, as calculated in 10.1, by the density of water at the reference temperature, t_2, as obtained from Table 2 . Relative density (specific gravity) at t_1/15.56°C (t/60°F where t is expressed in degrees Fahrenheit) can be changed to the conventional 15.56/15.56°C (60/60°F) relative density (specific gravity) by use of the appropriate Table 23 in Guide D 1250, provided that the glass expansion factor has been excluded.

11.3 In reporting density, give the test temperature and the units (for example, density at 40°C = x.xxxx g/mL). In reporting relative density (specific gravity), give both the test temperature and the reference temperature, but no units (for

TABLE 1 Vacuum Corrections

	Correction[A] Plus		Correction[A] Plus
0.70	0.000 36	0.85	0.000 18
0.71	0.000 35	0.86	0.000 17
0.72	0.000 33	0.87	0.000 16
0.73	0.000 32	0.88	0.000 14
0.74	0.000 31	0.89	0.000 13
0.75	0.000 30	0.90	0.000 12
0.76	0.000 29	0.91	0.000 11
0.77	0.000 28	0.92	0.000 10
0.78	0.000 26	0.93	0.000 09
0.79	0.000 25	0.94	0.000 07
0.80	0.000 24	0.95	0.000 06
0.81	0.000 23	0.96	0.000 05
0.82	0.000 22	0.97	0.000 04
0.83	0.000 20	0.98	0.000 03
0.84	0.000 19	0.99	0.000 01

[A] This table applies for all air density values between 0.0011 and 0.0013 g/mL. For air densities outside this range, the vacuum correction shall be calculated from the equation $C = (d_a/0.998\ 23) \times [0.998\ 23 - (W/V)]d_a$ being the density of the air in the balance case in grams per millilitre.

TABLE 2 Density of Water[A]

Temperature, ° C	Density, g/mL	Temperature, °C	Density, g/mL	Temperature, °C	Density, g/mL
0	0.999 840	21	0.997 991	40	0.992 212
3	0.999 964	22	0.997 769	45	0.990 208
4	0.999 972	23	0.997 537	50	0.988 030
5	0.999 964	24	0.997 295	55	0.985 688
10	0.999 699	25	0.997 043	60	0.983 191
15	0.999 099	26	0.996 782	65	0.980 546
15.56	0.999 012	27	0.996 511	70	0.977 759
16	0.998 943	28	0.996 231	75	0.974 837
17	0.998 774	29	0.995 943	80	0.971 785
18	0.998 595	30	0.995 645	85	0.968 606
19	0.998 404	35	0.994 029	90	0.965 305
20	0.998 203	37.78	0.993 042	100	0.958 345

[A] Densities conforming to the International Temperature Scale 1990 (ITS 90) were extracted from Appendix G, *Standard Methods for Analysis of Petroleum and Related Products 1991*, Institute of Petroleum, London.

example, relative density (specific gravity), 40°C/15.56°C = x.xxxx). Carry out all calculations to five figures, and round off the final results to four figures.

12. Precision and Bias

12.1 The precision of the method as obtained by statistical examination of interlaboratory test results is as follows:

12.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Pycnometer Volume, mL	Repeatability, g/mL
10	0.000 15

12.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Pycnometer Volume, mL	Reproducibility, g/mL
10	0.000 35

NOTE 5—If pycnometers of other than 10 mL in volume are used, this precision statement may not apply.

12.2 *Bias*—The difference of results from the established value when compared to pure reference materials is not expected to be more than 0.000 35 g/mL. Specific bias has not been established by cooperative testing.

13. Keywords

13.1 density; gravity; pycnometer; relative density; specific gravity

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1518: Standard Test Method for Thermal Transmittance of Textile Materials

CFR Section(s): 46 CFR 160.174-17(f)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1518 – 85 (Reapproved 1998)$^{\epsilon 1}$

Standard Test Method for Thermal Transmittance of Textile Materials[1]

This standard is issued under the fixed designation D 1518; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

ϵ^1 Note—Editorial changes were made throughout June 1998.

1. Scope

1.1 This test method covers the determination of the overall thermal transmission coefficients due to the combined action of conduction, convection, and radiation for dry specimens of textile fabrics, battings, and other materials within the limits specified in 1.2. It measures the time rate of heat transfer from a warm, dry, constant-temperature, horizontal flat-plate up through a layer of the test material to a relatively calm, cool atmosphere.

1.2 For practical purposes, this test method is limited to determinations on specimens of fabrics, layered fabric assemblies, and battings having thermal transmittances (U_2, as defined in 3.1.2) within a range of 0.7 to 14 W/m^2·K and thicknesses not in excess of 50 mm.

1.3 The coefficients obtained apply strictly only to the particular specimens tested and for the specified thermal and environmental conditions of each test. This test method gives values that are valid for comparison under the same conditions of test, that is, with the specified air velocity, temperature difference between the warm plate and the cool air, and air gap for measuring cool air temperature.

1.4 The values stated in metric units are to be regarded as the standard. Conversion factors, for thermal conductance and conductivity and thermal resistance and resistivity, to other units in common use are given in Tables 1-5

1.5 *This standard does not purport to address the safety concerns associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[2]

D 1777 Method for Measuring Thickness of Textile Materials[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *bulk density, n*—apparent mass per unit volume.

3.1.1.1 *Discussion*—In testing the thermal transmittance of fabrics, bulk density is calculated from the fabric weight per unit area and the thickness value used to calculate thermal conductivity.

3.1.2 *clo, n*—unit of thermal resistance defined as the insulation required to keep a resting man (producing heat at the rate of 58 W/m^2) comfortable in an environment at 21°C, air movement 0.1 m/s, or roughly the insulation value of typical indoor clothing.[3,4] (Syn. intrinsic clo).

3.1.2.1 *Discussion*—Numerically the clo is equal to 0.155 K·m^2/W.

3.1.3 *heat transfer coefficient, n*—see *thermal transmittance.*

3.1.4 *intrinsic clo, n*—see clo.

3.1.5 *specific clo, n*—the specific thermal resistance in clo units per unit thickness.

3.1.6 *thermal conductance, n*—see *thermal transmittance.*

3.1.7 *thermal conductivity, n*—time rate of unidirectional heat transfer per unit area, in the steady-state, between parallel planes separated by unit distance, per unit difference of temperature of the planes.

3.1.7.1 *Discussion*—Numerically, thermal conductivity equals the product of the heat transfer coefficient and the distance separating the planes. Thus, k, the thermal conductivity of the fabric only, is the product of U_2 and the fabric thickness. Units of thermal conductivity are W/m·K.

3.1.8 *thermal resistance, n*—reciprocal of thermal transmittance.

3.1.9 *thermal resistivity, n*—reciprocal of thermal conductivity.

3.1.10 *thermal transmittance, n*—time rate of unidirectional heat transfer per unit area, in the steady-state, between parallel planes, per unit difference of temperature of the planes (Syn. thermal conductance, heat transfer coefficient).

3.1.10.1 *Discussion*—Thermal transmittance is expressed as watts per square metre of test specimen per kelvin difference

---

[1] This test method is under the jurisdiction of ASTM Committee D-13 on Textiles and is the direct responsibility of Subcommittee D13.51 on Chemical Conditioning and Performance.

Current edition approved July 26, 1985. Published September 1985. Originally published as D 1518 – 57 T. Last previous edition D 1518 – 77.

[2] *Annual Book of ASTM Standards,* Vol 07.01.

[3] American Society of Heating, Refrigerating, and Air-Conditioning Engineers.

[4] Gagge, A. P., Burton, A. C., Bazett, H. C., *Science,* Vol 94, Nov. 7, 1941, pp. 428–430.

TABLE 1 Conversion Factors for Thermal Conductivity[A]

To Convert Thermal Conductivity	Multiply by									
From to	W/m·K[B]	W·cm/m^2·K	W/cm·K	cal/s·cm·K	kg-cal/h·m·K	kg-cal·cm/ h·m^2·K	Btu/h·ft·°F	Btu·in/ h·ft^2·°F	in/clo	mm/clo
W/m·K	1.	$1. \times 10^{+2}$	$1. \times 10^{-2}$	2.388×10^{-3}	8.598×10^{-1}	$8.598 \times 10^{+1}$	5.778×10^{-1}	6.934	6.093	$1.548 \times 10^{+2}$
W·cm/ m^2·K	$1. \times 10^{-2}$	1.	$1. \times 10^{-4}$	2.388×10^{-5}	8.598×10^{-3}	8.598×10^{-1}	5.778×10^{-3}	6.934×10^{-2}	6.093×10^{-2}	1.548
W/cm·K	$1. \times 10^{+2}$	$1. \times 10^{+4}$	1.	2.388×10^{-1}	$8.598 \times 10^{+1}$	$8.598 \times 10^{+3}$	$5.778 \times 10^{+1}$	$6.934 \times 10^{+2}$	$6.093 \times 10^{+2}$	$1.548 \times 10^{+4}$
cal/s·cm·K	$4.187 \times 10^{+2}$	$4.187 \times 10^{+4}$	4.187	1.	$3.6 \times 10^{+2}$	$3.6 \times 10^{+4}$	$2.419 \times 10^{+2}$	$2.903 \times 10^{+3}$	$2.551 \times 10^{+3}$	$6.480 \times 10^{+4}$
kg-cal/ h·m·K	1.163	$1.163 \times 10^{+2}$	1.163×10^{-2}	2.778×10^{-3}	1.	$1. \times 10^{+2}$	6.720×10^{-1}	8.064	7.087	$1.8 \times 10^{+2}$
kg-cal·cm/ h·m^2·K	1.163×10^{-2}	1.163	1.163×10^{-4}	2.778×10^{-5}	$1. \times 10^{-2}$	1.	6.720×10^{-3}	8.064×10^{-2}	7.087×10^{-2}	1.8
Btu/h·ft·°F	1.731	$1.731 \times 10^{+2}$	1.731×10^{-2}	4.134×10^{-3}	1.488	$1.488 \times 10^{+2}$	1.	$1.2 \times 10^{+1}$	$1.055 \times 10^{+1}$	$2.679 \times 10^{+2}$
Btu·in/ h·ft^2·°F	1.442×10^{-1}	$1.442 \times 10^{+1}$	1.442×10^{-3}	3.445×10^{-4}	1.240×10^{-1}	$1.240 \times 10^{+1}$	8.333×10^{-2}	1.	8.788×10^{-1}	$2.232 \times 10^{+1}$
in/clo	1.641×10^{-1}	$1.641 \times 10^{+1}$	1.641×10^{-3}	3.920×10^{-4}	1.411×10^{-1}	1.411×10^{-1}	9.482×10^{-2}	1.138	1.	$2.540 \times 10^{+1}$
mm/clo	6.461×10^{-3}	6.461×10^{-1}	6.461×10^{-5}	1.543×10^{-5}	5.556×10^{-3}	5.556×10^{-1}	3.733×10^{-3}	4.480×10^{-3}	3.937×10^{-2}	1.

[A] Units are given in terms of: (1) the absolute joule per second, or watt; (2) the calorie (International Table) = 4.1868 J; (3) the British thermal unit (International Table) = 1055.06 J; and (4) the clo (unit of clothing resistance) = 0.155 K·m^2/W.

[B] Recommended (SI) units.

TABLE 2 Conversion Factors for Thermal Transmittance[A]

To Convert Thermal Transmittance	Multiply by					
From to	W/m^2·K[B]	W/cm^2·K	cal/s·cm^2·K	kg-cal/h·m^2·K	Btu/h·ft^2·°F	clo^{-1}
W/m^2·K	1.	$1. \times 10^{-4}$	2.388×10^{-5}	8.598×10^{-1}	1.761×10^{-1}	1.548×10^{-1}
W/cm^2·K	$1. \times 10^{+4}$	1.	2.388×10^{-1}	$8.598 \times 10^{+3}$	$1.761 \times 10^{+3}$	$1.548 \times 10^{+3}$
cal/s·cm^2·K	$4.187 \times 10^{+4}$	4.187	1.	$3.6 \times 10^{+4}$	$7.373 \times 10^{+3}$	$6.480 \times 10^{+3}$
kg-cal/h·m^2·K	1.163	1.163×10^{-4}	2.778×10^{-5}	1.	2.048×10^{-1}	1.8×10^{-1}
Btu/h·ft^2·°F	5.678	5.678×10^{-4}	1.356×10^{-4}	4.882	1.	8.788×10^{-1}
clo^{-1}	6.461	6.461×10^{-4}	1.543×10^{-4}	5.556	1.138	1.

[A] Units are given in terms of: (1) the absolute joule per second, or watt; (2) the calorie (International Table) = 4.1868 J; (3) the British thermal unit (International Table) = 1055.06 J; and (4) the clo (unit of clothing resistance) = 0.155 K·m^2/W.

[B] Recommended (SI) units.

between the hot plate and the cool atmosphere (W/m^2·K).

Thermal transmittance for three different cases is determined in this method:

U_1 = combined thermal transmittance of the test specimen and air.

U_{bp} = thermal transmittance of the plate without fabric cover ("bare plate"). This property reflects the instrument constant and is used to standardize the plate, and, in conjunction with U_1, is used in the calculation of U_2.

U_2 = thermal transmittance of fabric only. This value corresponds to the *C* value (W/m^2·K) defined and used by ASTM and ASHRAE.[4] In the calculation of this value the assumption is made that the boundary layers of the bare plate and the boundary layers of the fabric are equal. Experimental results indicate that the U_2 values are valid when tested within the limits specified in Section 1.

3.1.11 *total clo, n*—the intrinsic clo plus the thermal resistance from the air boundary.

3.1.12 For definitions of other textile terms used in this method, refer to Terminology D 123.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *effective insulation ratio, n*—indicates the increase in insulation afforded by the fabric in comparison to the uncovered test plate under specified conditions of test.

3.2.2 *mean temperature, n*—the average of the hot plate temperature and the temperature of the calm, cool air that prevailed during the test.

4. Significance and Use

4.1 The thermal transmittance of a fabric or batting is of considerable importance in determining its suitability for use in fabricating cold weather protective gear and clothing. The thermal interchange between man and his environment is, however, an extremely complicated subject which involves many factors in addition to the equilibrium insulation values of fabrics and battings. Therefore, measured thermal transmittance coefficients can only indicate relative merit of a particular material.

4.2 The measurement of heat transfer coefficients is a very difficult and highly technical field, and it is not practical in a test method of this scope to establish details sufficient to cover all contingencies. Departures from the instructions of Test

TABLE 3 Conversion Factors for Thermal Resistivity[A]

To Convert Thermal Resistivity[B] From to	Multiply by									
	m·K/W[B]	m²·K/W·cm	cm·K/W	cm·K·s/cal	m·K·h/kg-cal	m²·K·h/kg-cal·cm	ft·°F·h/Btu	ft²·°F·h/Btu·in	clo/in	clo/mm
m·K/W	1.	$1. \times 10^{-2}$	$1. \times 10^{+2}$	$4.187 \times 10^{+2}$	1.163	1.163×10^{-2}	1.731	1.442×10^{-1}	1.641×10^{-1}	6.461×10^{-3}
m²·K/W·cm	$1. \times 10^{+2}$	1.	$1. \times 10^{+4}$	$4.187 \times 10^{+4}$	$1.163 \times 10^{+2}$	1.163	$1.731 \times 10^{+2}$	$1.442 \times 10^{+1}$	1.641×10^{-1}	6.461×10^{-1}
cm·K/W	$1. \times 10^{-2}$	$1. \times 10^{-4}$	1.	4.187	1.163×10^{-2}	1.163×10^{-4}	1.731×10^{-2}	1.442×10^{-3}	1.641×10^{-3}	6.461×10^{-5}
cm·K·s/cal	2.388×10^{-3}	2.388×10^{-5}	2.388×10^{-1}	1.	2.778×10^{-3}	2.778×10^{-5}	4.134×10^{-3}	3.445×10^{-4}	3.920×10^{-4}	1.543×10^{-5}
m·K·h/kg-cal	8.598×10^{-1}	8.598×10^{-3}	$8.598 \times 10^{+1}$	$3.6 \times 10^{+2}$	1.	$1. \times 10^{-2}$	1.488	1.240×10^{-1}	1.411×10^{-1}	5.556×10^{-3}
m²·K·h/kg-cal·cm	$8.598 \times 10^{+1}$	8.598×10^{-1}	$8.598 \times 10^{+3}$	$3.6 \times 10^{+4}$	$1. \times 10^{+2}$	1.	$1.488 \times 10^{+2}$	$1.240 \times 10^{+1}$	$1.411 \times 10^{+1}$	5.556×10^{-1}
ft·°F·h/Btu	5.778×10^{-1}	5.778×10^{-3}	$5.778 \times 10^{+1}$	$2.419 \times 10^{+2}$	6.720×10^{-1}	6.720×10^{-3}	1.	8.333×10^{-2}	9.482×10^{-2}	3.733×10^{-3}
ft²·°F·h/Btu·in	6.934	6.934×10^{-2}	$6.934 \times 10^{+2}$	$2.903 \times 10^{+3}$	8.064	8.064×10^{-2}	$1.2 \times 10^{+1}$	1.	1.138	4.480×10^{-3}
clo/in	6.093	6.093×10^{-2}	$6.093 \times 10^{+2}$	$2.551 \times 10^{+3}$	7.087	7.087×10^{-2}	$1.055 \times 10^{+1}$	8.788×10^{-1}	1.	3.937×10^{-2}
clo/mm	$1.548 \times 10^{+2}$	1.548	$1.548 \times 10^{+4}$	$6.480 \times 10^{+4}$	$1.8 \times 10^{+2}$	1.8	$2.679 \times 10^{+2}$	$2.232 \times 10^{+1}$	$2.540 \times 10^{+1}$	1.

[A] Units are given in terms of: (1) the absolute joule per second, or watt; (2) the calorie (International Table) = 4.1868 J; (3) the British thermal unit (International Table) = 1055.06 J; and (4) the clo (unit of clothing resistance) = 0.155 K·m²/W.

[B] Recommended (SI) units.

TABLE 4 Conversion Factors for Thermal Resistance[A]

To Convert Thermal Resistance From to	Multiply by					
	m²·K/W[B]	cm²·K/W	cm²·K·s/cal	m²·K·h/kg-cal	ft²·°F·h/Btu	clo
m²·K/W	1.	$1. \times 10^{+4}$	$4.187 \times 10^{+4}$	1.163	5.678	6.461
cm²·K/W	$1. \times 10^{-4}$	1.	4.187	1.163×10^{-4}	5.678×10^{-4}	6.461×10^{-4}
cm²·K·s/cal	2.388×10^{-5}	2.388×10^{-1}	1.	2.778×10^{-5}	1.356×10^{-4}	1.543×10^{-4}
m²·K·h/kg-cal	8.598×10^{-1}	$8.598 \times 10^{+3}$	$3.6 \times 10^{+4}$	1.	4.882	5.556
ft²·°F·h/Btu	1.761×10^{-1}	$1.761 \times 10^{+3}$	$7.373 \times 10^{+3}$	2.048×10^{-1}	1.	1.138
clo	1.548×10^{-1}	$1.548 \times 10^{+3}$	$6.480 \times 10^{+3}$	1.8×10^{-1}	8.788×10^{-1}	1.

[A] Units are given in terms of: (1) the absolute joule per second, or watt; (2) the calorie (International Table) = 4.1868 J; (3) the British thermal unit (International Table) = 1055.06 J; and (4) the clo (unit of clothing resistance) = 0.155 K·m²/W.

[B] Recommended (SI) units.

TABLE 5 Miscellaneous Conversion Factors

Properties	To Convert from a Value Expressed as	To a Value Expressed as	Multiply by
Mass per unit area	oz/yd²	g/m²	33.91
	mg/cm²	g/m²	10.0
Thickness	in.	mm	25.4
	1/1000 in. (mil)	mm	0.0254
Bulk density	lb/ft³	kg/m³	16.02
	(oz/yd²)/in	kg/m³	1.335
	(g/m²)/mm	kg/m³	1.0

Method D 1518 may lead to significantly different test results. Technical knowledge concerning the theory of heat flow, temperature measurement, and testing practices is needed to evaluate which departures from the instructions are significant. Standardization of the method reduces, but does not eliminate the need for such technical knowledge. Any significant departures are to be reported with the results.

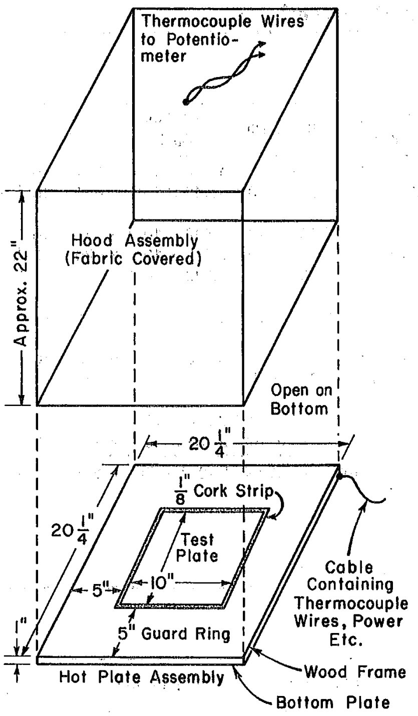
4.3 Test Method D 1518 for the determination of the thermal transmittance of textile materials is considered satisfactory for acceptance testing of commercial shipments of textile materials because the test method has been used in the trade for acceptance testing. And it is the best test method known for this purpose.

4.3.1 In case of a dispute arising from differences in reported results when using Test Method D 1518 for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine if there is a statistical bias between their laboratories. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the two parties should take a group of test specimens which are as homogeneous as possible and which are from a lot of material of the type in question. The test specimens should then be sent to each laboratory for testing. The average results from the two laboratories should be compared using Student's *t*-test for paired data and an acceptable probability level chosen by the two parties before testing is begun. If a bias is found, either its cause must be found and corrected or the purchaser and the supplier must agree to interpret future test results with consideration to the known bias.

5. Apparatus (Fig. 1, Fig. 2, and Fig. 3)

NOTE 1—The drawings and illustrations are intended as suggested designs only. The final design of equipment, including necessary wiring, will be dictated by the choice of the electrical measuring and control equipment.

5.1 *Hot Plate*—A guard ring flat plate composed of a test plate, guardring, and bottom plate as follows, each electrically maintained at a constant temperature in the range of human



Entire Assembly to be Located in a Calm Atmosphere, 40 to 70 F Temperature Fluctuations Less Than ± 2.5 F

FIG. 1 Guard Ring Hot Plate For Thermal Transmittance Test

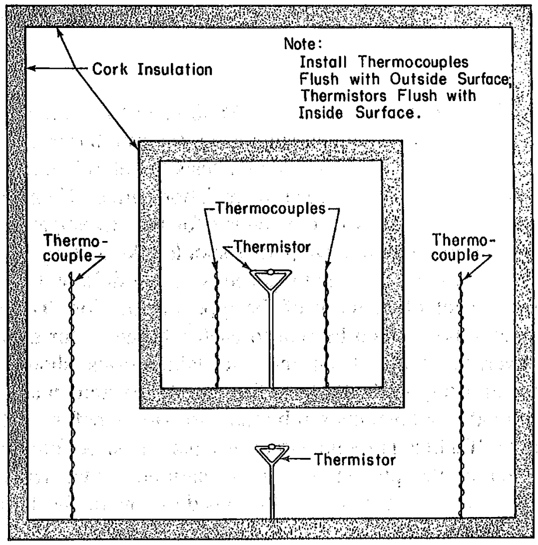


FIG. 2 Hot Plate, Top View, Showing Location of Thermistors and Thermocouples on Test Section and Guard Ring

skin temperature [33 to 36°C (91.4 to 98.8°F)].

5.1.1 *Test Plate*—The test plate portion of the hot plate shall be at least 150 mm (6.0 in.) square and shall be placed at the center of the upper surface of the hot-plate assembly. It shall be made of aluminum or copper and painted a dull black to approximate the emissivity of the human skin. The heating element shall consist of parallel wires, preferably of constantan metal, insulated from, but mounted within 3 mm (0.1 in.) of the upper plate.

5.1.2 *Guard Ring*—The guard ring bordering the test plate shall be at least 63.5 mm (2.5 in.) in width and shall be of the same thickness, composition, and type of construction as the test plate. It shall be coplanar with the test plate, and shall be separated from it by means of a strip of cork or other suitable insulating material approximately 3-mm (0.1-in.) wide. The guard ring shall be designed to prevent lateral loss of heat from the test plate.

5.1.3 *Bottom Plate*—The bottom plate shall be of the same thickness, composition, and type of construction as the test plate and guard ring. The bottom plate shall be in a plane parallel to the test plate and guard ring, and at a distance of at least 25 mm (1.0 in.) but not in excess of 75 mm (3.0 in.) beneath them. It shall be separated from the test plate and guard ring by a wooden framework and the air pocket formed thereby, or by other means of causing air entrapment. The dimensions offered as suggested design specifications are shown in Fig. 3. The purpose of the bottom plate is to prevent a downward loss of heat from the test plate and guard ring.

5.2 *Temperature Control*—Separate control of the temperatures of the three sections of the hot plate (test plate, guard ring, and bottom plate) shall be established by independent adjustments of the heater currents through adjustable transformers, variable impedances, or intermittent heating cycles. Automatic regulation of temperatures is recommended. Use a constant voltage supply, controlled to ±1 % to minimize fluctuations in temperature.

5.3 *Power-Measuring Instruments*—One of any of the following instruments shall be used for measuring power:

5.3.1 Wattmeter,

5.3.2 Watt-hour meter and clock,

5.3.3 Voltmeter and ammeter, or

5.3.4 Either a voltmeter *or* an ammeter can be used if the test plate heater resistance at operating temperature is exactly known. These devices shall be operated in accordance with standard practice and shall be calibrated to measure power with an accuracy of ±2 %.

5.4 *Clocks*—When heater power is supplied on an intermittent basis, a running-time clock, energized in synchronism with the heater, shall be used to indicate the total time of heating. Another similar clock shall be used to indicate either the total time or the time during which the heater is not energized. The total limit of error of such clocks shall be less than 1 % under service conditions.

5.5 *Equipment for Measuring the Several Plate Temperatures*:

5.5.1 *Thermocouples*—The test plate, guard ring, and bottom plate shall each contain one or more thermocouples made of a junction of wires of copper and constantan, each of B & S Gage No. 30 [0.255 mm (0.01 in.)]. After calibration, these thermocouples shall be positioned within the material of the plates as close to the external plate surfaces as physically possible [1.6 mm (0.06 in.)] to measure the temperatures of the respective surfaces.

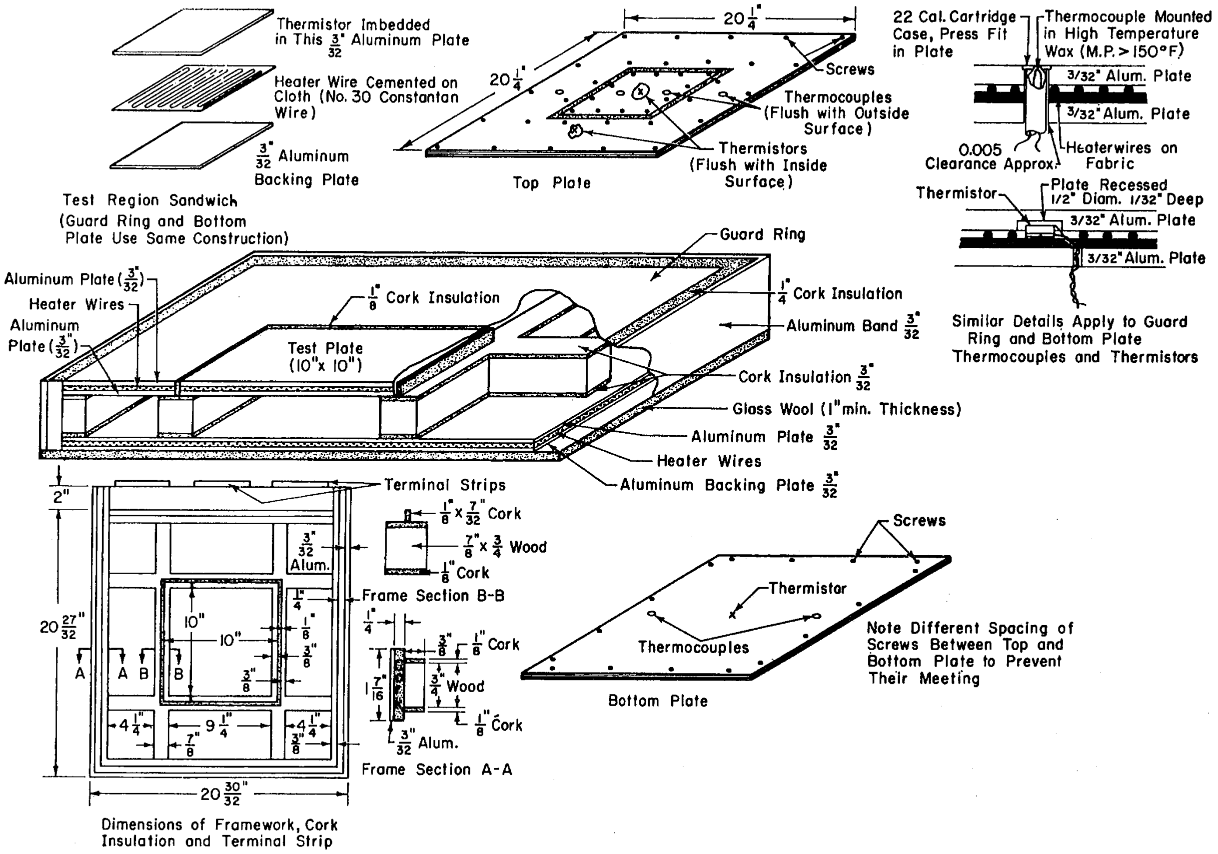


FIG. 3 Apparatus for Measurement of Thermal Transmittance, Showing Dimensions

5.5.2 *Ice Bath*, as a reference junction for the thermocouples, or equivalent device.

5.5.3 *Potentiometer*, accurate within ±2.5 µV, to measure the thermocouple emf's.

5.5.4 *Switch*—A thermocouple selector switch for separately connecting to each set of thermocouples.

5.6 *Test Chamber*—A chamber to house the hot plate that can be maintained at selected temperatures between 4.5 and 21.1°C (40 to 70°F) with a constancy of ±0.5°C (± 2.5°F). The walls of the test chamber shall not be highly reflective, and the wall temperature shall be equal to that of the air in the chamber. The chamber shall be equipped with the following instruments for maintaining the relative humidity at 50± 30 % for maintaining the air temperature, and for controlling the air velocity at the approximate rate of 0.1 m/s (0.33 ft/s). The hood for maintaining nearly still air conditions, shown in Fig. 1, is needed.

5.6.1 *Relative Humidity Measuring Equipment*—Either a wet-and-dry bulb psychrometer or a calibrated humidity-sensitive electrical conductor.

5.6.2 *Air Temperature Detector*—A thermocouple similar to those in the plates is suspended with the measuring junction exposed to the air at a point 500 mm (20.0 in.) above the center of the test plate, inside hood.

5.6.3 *Air Velocity Indicator*—Any calibrated means of measuring air velocity at the specified rate.

6. Sampling

6.1 *Lot Sample*—for acceptance testing take a lot sample as directed in the applicable material specification, or as agreed upon between purchaser and supplier. In the absence of such a specification or other agreement, take a laboratory sample as directed in 6.2.

6.2 Take a laboratory sample from each roll or piece of fabric in the lot sample. The laboratory sample should be full width and at least 600 mm (24 in.) long and should not be taken any closer to the end of the roll or piece of fabric than 1 m (1 yd).

6.3 Sample shipments of garments or other textile materials as agreed upon between purchaser and seller.

6.4 Test three specimens from each laboratory sample, unless otherwise specified in the material specification.

7. Preparation of Test Specimens

7.1 *Modification of a Thick Material to Facilitate Testing*—Materials more than 25-mm (1–in.) thick, such as some fibrous battings, require an extremely long period for reaching equilibrium. In such a case, if the specimen has a homogeneous structure, and it is physically possible to slice through the material in such a manner as to split it into two or more uniform layers thinner than the original, one of the thin layers may be tested and its coefficient determined. (This is not applicable to fabric assemblies or to otherwise heterogeneous material.)

7.2 *Specimen Preparation*—Cut the test specimens large enough to cover completely the entire surface of the hot plate and the guard plates, or about 510 mm (20 in.) square. Remove any wrinkles from the test specimens by allowing to hang free or by ironing. For quilted fabrics or batts, sew or seal the edges or use retaining slats during the testing.

7.3 *Conditioning*—Allow the test specimens to come into equilibrium with the atmosphere of the testing chamber. Moisture equilibrium for testing is considered as having been reached when the rate of increase in mass of a sample or specimen does not exceed that specified for the material being tested.

7.3.1 In the absence of a specified rate, an increase of less than 0.1 % of the sample mass after a 2-h exposure is considered satisfactory.

8. Preparation and Standardization of Apparatus

8.1 *Test Conditions*—Unless otherwise specified in the detail specification, use the following test conditions:

8.1.1 *Temperature of the Test Plate, Guard Ring, and Bottom Plate*—Select a temperature in the range from 33 to 36°C (91.4 to 98°F) to be maintained for the duration of the test for the test plate, guard ring, and the bottom plate.

8.1.2 *Maximum Difference in Temperature*—Maintain and stabilize the test equipment to have a maximum temperature difference between either the guard ring or bottom plate and the test plate of ±0.3°C.

8.2 *Temperature of Test Chamber* (External to the Hood)—Maintain the average temperature of the test chamber at a specified temperature between 4.5 and 21.1°C (40 to 70°F) with a range in temperature not to exceed ±0.5°C (±2.5°F).

8.3 *Relative Humidity Within the Test Chamber*—Maintain the relative humidity within the test chamber at a selected level between 20 and 80 % with a range not to exceed ±5 %.

9. Procedure

9.1 Determine the thickness of the original specimen and, if necessary (see 6.1), the component layer to be tested to within 0.3 mm (0.01 in.) at a loading pressure of 0.07 kPa (0.01 psi) as directed in Method D 1777. Use any suitable thickness gage having a presser foot diameter of at least 50 mm (2 in.).

9.2 Spread the test specimen flat on the hot plate with the finished side up, unless otherwise specified. Ensure good thermal contact by smoothing out any abnormal wrinkles or air pockets between the specimen and the plate surface. Unless otherwise specified, use no supplemental loading beyond the intrinsic mass of the specimen.

9.3 Bring the hot plate to the operating temperature and allow the system (specimen plus plate) to reach equilibrium, defined as that state in which the test-plate temperature and the power input remains constant. The temperature shall be held within ±0.5°C and the average temperature shall not be allowed to drift more than ±0.05°C during a period of 30 min. To ensure a constant power input for the duration of the test, the temperature equilibrium shall be maintained using an on-off ratio for power of 50 to 60 % of the time required to complete the test.

9.4 After the assembly reaches equilibrium conditions, record measurements for each of the following conditions at least every 3 min. The average of these measurements taken over a period of 30 min shall be sufficient to determine the combined transmittance coefficient of the specimen plus the air, U_1.

9.4.1 Test plate temperature,

9.4.2 Test plate heater wattage,

9.4.3 Air temperature,

9.4.4 Guard ring temperature, and

9.4.5 Bottom plate temperature.

NOTE 2—If the foregoing observations are not consistent with equilibrium conditions, the test shall not be valid and shall be repeated after establishment of equilibrium.

9.5 *Bare Plate*—Measure the bare-plate transmittance coefficient, U_{bp}, in the same manner as that for U_1 except that the hot plate shall be uncovered during this measurement.

10. Calculations

10.1 Calculate the combined transmittance of the specimen plus the air, U_1, to within 0.005 W/m²·K, using Eq 1:

$$U_1 = P/[A \times (T_p - T_a)] \tag{1}$$

where:

P = power loss from test plate, W,
A = area of test plate, m²,
T_p = test plate temperature, °C, and
T_a = air temperature, °C.

10.2 Calculate the bare-plate transmittance, U_{bp}, as for U_1 in 10.1.

10.3 Calculate the intrinsic transmittance of the fabric alone, U_2, using Eq 2 or Eq 3:

$$1/U_2 = (1/U_1) - (1/U_{bp}) \tag{2}$$

or

$$U_2 = (U_{bp} \times U_1)/(U_{bp} - U_1) \tag{3}$$

10.4 Calculate the intrinsic thermal conductivity of the fabric alone, k, using Eq 4:

$$k = U_2 \times t_i/1000 \tag{4}$$

where:

t_i = thickness of the specimen, mm, at 0.07 kPa pressure.

10.5 Calculate the intrinsic thermal resistance of the fabric alone, R (Note 3), using Eq 5:

$$R = 1/U_2 \tag{5}$$

NOTE 3—The addition of values of R measured independently for two or more fabrics (one fabric of which is less than 1.3 mm thick) to calculate the thermal resistance of an ensemble, is often invalid, due to the influence of one fabric on the thermal resistance associated with the other. For example, a fleece-lined windbreaker affords far more insulation, in moving air, than the sum of the insulation of the lining and outer fabric taken separately.

10.6 Calculate the intrinsic thermal resistivity of the fabric alone, R' (Note 3), using Eq 6:

$$R' = 1/k \tag{6}$$

10.7 Calculate the intrinsic thermal resistance in Clo units using equation (7):

$$\text{Intrinsic Clo} = 1.137/U \tag{7}$$

10.8 Calculate the specific thermal resistance in Clo units using equation (8):

$$\text{Specific Clo} = 1.137/k \qquad (8)$$

10.9 Calculate the bulk density, B, of the fabric, using Eq 9:

$$B = M/t \qquad (9)$$

where:

B = bulk density, kg/m^3,
M = mass/unit area of fabric, g/m^2, and
t = thickness of fabric, mm.

10.10 Calculate the split-specimen coefficient, U_2, for the thin section in accordance with 10.1, 10.2, and 10.3. Calculate the U_2 of the original section by multiplying the thin section coefficient by the thickness ratio of the thin section to the original section, using Eq 10:

$$U_{20} = U_{2t} \times (t_t/t_0) \qquad (10)$$

where:

U_{20} = original split-specimen coefficient,
U_{2t} = thin section, split-specimen coefficient,
t_t = thickness of thin section, and
t_0 = thickness of the original section.

10.11 Calculate the mean temperature, T_m, for each determination using Eq 11:

$$T_m = (T_a + T_p)/2 \qquad (11)$$

where:

T_a = atmosphere temperature, and
T_p = plate surface temperature.

10.12 Calculate the effective insulation ratio, I_r, using Eq 12:

$$I_r = U_{bp}/U_1 \qquad (12)$$

10.13 To convert heat transfer quantities from SI to mixed, engineering, or clothing units or vice-versa multiply by the appropriate factor from Tables 1-5.

11. Report

11.1 State that the specimens were tested as directed in ASTM Test Method D 1518. Describe the materials or products sampled and the method of sampling used.

11.2 Report the following information:

11.2.1 Mean temperature of the test.

11.2.2 Average heat transfer coefficient of the bare plate alone, U_{bp},

11.2.3 Average of the heat transfer coefficient of the plate and fabric combined, U_1,

11.2.4 Thermal conductance of the fabric, U_2,

11.2.5 Fabric weight, thickness, and bulk density, and

11.2.6 Thermal conductivity, resistance, and resistivity of the fabric, as required.

11.2.7 The temperature and relative humidity used.

12. Precision and Bias

12.1 *Summary*—In comparing two single observations for the thermal transmittance expressed as U_2, the difference should not exceed 4.5 % of the average of two observations in 95 out of 100 cases when both observations are taken by the same well-trained operator using the same piece of testing equipment and specimens randomly drawn from the same sample of material. Larger differences are likely to occur under other circumstances.

12.2 *Interlaboratory Test Data*—An interlaboratory test was run in 1980 and 1981 in which randomly drawn samples of five materials were tested in each of five laboratories. Two operators in each of the five laboratories tested two specimens of each material. The components of variance for the thermal transmittance results are shown in Table 6. Components of variance expressed as coefficients of variation were calculated as follows:

TABLE 6 Components of Variance

Single-operator component	2.2 % of the average
Between-laboratory component	10.7 % of the average

12.3 *Critical Difference*—For the components of variance reported in Table 6, two averages of observed values should be considered significantly different at the 95 % probability level if the differences equal or exceed critical differences shown in Table 7.

TABLE 7 Critical Difference for the Components of Variance

Number of Observations in Each Average	Critical Difference, % of Grand Average for the Condition Noted[A,B] Single Operator Precision	Between Laboratory Precision
1	6.2	29.7
5	2.8	13.2
10	2.0	9.4

[A] The critical differences were calculated using t = 1.960, which is based on infinite degrees of freedom.

[B] To convert the values of critical differences to units of measure, multiply the critical difference by the average of the two specific sets of data being compared, then divide by 100.

12.4 *Confidence Limits*—For the components of variance in Table 6 single averages of observed values have 95 % confidence limits (Note 4) in Table 8.

TABLE 8 Confidence Limits

Number of Observations in Each Average	Width of 95 % Confidence Limits, % of Grand Average for the Condition Noted Single Operator Precision	Between Laboratory Precision
1	4.4	20.9
5	2.0	9.4
10	1.4	6.6

NOTE 4—The tabulated values of the critical differences and confidence limits should be considered to be a general statement, particularly with respect to between-laboratory precision. Before a meaningful statement can be made about two specific laboratories, the amount of statistical bias, if any, between them must be established, with each comparison being based on recent data obtained on specimens randomly drawn from one sample of the material to be evaluated.

12.5 *Bias*—The value of the thermal transmittance can only be defined in terms of a specific test. Within this limitation Test Method D 1518 has no known bias.

13. Keywords

13.1 batting; textile fabrics; thermal transmittance

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM D1535: Specifying Color by the Munsell System

CFR Section(s): 7 CFR 1755.860(c)(3)

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1535 – 89

Standard Test Method for Specifying Color by the Munsell System[1]

This standard is issued under the fixed designation D 1535; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides a means of specifying the colors of objects in terms of the Munsell color order system, a system based on the color-perception attributes hue, lightness, and chroma. The test method is limited to opaque objects, such as painted surfaces viewed in daylight by an observer having normal color vision. This test method provides a simple visual method as an alternative to the more precise and more complex method based on spectrophotometry and the CIE system (see Method E 308 and Practice E 1164). Provision is made for conversion of CIE data to Munsell notation.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1729 Practice for Visual Evaluation of Color Differences of Opaque Materials[2]

E 284 Definitions of Terms Relating to Appearance of Materials[3]

E 308 Method for Computing the Colors of Objects by Using the CIE System[3]

E 1164 Practice for Obtaining Spectrophotometric Data for Object-Color Evaluation[3]

3. Terminology

3.1 *Descriptions of Terms:*

3.1.1 *Munsell surface-color perception solid, n*—a spatial representation of colors in the form of a cylindrical coordinate system based on the three perceptual attributes: hue, lightness and chroma, as shown in Fig. 1.[4] This solid forms the basis of the Munsell notation in which Munsell hue corresponds to hue, Munsell value corresponds to lightness, and Munsell chroma corresponds to chroma. The central, vertical axis dimension represents neutral colors, ranging from black at the bottom, through a gradation of grays, to white at the top. The lightness of a color perceived as chromatic (not gray) is represented by the distance above the base plane. Hue is represented by the angular position about this axis (see 3.1.1.1). Chroma is represented by the perpendicular distance from the central axis. If the observer has normal color vision, is adapted to daylight, and views the specimen illuminated by CIE source c or D65, against a medium gray to white background, the Munsell value of the specimen correlates well with the observer's perception of the lightness of the color. Under the same conditions, the Munsell hue correlates well with the observer's perception of hue and the Munsell chroma with the perception of chroma.

3.1.1.1 *Discussion*—Although the original system proposed by Munsell was a left-handed coordinate system, the system is often represented as a right-handed system because it facilitates comparison to the CIE chromaticity diagram, taken to be right-handed.

3.2 *Definitions*—see also Definitions E 284.

3.2.1 *Munsell notation, n*—(*1*) the Munsell hue, value, and chroma assigned to the color of a specimen by visually comparing the specimen to the chips in the *Munsell Book of Color*;[5] (*2*) a notation in the Munsell color system, derived from daylight luminous reflectance factor Y and chromaticity coordinates x and y, in the CIE system, by the use of scales defined by the Optical Society of America Subcommittee on the Spacing of the Munsell Colors.[6]

3.2.1.1 *Discussion*—The Munsell notation is written as a combination of letters and numbers by which the color of an opaque object may be specified with respect to Munsell hue H, Munsell value V, and Munsell chroma C, written in the form $H\ V/C$.

3.2.2 *hue, n*—the attribute of color perception by means of which a color is judged to be red, orange, yellow, green, blue, purple, or intermediate between adjacent pairs of these, considered in a closed ring (red and purple being an adjacent pair).

3.2.3 *Munsell hue, n*—an attribute of color used in the Munsell color system to indicate the hue of a specimen viewed in daylight.

3.2.3.1 *Discussion*—Two systems of designating Munsell hue are shown in Fig. 2, a letter-number system and an all-number system. The two systems are equivalent, but the letter-number system is preferred, because it requires no prior knowledge or memory of the correspondence of numbers to hues. The hue circle is graduated in steps judged

---

[1] This test method is under the jurisdiction of ASTM Committee E-12 on Appearance of Materials and is the direct responsibility of Subcommittee E12.09 on Color Order Systems USTAGTC 187.

Current edition approved Oct. 27, 1989. Published December 1989. Originally published as D 1535 – 58 T. Last previous edition D 1535 – 80.

[2] *Annual Book of ASTM Standards*, Vol 06.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Taken from Nimeroff, I., "Colorimetry," *Monograph 104*, Nat. Bureau Standards, NBS, January 1968.

[5] Available from the Munsell Color Co., 2441 N. Calvert St., Baltimore, MD 21218.

[6] Newhall, S. M., Nickerson, D., and Judd, D. B., "Final Report of the OSA Subcommittee on the Spacing of the Munsell Colors," *Journal, Optical Society of America*, Vol 33, 1943, p. 385.

visually to be approximately equal.

3.2.4 *lightness, n*—the attribute of color perception by which a non-self-luminous body is judged to reflect more or less light.

3.2.5 *Munsell value, n*—an attribute of color used in the Munsell color system to indicate the lightness of a specimen viewed in daylight, on a scale extending from 0 for ideal black to 10 for ideal white, in steps that are visually approximately equal in magnitude.

3.2.5.1 *Discussion*—Achromatic or neutral colors are designated *N* followed by the value notation, thus: *N* 5.61/. Strictly interpreted, the neutral *N* implies zero chroma, but the *N* is often used to designate colors with chromas under 0.20, the letter symbol for the nearest of the principal hues being included in parentheses with the chroma, as in *N* 8.73/(2.58*Y*, 0.12).

3.2.6 *chroma, n*—the attribute of color used to indicate the degree of departure of the color from a gray of the same lightness.

3.2.7 *Munsell chroma, n*—an attribute of color used in the Munsell color system to indicate the degree of departure of a color from a gray of the same Munsell value, in steps that are visually approximately equal in magnitude.

4. Apparatus

4.1 *Munsell Book of Color*, matte or glossy edition.[5]

4.2 *Gray Masks*, with rectangular openings the size of the chips in the *Munsell Book of Color*.

4.3 *Daylight Illuminating Equipment*, as described in Practice D 1729.

5. Preparation of Test Specimens

5.1 This test method does not cover the preparation of test specimens. If preparation is necessary, see other ASTM standards covering the appropriate materials or agree among interested parties on what the procedure shall be.

6. Munsell Notation by Visual Means

6.1 *Lighting and Viewing Conditions:*

6.1.1 Specimens must be examined by an observer with normal color vision.

6.1.2 For critical applications, use daylight illuminating equipment as described in Practice D 1729.

6.1.3 If the lighting equipment described in Practice D 1729 is not available, natural daylight can be used to obtain notations having accuracy adequate for many purposes.

6.2 *Procedure:*

6.2.1 When using daylight illuminating equipment, follow the lighting and viewing recommendations of Practice D 1729.

6.2.2 When determining the Munsell notation with natural daylight, select a window through which the sun is not shining. A north window is usually used in the northern hemisphere, and a south window is usually used in the southern hemisphere. Place a working surface at the window so the light reaches the surface from the observer's side, chiefly from the sky, and at angles centering on 45° above the horizontal. Place a canopy of black cloth above the working surface to prevent errors caused by the ceiling or other objects being reflected from the surface of the specimens, or by light other than daylight falling on the work surface. Place the specimen on a neutral medium gray to white background, where it is uniformly illuminated by daylight. View the specimen along a direction just far enough from the normal to avoid reflection of your forehead. Although 45° illumination and perpendicular viewing are recommended by the CIE, converse conditions are equivalent if a black matte surface is placed opposite the observer to minimize the amount of light reflected from the specimen surface.

6.2.3 If both matte and glossy editions of the *Munsell Book of Color* are available, use the one having gloss most like the specimen. Select the two adjacent Munsell constant-hue charts or chips between which the hue of the specimen lies. Place one on each side of the specimen. Cover the specimen and charts with the gray masks so the specimen and one chip from each chart can be seen. Move the masks from chip to chip to find the chips most like the specimen. The glossy chips are removable. They should be removed and placed immediately adjacent to the specimen. Estimate, in the following order, the value, the chroma, and the hue by interpolation or extrapolation of the notations on the chips, as described in 6.2.3.1 to 6.2.3.3. Interchange the positions of the charts, repeat the estimations, and average the results.

6.2.3.1 *Value*—Find the chips between which the value of the specimen lies. Estimate the value of the specimen to the nearest tenth of the one-value-step interval between adjacent value levels. Record the estimated Munsell value in front of the slant, for example 4.2/.

6.2.3.2 *Chroma*—Move the masks to present successive colors of the same chroma and, by interpolation or extrapolation, determine the Munsell chroma. Pay chief attention to the Munsell chips having values nearest that of the specimen and secondary attention to those next nearest. Although all Munsell chips of the same Munsell chroma are intended to appear to have the same perceptual chroma, a slightly different estimate of chroma may be obtained by comparison with the chips of the next value. In such cases, average the estimated Munsell chromas. Note that there are usually two chroma steps between adjacent columns of a chart. Estimate chroma to the nearest fifth of the 2-chroma interval and record the estimated Munsell chroma after the slant, for example /6.4.

6.2.3.3 *Hue*—Estimate the hue of the specimen by interpolation between the chips of the nearest Munsell value and chroma in the selected hue charts. Estimate to the nearest fifth of the 2.5-hue steps between adjacent hue charts. Record the hue estimate in front of the value-chroma estimate and separated from it by a space, for example 4.5R 4.2/6.4. If the value and chroma of the specimen do not correspond closely to those of any chip, repeat the interpolation of hue with the next closest pair of chips and record the average.

7. Munsell Color Notation from CIE Measurement[7]

NOTE 1—The CIE results for the specimen must be based upon color measurements in which the specular component was excluded, and with

---

[7] Computer programs that convert CIE data to Munsell color notations are available from the Davidson Colleagues, P.O. Box 490, Tatamy, PA 18085; the Munsell Color Company, 2441 N. Calvert St., Baltimore, MD 21218; and Applied Color Systems, P.O. Box 5800, Princeton, NJ 08543.

calculations made using the 1931 2° standard observer and illuminant C.

7.1 *Procedure*—Convert the luminous reflectance, Y, and the chromaticity coordinates, x, y, of the specimen to Munsell color notation by use of Table 1 and Figs. 3 to 16.[8]

NOTE 2—For further information concerning Figs. 3 to 7, 9, 11, 13, and 15 see Newhall, et al.[6] For further information concerning Figs. 8 and 10, see I. Nimeroff.[4]

7.2 In Table 1, find the value, V, equivalent to the luminous reflectance, Y. Use Figs. 3 to 16 to estimate hue and chroma for value levels above and below the value found and linearly interpolate the hues and chromas for the desired value level. If the required value level differs from the nearest level by 0.05 or less, simply use the hue and chroma for the nearest level.

NOTE 3—*Example*—Given the CIE data $Y = 46.02$, $x = 0.500$ and $y = 0.454$, find the Munsell notation.

(*1*) In Table 1, $Y = 46.02$ corresponds to Munsell value 7.28.

(*2*) The value lies between 7 and 8, so the hue and chroma will be found by interpolating these quantities between those found in Figs. 11 and 13. On Fig. 11, $x = 0.500$ and $y = 0.454$ corresponds to a hue of 10.0*YR* and a chroma of 13.1. On Fig. 13, the same x and y correspond to a hue just a small amount redder than 10.0*YR*, an amount less than 0.25 hue step, so the hue is read as 10.0*YR*. The chroma is 14.6.

(*3*) The value is 7.28, which is 0.28 of the way from 7 to 8, so the interpolated hue is that for value 7 plus 0.28 times the difference between the hues found at those two value levels. Since the difference was zero, the interpolated hue is simply the hue found for value 7. The interpolated chroma is found in the same way. The difference in chroma for the two value levels is 14.6–13.1 = 1.5. The difference is multiplied by the interpolation factor: 1.5 × 0.28 = 0.42, which may be rounded to 0.4. This amount is added to the chroma for value level 7: 0.4 + 13.1 = 13.5.

(*4*) The Munsell notation is 10.0*YR* 7.2/13.5.

7.3 *Munsell Notation of Dark Colors*—If the Munsell value is less than 1.0, use the extension of the Munsell system to very dark colors.[9]

7.4 Table 1 was derived from the following relationships:[10]

$$\text{For } Y \leq 0.9\text{: } V = \mathrm{U}Y$$

$$\begin{aligned}\text{For } Y \geq 0.9\text{: } V = {} & \{AY^{1/3}\} - B - \{C/[(DY - E)^2 + F]\} \\ & + \{G/(Y^H)\} + \{J\sin(KY^{1/3} + 1)\} \\ & + \{(M/Y)\sin[N(Y - 2)]\} \\ & - \{[P/(QY)]\sin\,[S(Y - T)]\}\end{aligned}$$

where:

$A = 2.49268$
$B = 1.5614$
$C = 0.985$
$D = 0.1073$
$E = 3.084$
$F = 7.54$
$G = 0.0133$
$H = 2.3$
$J = 0.0084$
$K = 4.1$
$M = 0.0221$
$N = 0.39$
$P = 0.0037$
$Q = 0.44$
$S = 1.28$
$T = 0.53$
$U = 0.87445$
$W = 0.9967$

8. Report

8.1 Report the notation in the Munsell system, specifying whether the notation was obtained visually, using the matte or glossy *Munsell Book of Color*, or by conversion of CIE colorimetric data.

8.1.1 If obtained visually, note the source of illumination (artificial or daylight).

8.1.2 If obtained from colorimetric data, note the instrument used.

9. Precision

9.1 The estimated precision within which a color notation can be determined by visual interpolation is 0.5 hue step, 0.1 value step, and 0.4 chroma step.

10. Keywords

10.1 color; Munsell; Munsell color order system; Munsell notation

---

[8] Figures 8, 10, 12, 14, and 16 are enlargements of the low-chroma areas of Figs. 7, 9, 11, 13, and 15. Large-scale diagrams of Figs. 3 through 16 are available from the Munsell Color Company, 2441 N. Calvert St., Baltimore, MD 21218.

[9] Judd, D. B., and Wyszecki, G., "Extension of the Munsell Renotation System to Very Dark Colors," *Journal, Optical Society of America*, Vol 46, 1956, p. 281.

[10] McCamy, C. S., Macbeth Division of Kollmorgen Instruments Corporation, private communication to ASTM Committee D-1, January, 1987.

TABLE 1 Munsell Value *V* for Given Luminous Reflectance Factor *Y*, in Percent, Relative to the Perfect Reflecting Diffuser

Y	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*
0.01	0.01	0.71	0.62	1.41	1.16	2.11	1.57	2.81	1.90
0.02	0.02	0.72	0.63	1.42	1.17	2.12	1.58	2.82	1.90
0.03	0.03	0.73	0.64	1.43	1.18	2.13	1.58	2.83	1.91
0.04	0.04	0.74	0.65	1.44	1.18	2.14	1.59	2.84	1.91
0.05	0.04	0.75	0.66	1.45	1.19	2.15	1.59	2.85	1.92
0.06	0.05	0.76	0.67	1.46	1.20	2.16	1.60	2.86	1.92
0.07	0.06	0.77	0.67	1.47	1.20	2.17	1.60	2.87	1.92
0.08	0.07	0.78	0.68	1.48	1.21	2.18	1.61	2.88	1.93
0.09	0.08	0.79	0.69	1.49	1.22	2.19	1.61	2.89	1.93
0.10	0.09	0.80	0.70	1.50	1.22	2.20	1.62	2.90	1.94
0.11	0.10	0.81	0.71	1.51	1.23	2.21	1.62	2.91	1.94
0.12	0.11	0.82	0.72	1.52	1.24	2.22	1.63	2.92	1.94
0.13	0.11	0.83	0.73	1.53	1.24	2.23	1.63	2.93	1.95
0.14	0.12	0.84	0.73	1.54	1.25	2.24	1.64	2.94	1.95
0.15	0.13	0.85	0.74	1.55	1.25	2.25	1.64	2.95	1.96
0.16	0.14	0.86	0.75	1.56	1.26	2.26	1.65	2.96	1.96
0.17	0.15	0.87	0.76	1.57	1.27	2.27	1.65	2.97	1.97
0.18	0.16	0.88	0.77	1.58	1.27	2.28	1.66	2.98	1.97
0.19	0.17	0.89	0.78	1.59	1.28	2.29	1.66	2.99	1.97
0.20	0.18	0.90	0.79	1.60	1.29	2.30	1.67	3.00	1.98
0.21	0.18	0.91	0.79	1.61	1.29	2.31	1.67	3.01	1.98
0.22	0.19	0.92	0.80	1.62	1.30	2.32	1.68	3.02	1.99
0.23	0.20	0.93	0.81	1.63	1.30	2.33	1.68	3.03	1.99
0.24	0.21	0.94	0.81	1.64	1.31	2.34	1.69	3.04	1.99
0.25	0.22	0.95	0.82	1.65	1.32	2.35	1.69	3.05	2.00
0.26	0.23	0.96	0.83	1.66	1.32	2.36	1.70	3.06	2.00
0.27	0.24	0.97	0.84	1.67	1.33	2.37	1.70	3.07	2.01
0.28	0.25	0.98	0.85	1.68	1.33	2.38	1.71	3.08	2.01
0.29	0.25	0.99	0.86	1.69	1.34	2.39	1.71	3.09	2.01
0.30	0.26	1.00	0.86	1.70	1.35	2.40	1.72	3.10	2.02
0.31	0.27	1.01	0.87	1.71	1.35	2.41	1.72	3.11	2.02
0.32	0.28	1.02	0.88	1.72	1.36	2.42	1.72	3.12	2.03
0.33	0.29	1.03	0.89	1.73	1.36	2.43	1.73	3.13	2.03
0.34	0.30	1.04	0.90	1.74	1.37	2.44	1.73	3.14	2.03
0.35	0.31	1.05	0.90	1.75	1.38	2.45	1.74	3.15	2.04
0.36	0.32	1.06	0.91	1.76	1.38	2.46	1.74	3.16	2.04
0.37	0.32	1.07	0.92	1.77	1.39	2.47	1.75	3.17	2.05
0.38	0.33	1.08	0.93	1.78	1.39	2.48	1.75	3.18	2.05
0.39	0.34	1.09	0.94	1.79	1.40	2.49	1.76	3.19	2.05
0.40	0.35	1.10	0.94	1.80	1.40	2.50	1.76	3.20	2.06
0.41	0.36	1.11	0.95	1.81	1.41	2.51	1.77	3.21	2.06
0.42	0.37	1.12	0.96	1.82	1.42	2.52	1.77	3.22	2.06
0.43	0.38	1.13	0.97	1.83	1.42	2.53	1.78	3.23	2.07
0.44	0.39	1.14	0.97	1.84	1.43	2.54	1.78	3.24	2.07
0.45	0.39	1.15	0.98	1.85	1.43	2.55	1.78	3.25	2.08
0.46	0.40	1.16	0.99	1.86	1.44	2.56	1.79	3.26	2.08
0.47	0.41	1.17	1.00	1.87	1.44	2.57	1.79	3.27	2.08
0.48	0.42	1.18	1.00	1.88	1.45	2.58	1.80	3.28	2.09
0.49	0.43	1.19	1.01	1.89	1.45	2.59	1.80	3.29	2.09
0.50	0.44	1.20	1.02	1.90	1.46	2.60	1.81	3.30	2.10
0.51	0.45	1.21	1.03	1.91	1.47	2.61	1.81	3.31	2.10
0.52	0.46	1.22	1.03	1.92	1.47	2.62	1.82	3.32	2.10
0.53	0.46	1.23	1.04	1.93	1.48	2.63	1.82	3.33	2.11
0.54	0.47	1.24	1.05	1.94	1.48	2.64	1.82	3.34	2.11
0.55	0.48	1.25	1.05	1.95	1.49	2.65	1.83	3.35	2.11
0.56	0.49	1.26	1.06	1.96	1.49	2.66	1.83	3.36	2.12
0.57	0.50	1.27	1.07	1.97	1.50	2.67	1.84	3.37	2.12
0.58	0.51	1.28	1.08	1.98	1.50	2.68	1.84	3.38	2.13
0.59	0.52	1.29	1.08	1.99	1.51	2.69	1.85	3.39	2.13
0.60	0.53	1.30	1.09	2.00	1.51	2.70	1.85	3.40	2.13
0.61	0.53	1.31	1.10	2.01	1.52	2.71	1.86	3.41	2.14
0.62	0.54	1.32	1.10	2.02	1.53	2.72	1.86	3.42	2.14
0.63	0.55	1.33	1.11	2.03	1.53	2.73	1.86	3.43	2.14
0.64	0.56	1.34	1.12	2.04	1.54	2.74	1.87	3.44	2.15
0.65	0.57	1.35	1.12	2.05	1.54	2.75	1.87	3.45	2.15
0.66	0.58	1.36	1.13	2.06	1.55	2.76	1.88	3.46	2.15
0.67	0.59	1.37	1.14	2.07	1.55	2.77	1.88	3.47	2.16
0.68	0.60	1.38	1.14	2.08	1.56	2.78	1.89	3.48	2.16
0.69	0.60	1.39	1.15	2.09	1.56	2.79	1.89	3.49	2.17
0.70	0.61	1.40	1.16	2.10	1.57	2.80	1.89	3.50	2.17

TABLE 1 *Continued*

Y	V	Y	V	Y	V	Y	V	Y	V
3.51	2.17	4.21	2.41	4.91	2.62	5.61	2.81	6.31	2.98
3.52	2.18	4.22	2.41	4.92	2.62	5.62	2.81	6.32	2.98
3.53	2.18	4.23	2.42	4.93	2.62	5.63	2.81	6.33	2.98
3.54	2.18	4.24	2.42	4.94	2.63	5.64	2.81	6.34	2.99
3.55	2.19	4.25	2.42	4.95	2.63	5.65	2.82	6.35	2.99
3.56	2.19	4.26	2.43	4.96	2.63	5.66	2.82	6.36	2.99
3.57	2.19	4.27	2.43	4.97	2.64	5.67	2.82	6.37	2.99
3.58	2.20	4.28	2.43	4.98	2.64	5.68	2.83	6.38	3.00
3.59	2.20	4.29	2.44	4.99	2.64	5.69	2.83	6.39	3.00
3.60	2.21	4.30	2.44	5.00	2.64	5.70	2.83	6.40	3.00
3.61	2.21	4.31	2.44	5.01	2.65	5.71	2.83	6.41	3.00
3.62	2.21	4.32	2.44	5.02	2.65	5.72	2.84	6.42	3.01
3.63	2.22	4.33	2.45	5.03	2.65	5.73	2.84	6.43	3.01
3.64	2.22	4.34	2.45	5.04	2.66	5.74	2.84	6.44	3.01
3.65	2.22	4.35	2.45	5.05	2.66	5.75	2.84	6.45	3.01
3.66	2.23	4.36	2.46	5.06	2.66	5.76	2.85	6.46	3.01
3.67	2.23	4.37	2.46	5.07	2.66	5.77	2.85	6.47	3.02
3.68	2.23	4.38	2.46	5.08	2.67	5.78	2.85	6.48	3.02
3.69	2.24	4.39	2.47	5.09	2.67	5.79	2.85	6.49	3.02
3.70	2.24	4.40	2.47	5.10	2.67	5.80	2.86	6.50	3.02
3.71	2.24	4.41	2.47	5.11	2.67	5.81	2.86	6.51	3.03
3.72	2.25	4.42	2.48	5.12	2.68	5.82	2.86	6.52	3.03
3.73	2.25	4.43	2.48	5.13	2.68	5.83	2.86	6.53	3.03
3.74	2.25	4.44	2.48	5.14	2.68	5.84	2.87	6.54	3.03
3.75	2.26	4.45	2.48	5.15	2.69	5.85	2.87	6.55	3.04
3.76	2.26	4.46	2.49	5.16	2.69	5.86	2.87	6.56	3.04
3.77	2.26	4.47	2.49	5.17	2.69	5.87	2.87	6.57	3.04
3.78	2.27	4.48	2.49	5.18	2.69	5.88	2.88	6.58	3.04
3.79	2.27	4.49	2.50	5.19	2.70	5.89	2.88	6.59	3.05
3.80	2.28	4.50	2.50	5.20	2.70	5.90	2.88	6.60	3.05
3.81	2.28	4.51	2.50	5.21	2.70	5.91	2.88	6.61	3.05
3.82	2.28	4.52	2.51	5.22	2.70	5.92	2.89	6.62	3.05
3.83	2.29	4.53	2.51	5.23	2.71	5.93	2.89	6.63	3.05
3.84	2.29	4.54	2.51	5.24	2.71	5.94	2.89	6.64	3.06
3.85	2.29	4.55	2.51	5.25	2.71	5.95	2.89	6.65	3.06
3.86	2.30	4.56	2.52	5.26	2.72	5.96	2.90	6.66	3.06
3.87	2.30	4.57	2.52	5.27	2.72	5.97	2.90	6.67	3.06
3.88	2.30	4.58	2.52	5.28	2.72	5.98	2.90	6.68	3.07
3.89	2.31	4.59	2.53	5.29	2.72	5.99	2.90	6.69	3.07
3.90	2.31	4.60	2.53	5.30	2.73	6.00	2.91	6.70	3.07
3.91	2.31	4.61	2.53	5.31	2.73	6.01	2.91	6.71	3.07
3.92	2.32	4.62	2.54	5.32	2.73	6.02	2.91	6.72	3.07
3.93	2.32	4.63	2.54	5.33	2.73	6.03	2.91	6.73	3.08
3.94	2.32	4.64	2.54	5.34	2.74	6.04	2.91	6.74	3.08
3.95	2.33	4.65	2.54	5.35	2.74	6.05	2.92	6.75	3.08
3.96	2.33	4.66	2.55	5.36	2.74	6.06	2.92	6.76	3.08
3.97	2.33	4.67	2.55	5.37	2.74	6.07	2.92	6.77	3.09
3.98	2.34	4.68	2.55	5.38	2.75	6.08	2.92	6.78	3.09
3.99	2.34	4.69	2.56	5.39	2.75	6.09	2.93	6.79	3.09
4.00	2.34	4.70	2.56	5.40	2.75	6.10	2.93	6.80	3.09
4.01	2.35	4.71	2.56	5.41	2.76	6.11	2.93	6.81	3.10
4.02	2.35	4.72	2.56	5.42	2.76	6.12	2.93	6.82	3.10
4.03	2.35	4.73	2.57	5.43	2.76	6.13	2.94	6.83	3.10
4.04	2.36	4.74	2.57	5.44	2.76	6.14	2.94	6.84	3.10
4.05	2.36	4.75	2.57	5.45	2.77	6.15	2.94	6.85	3.10
4.06	2.36	4.76	2.58	5.46	2.77	6.16	2.94	6.86	3.11
4.07	2.37	4.77	2.58	5.47	2.77	6.17	2.95	6.87	3.11
4.08	2.37	4.78	2.58	5.48	2.77	6.18	2.95	6.88	3.11
4.09	2.37	4.79	2.58	5.49	2.78	6.19	2.95	6.89	3.11
4.10	2.37	4.80	2.59	5.50	2.78	6.20	2.95	6.90	3.12
4.11	2.38	4.81	2.59	5.51	2.78	6.21	2.96	6.91	3.12
4.12	2.38	4.82	2.59	5.52	2.78	6.22	2.96	6.92	3.12
4.13	2.38	4.83	2.60	5.53	2.79	6.23	2.96	6.93	3.12
4.14	2.39	4.84	2.60	5.54	2.79	6.24	2.96	6.94	3.12
4.15	2.39	4.85	2.60	5.55	2.79	6.25	2.97	6.95	3.13
4.16	2.39	4.86	2.61	5.56	2.79	6.26	2.97	6.96	3.13
4.17	2.40	4.87	2.61	5.57	2.80	6.27	2.97	6.97	3.13
4.18	2.40	4.88	2.61	5.58	2.80	6.28	2.97	6.98	3.13
4.19	2.40	4.89	2.61	5.59	2.80	6.29	2.97	6.99	3.14
4.20	2.41	4.90	2.62	5.60	2.80	6.30	2.98	7.00	3.14

TABLE 1 *Continued*

Y	V	Y	V	Y	V	Y	V	Y	V
7.01	3.14	7.71	3.29	8.41	3.43	9.11	3.56	9.81	3.69
7.02	3.14	7.72	3.29	8.42	3.43	9.12	3.56	9.82	3.69
7.03	3.14	7.73	3.29	8.43	3.43	9.13	3.57	9.83	3.69
7.04	3.15	7.74	3.30	8.44	3.44	9.14	3.57	9.84	3.69
7.05	3.15	7.75	3.30	8.45	3.44	9.15	3.57	9.85	3.70
7.06	3.15	7.76	3.30	8.46	3.44	9.16	3.57	9.86	3.70
7.07	3.15	7.77	3.30	8.47	3.44	9.17	3.57	9.87	3.70
7.08	3.16	7.78	3.30	8.48	3.44	9.18	3.58	9.88	3.70
7.09	3.16	7.79	3.31	8.49	3.45	9.19	3.58	9.89	3.70
7.10	3.16	7.80	3.31	8.50	3.45	9.20	3.58	9.90	3.70
7.11	3.16	7.81	3.31	8.51	3.45	9.21	3.58	9.91	3.71
7.12	3.16	7.82	3.31	8.52	3.45	9.22	3.58	9.92	3.71
7.13	3.17	7.83	3.31	8.53	3.45	9.23	3.59	9.93	3.71
7.14	3.17	7.84	3.32	8.54	3.46	9.24	3.59	9.94	3.71
7.15	3.17	7.85	3.32	8.55	3.46	9.25	3.59	9.95	3.71
7.16	3.17	7.86	3.32	8.56	3.46	9.26	3.59	9.96	3.71
7.17	3.18	7.87	3.32	8.57	3.46	9.27	3.59	9.97	3.72
7.18	3.18	7.88	3.32	8.58	3.46	9.28	3.59	9.98	3.72
7.19	3.18	7.89	3.33	8.59	3.47	9.29	3.60	9.99	3.72
7.20	3.18	7.90	3.33	8.60	3.47	9.30	3.60	10.00	3.72
7.21	3.18	7.91	3.33	8.61	3.47	9.31	3.60	10.01	3.72
7.22	3.19	7.92	3.33	8.62	3.47	9.32	3.60	10.02	3.72
7.23	3.19	7.93	3.34	8.63	3.47	9.33	3.60	10.03	3.73
7.24	3.19	7.94	3.34	8.64	3.48	9.34	3.60	10.04	3.73
7.25	3.19	7.95	3.34	8.65	3.48	9.35	3.61	10.05	3.73
7.26	3.19	7.96	3.34	8.66	3.48	9.36	3.61	10.06	3.73
7.27	3.20	7.97	3.34	8.67	3.48	9.37	3.61	10.07	3.73
7.28	3.20	7.98	3.35	8.68	3.48	9.38	3.61	10.08	3.73
7.29	3.20	7.99	3.35	8.69	3.48	9.39	3.61	10.09	3.74
7.30	3.20	8.00	3.35	8.70	3.49	9.40	3.62	10.10	3.74
7.31	3.21	8.01	3.35	8.71	3.49	9.41	3.62	10.11	3.74
7.32	3.21	8.02	3.35	8.72	3.49	9.42	3.62	10.12	3.74
7.33	3.21	8.03	3.36	8.73	3.49	9.43	3.62	10.13	3.74
7.34	3.21	8.04	3.36	8.74	3.49	9.44	3.62	10.14	3.74
7.35	3.21	8.05	3.36	8.75	3.50	9.45	3.62	10.15	3.75
7.36	3.22	8.06	3.36	8.76	3.50	9.46	3.63	10.16	3.75
7.37	3.22	8.07	3.36	8.77	3.50	9.47	3.63	10.17	3.75
7.38	3.22	8.08	3.37	8.78	3.50	9.48	3.63	10.18	3.75
7.39	3.22	8.09	3.37	8.79	3.50	9.49	3.63	10.19	3.75
7.40	3.22	8.10	3.37	8.80	3.51	9.50	3.63	10.20	3.76
7.41	3.23	8.11	3.37	8.81	3.51	9.51	3.64	10.21	3.76
7.42	3.23	8.12	3.37	8.82	3.51	9.52	3.64	10.22	3.76
7.43	3.23	8.13	3.38	8.83	3.51	9.53	3.64	10.23	3.76
7.44	3.23	8.14	3.38	8.84	3.51	9.54	3.64	10.24	3.76
7.45	3.24	8.15	3.38	8.85	3.51	9.55	3.64	10.25	3.76
7.46	3.24	8.16	3.38	8.86	3.52	9.56	3.64	10.26	3.77
7.47	3.24	8.17	3.38	8.87	3.52	9.57	3.65	10.27	3.77
7.48	3.24	8.18	3.39	8.88	3.52	9.58	3.65	10.28	3.77
7.49	3.24	8.19	3.39	8.89	3.52	8.59	3.65	10.29	3.77
7.50	3.25	8.20	3.39	8.90	3.52	9.60	3.65	10.30	3.77
7.51	3.25	8.21	3.39	8.91	3.53	9.61	3.65	10.31	3.77
7.52	3.25	8.22	3.39	8.92	3.53	9.62	3.65	10.32	3.78
7.53	3.25	8.23	3.40	8.93	3.53	9.63	3.66	10.33	3.78
7.54	3.25	8.24	3.40	8.94	3.53	9.64	3.66	10.34	3.78
7.55	3.26	8.25	3.40	8.95	3.53	9.65	3.66	10.35	3.78
7.56	3.26	8.26	3.40	8.96	3.54	9.66	3.66	10.36	3.78
7.57	3.26	8.27	3.40	8.97	3.54	9.67	3.66	10.37	3.78
7.58	3.26	8.28	3.41	8.98	3.54	9.68	3.67	10.38	3.79
7.59	3.26	8.29	3.41	8.99	3.54	9.69	3.67	10.39	3.79
7.60	3.27	8.30	3.41	9.00	3.54	9.70	3.67	10.40	3.79
7.61	3.27	8.31	3.41	9.01	3.54	9.71	3.67	10.41	3.79
7.62	3.27	8.32	3.41	9.02	3.55	9.72	3.67	10.42	3.79
7.63	3.27	8.33	3.41	9.03	3.55	9.73	3.67	10.43	3.79
7.64	3.28	8.34	3.42	9.04	3.55	9.74	3.68	10.44	3.80
7.65	3.28	8.35	3.42	9.05	3.55	9.75	3.68	10.45	3.80
7.66	3.28	8.36	3.42	9.06	3.55	9.76	3.68	10.46	3.80
7.67	3.28	8.37	3.42	9.07	3.56	9.77	3.68	10.47	3.80
7.68	3.28	8.38	3.42	9.08	3.56	9.78	3.68	10.48	3.80
7.69	3.29	8.39	3.43	9.09	3.56	9.79	3.68	10.49	3.80
7.70	3.29	8.40	3.43	9.10	3.56	9.80	3.69	10.50	3.81

TABLE 1 *Continued*

Y	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*
10.51	3.81	11.21	3.92	11.91	4.03	12.61	4.14	13.31	4.24
10.52	3.81	11.22	3.92	11.92	4.03	12.62	4.14	13.32	4.24
10.53	3.81	11.23	3.92	11.93	4.03	12.63	4.14	13.33	4.24
10.54	3.81	11.24	3.93	11.94	4.04	12.64	4.14	13.34	4.25
10.55	3.81	11.25	3.93	11.95	4.04	12.65	4.14	13.35	4.25
10.56	3.82	11.26	3.93	11.96	4.04	12.66	4.15	13.36	4.25
10.57	3.82	11.27	3.93	11.97	4.04	12.67	4.15	13.37	4.25
10.58	3.82	11.28	3.93	11.98	4.04	12.68	4.15	13.38	4.25
10.59	3.82	11.29	3.93	11.99	4.04	12.69	4.15	13.39	4.25
10.60	3.82	11.30	3.94	12.00	4.05	12.70	4.15	13.40	4.25
10.61	3.82	11.31	3.94	12.01	4.05	12.71	4.15	13.41	4.26
10.62	3.83	11.32	3.94	12.02	4.05	12.72	4.15	13.42	4.26
10.63	3.83	11.33	3.94	12.03	4.05	12.73	4.16	13.43	4.26
10.64	3.83	11.34	3.94	12.04	4.05	12.74	4.16	13.44	4.26
10.65	3.83	11.35	3.94	12.05	4.05	12.75	4.16	13.45	4.26
10.66	3.83	11.36	3.95	12.06	4.05	12.76	4.16	13.46	4.26
10.67	3.83	11.37	3.95	12.07	4.06	12.77	4.16	13.47	4.26
10.68	3.84	11.38	3.95	12.08	4.06	12.78	4.16	13.48	4.27
10.69	3.84	11.39	3.95	12.09	4.06	12.79	4.16	13.49	4.27
10.70	3.84	11.40	3.95	12.10	4.06	12.80	4.17	13.50	4.27
10.71	3.84	11.41	3.95	12.11	4.06	12.81	4.17	13.51	4.27
10.72	3.84	11.42	3.95	12.12	4.06	12.82	4.17	13.52	4.27
10.73	3.84	11.43	3.96	12.13	4.07	12.83	4.17	13.53	4.27
10.74	3.85	11.44	3.96	12.14	4.07	12.84	4.17	13.54	4.27
10.75	3.85	11.45	3.96	12.15	4.07	12.85	4.17	13.55	4.28
10.76	3.85	11.46	3.96	12.16	4.07	12.86	4.18	13.56	4.28
10.77	3.85	11.47	3.96	12.17	4.07	12.87	4.18	13.57	4.28
10.78	3.85	11.48	3.96	12.18	4.07	12.88	4.18	13.58	4.28
10.79	3.85	11.49	3.97	12.19	4.07	12.89	4.18	13.59	4.28
10.80	3.85	11.50	3.97	12.20	4.08	12.90	4.18	13.60	4.28
10.81	3.86	11.51	3.97	12.21	4.08	12.91	4.18	13.61	4.28
10.82	3.86	11.52	3.97	12.22	4.08	12.92	4.18	13.62	4.29
10.83	3.86	11.53	3.97	12.23	4.08	12.93	4.19	13.63	4.29
10.84	3.86	11.54	3.97	12.24	4.08	12.94	4.19	13.64	4.29
10.85	3.86	11.55	3.98	12.25	4.08	12.95	4.19	13.65	4.29
10.86	3.86	11.56	3.98	12.26	4.09	12.96	4.19	13.66	4.29
10.87	3.87	11.57	3.98	12.27	4.09	12.97	4.19	13.67	4.29
10.88	3.87	11.58	3.98	12.28	4.09	12.98	4.19	13.68	4.29
10.89	3.87	11.59	3.98	12.29	4.09	12.99	4.19	13.69	4.30
10.90	3.87	11.60	3.98	12.30	4.09	13.00	4.20	13.70	4.30
10.91	3.87	11.61	3.98	12.31	4.09	13.01	4.20	13.71	4.30
10.92	3.87	11.62	3.99	12.32	4.09	13.02	4.20	13.72	4.30
10.93	3.88	11.63	3.99	12.33	4.10	13.03	4.20	13.73	4.30
10.94	3.88	11.64	3.99	12.34	4.10	13.04	4.20	13.74	4.30
10.95	3.88	11.65	3.99	12.35	4.10	13.05	4.20	13.75	4.30
10.96	3.88	11.66	3.99	12.36	4.10	13.06	4.20	13.76	4.31
10.97	3.88	11.67	3.99	12.37	4.10	13.07	4.21	13.77	4.31
10.98	3.88	11.68	4.00	12.38	4.10	13.08	4.21	13.78	4.31
10.99	3.89	11.69	4.00	12.39	4.10	13.09	4.21	13.79	4.31
11.00	3.89	11.70	4.00	12.40	4.11	13.10	4.21	13.80	4.31
11.01	3.89	11.71	4.00	12.41	4.11	13.11	4.21	13.81	4.31
11.02	3.89	11.72	4.00	12.42	4.11	13.12	4.21	13.82	4.31
11.03	3.89	11.73	4.00	12.43	4.11	13.13	4.21	13.83	4.32
11.04	3.89	11.74	4.00	12.44	4.11	13.14	4.22	13.84	4.32
11.05	3.90	11.75	4.01	12.45	4.11	13.15	4.22	13.85	4.32
11.06	3.90	11.76	4.01	12.46	4.12	13.16	4.22	13.86	4.32
11.07	3.90	11.77	4.01	12.47	4.12	13.17	4.22	13.87	4.32
11.08	3.90	11.78	4.01	12.48	4.12	13.18	4.22	13.88	4.32
11.09	3.90	11.79	4.01	12.49	4.12	13.19	4.22	13.89	4.32
11.10	3.90	11.80	4.01	12.50	4.12	13.20	4.22	13.90	4.32
11.11	3.91	11.81	4.02	12.51	4.12	13.21	4.23	13.91	4.33
11.12	3.91	11.82	4.02	12.52	4.12	13.22	4.23	13.92	4.33
11.13	3.91	11.83	4.02	12.53	4.13	13.23	4.23	13.93	4.33
11.14	3.91	11.84	4.02	12.54	4.13	13.24	4.23	13.94	4.33
11.15	3.91	11.85	4.02	12.55	4.13	13.25	4.23	13.95	4.33
11.16	3.91	11.86	4.02	12.56	4.13	13.26	4.23	13.96	4.33
11.17	3.91	11.87	4.03	12.57	4.13	13.27	4.24	13.97	4.33
11.18	3.92	11.88	4.03	12.58	4.13	13.28	4.24	13.98	4.34
11.19	3.92	11.89	4.03	12.59	4.13	13.29	4.24	13.99	4.34
11.20	3.92	11.90	4.03	12.60	4.14	13.30	4.24	14.00	4.34

TABLE 1 *Continued*

Y	V	Y	V	Y	V	Y	V	Y	V
14.01	4.34	14.71	4.44	15.41	4.53	16.11	4.62	16.81	4.71
14.02	4.34	14.72	4.44	15.42	4.53	16.12	4.62	16.82	4.71
14.03	4.34	14.73	4.44	15.43	4.53	16.13	4.62	16.83	4.71
14.04	4.34	14.74	4.44	15.44	4.53	16.14	4.62	16.84	4.71
14.05	4.35	14.75	4.44	15.45	4.53	16.15	4.62	16.85	4.71
14.06	4.35	14.76	4.44	15.46	4.54	16.16	4.63	16.86	4.71
14.07	4.35	14.77	4.44	15.47	4.54	16.17	4.63	16.87	4.72
14.08	4.35	14.78	4.45	15.48	4.54	16.18	4.63	16.88	4.72
14.09	4.35	14.79	4.45	15.49	4.54	16.19	4.63	16.89	4.72
14.10	4.35	14.80	4.45	15.50	4.54	16.20	4.63	16.90	4.72
14.11	4.35	14.81	4.45	15.51	4.54	16.21	4.63	16.91	4.72
14.12	4.36	14.82	4.45	15.52	4.54	16.22	4.63	16.92	4.72
14.13	4.36	14.83	4.45	15.53	4.54	16.23	4.64	16.93	4.72
14.14	4.36	14.84	4.45	15.54	4.55	16.24	4.64	16.94	4.72
14.15	4.36	14.85	4.46	15.55	4.55	16.25	4.64	16.95	4.73
14.16	4.36	14.86	4.46	15.56	4.55	16.26	4.64	16.96	4.73
14.17	4.36	14.87	4.46	15.57	4.55	16.27	4.64	16.97	4.73
14.18	4.36	14.88	4.46	15.58	4.55	16.28	4.64	16.98	4.73
14.19	4.37	14.89	4.46	15.59	4.55	16.29	4.64	16.99	4.73
14.20	4.37	14.90	4.46	15.60	4.55	16.30	4.64	17.00	4.73
14.21	4.37	14.91	4.46	15.61	4.56	16.31	4.65	17.01	4.73
14.22	4.37	14.92	4.46	15.62	4.56	16.32	4.65	17.02	4.73
14.23	4.37	14.93	4.47	15.63	4.56	16.33	4.65	17.03	4.74
14.24	4.37	14.94	4.47	15.64	4.56	16.34	4.65	17.04	4.74
14.25	4.37	14.95	4.47	15.65	4.56	16.35	4.65	17.05	4.74
14.26	4.37	14.96	4.47	15.66	4.56	16.36	4.65	17.06	4.74
14.27	4.38	14.97	4.47	15.67	4.56	16.37	4.65	17.07	4.74
14.28	4.38	14.98	4.47	15.68	4.56	16.38	4.65	17.08	4.74
14.29	4.38	14.99	4.47	15.69	4.57	16.39	4.66	17.09	4.74
14.30	4.38	15.00	4.48	15.70	4.57	16.40	4.66	17.10	4.74
14.31	4.38	15.01	4.48	15.71	4.57	16.41	4.66	17.11	4.75
14.32	4.38	15.02	4.48	15.72	4.57	16.42	4.66	17.12	4.75
14.33	4.38	15.03	4.48	15.73	4.57	16.43	4.66	17.13	4.75
14.34	4.39	15.04	4.48	15.74	4.57	16.44	4.66	17.14	4.75
14.35	4.39	15.05	4.48	15.75	4.57	16.45	4.66	17.15	4.75
14.36	4.39	15.06	4.48	15.76	4.57	16.46	4.66	17.16	4.75
14.37	4.39	15.07	4.48	15.77	4.58	16.47	4.67	17.17	4.75
14.38	4.39	15.08	4.49	15.78	4.58	16.48	4.67	17.18	4.75
14.39	4.39	15.09	4.49	15.79	4.58	16.49	4.67	17.19	4.76
14.40	4.39	15.10	4.49	15.80	4.58	16.50	4.67	17.20	4.76
14.41	4.40	15.11	4.49	15.81	4.58	16.51	4.67	17.21	4.76
14.42	4.40	15.12	4.49	15.82	4.58	16.52	4.67	17.22	4.76
14.43	4.40	15.13	4.49	15.83	4.58	16.53	4.67	17.23	4.76
14.44	4.40	15.14	4.49	15.84	4.59	16.54	4.67	17.24	4.76
14.45	4.40	15.15	4.50	15.85	4.59	16.55	4.68	17.25	4.76
14.46	4.40	15.16	4.50	15.86	4.59	16.56	4.68	17.26	4.76
14.47	4.40	15.17	4.50	15.87	4.59	16.57	4.68	17.27	4.77
14.48	4.41	15.18	4.50	15.88	4.59	16.58	4.68	17.28	4.77
14.49	4.41	15.19	4.50	15.89	4.59	16.59	4.68	17.29	4.77
14.50	4.41	15.20	4.50	15.90	4.59	16.60	4.68	17.30	4.77
14.51	4.41	15.21	4.50	15.91	4.59	16.61	4.68	17.31	4.77
14.52	4.41	15.22	4.50	15.92	4.60	16.62	4.68	17.32	4.77
14.53	4.41	15.23	4.51	15.93	4.60	16.63	4.69	17.33	4.77
14.54	4.41	15.24	4.51	15.94	4.60	16.64	4.69	17.34	4.77
14.55	4.41	15.25	4.51	15.95	4.60	16.65	4.69	17.35	4.78
14.56	4.42	15.26	4.51	15.96	4.60	16.66	4.69	17.36	4.78
14.57	4.42	15.27	4.51	15.97	4.60	16.67	4.69	17.37	4.78
14.58	4.42	15.28	4.51	15.98	4.60	16.68	4.69	17.38	4.78
14.59	4.42	15.29	4.51	15.99	4.60	16.69	4.69	17.39	4.78
14.60	4.42	15.30	4.51	16.00	4.61	16.70	4.69	17.40	4.78
14.61	4.42	15.31	4.52	16.01	4.61	16.71	4.70	17.41	4.78
14.62	4.42	15.32	4.52	16.02	4.61	16.72	4.70	17.42	4.78
14.63	4.43	15.33	4.52	16.03	4.61	16.73	4.70	17.43	4.79
14.64	4.43	15.34	4.52	16.04	4.61	16.74	4.70	17.44	4.79
14.65	4.43	15.35	4.52	16.05	4.61	16.75	4.70	17.45	4.79
14.66	4.43	15.36	4.52	16.06	4.61	16.76	4.70	17.46	4.79
14.67	4.43	15.37	4.52	16.07	4.61	16.77	4.70	17.47	4.79
14.68	4.43	15.38	4.53	16.08	4.62	16.78	4.70	17.48	4.79
14.69	4.43	15.39	4.53	16.09	4.62	16.79	4.71	17.49	4.79
14.70	4.43	15.40	4.53	16.10	4.62	16.80	4.71	17.50	4.79

TABLE 1 *Continued*

Y	V	Y	V	Y	V	Y	V	Y	V
17.51	4.79	18.21	4.88	18.91	4.96	19.61	5.04	20.31	5.12
17.52	4.80	18.22	4.88	18.92	4.96	19.62	5.04	20.32	5.12
17.53	4.80	18.23	4.88	18.93	4.96	19.63	5.04	20.33	5.12
17.54	4.80	18.24	4.88	18.94	4.96	19.64	5.04	20.34	5.12
17.55	4.80	18.25	4.88	18.95	4.97	19.65	5.05	20.35	5.12
17.56	4.80	18.26	4.89	18.96	4.97	19.66	5.05	20.36	5.12
17.57	4.80	18.27	4.89	18.97	4.97	19.67	5.05	20.37	5.13
17.58	4.80	18.28	4.89	18.98	4.97	19.68	5.05	20.38	5.13
17.59	4.80	18.29	4.89	18.99	4.97	19.69	5.05	20.39	5.13
17.60	4.81	18.30	4.89	19.00	4.97	19.70	5.05	20.40	5.13
17.61	4.81	18.31	4.89	19.01	4.97	19.71	5.05	20.41	5.13
17.62	4.81	18.32	4.89	19.02	4.97	19.72	5.05	20.42	5.13
17.63	4.81	18.33	4.89	19.03	4.98	19.73	5.05	20.43	5.13
17.64	4.81	18.34	4.89	19.04	4.98	19.74	5.06	20.44	5.13
17.65	4.81	18.35	4.90	19.05	4.98	19.75	5.06	20.45	5.13
17.66	4.81	18.36	4.90	19.06	4.98	19.76	5.06	20.46	5.14
17.67	4.81	18.37	4.90	19.07	4.98	19.77	5.06	20.47	5.14
17.68	4.82	18.38	4.90	19.08	4.98	19.78	5.06	20.48	5.14
17.69	4.82	18.39	4.90	19.09	4.98	19.79	5.06	20.49	5.14
17.70	4.82	18.40	4.90	19.10	4.98	19.80	5.06	20.50	5.14
17.71	4.82	18.41	4.90	19.11	4.98	19.81	5.06	20.51	5.14
17.72	4.82	18.42	4.90	19.12	4.99	19.82	5.07	20.52	5.14
17.73	4.82	18.43	4.91	19.13	4.99	19.83	5.07	20.53	5.14
17.74	4.82	18.44	4.91	19.14	4.99	19.84	5.07	20.54	5.14
17.75	4.82	18.45	4.91	19.15	4.99	19.85	5.07	20.55	5.15
17.76	4.83	18.46	4.91	19.16	4.99	19.86	5.07	20.56	5.15
17.77	4.83	18.47	4.91	19.17	4.99	19.87	5.07	20.57	5.15
17.78	4.83	18.48	4.91	19.18	4.99	19.88	5.07	20.58	5.15
17.79	4.83	18.49	4.91	19.19	4.99	19.89	5.07	20.59	5.15
17.80	4.83	18.50	4.91	19.20	4.99	19.90	5.07	20.60	5.15
17.81	4.83	18.51	4.91	19.21	5.00	19.91	5.08	20.61	5.15
17.82	4.83	18.52	4.92	19.22	5.00	19.92	5.08	20.62	5.15
17.83	4.83	18.53	4.92	19.23	5.00	19.93	5.08	20.63	5.15
17.84	4.83	18.54	4.92	19.24	5.00	19.94	5.08	20.64	5.16
17.85	4.84	18.55	4.92	19.25	5.00	19.95	5.08	20.65	5.16
17.86	4.84	18.56	4.92	19.26	5.00	19.96	5.08	20.66	5.16
17.87	4.84	18.57	4.92	19.27	5.00	19.97	5.08	20.67	5.16
17.88	4.84	18.58	4.92	19.28	5.00	19.98	5.08	20.68	5.16
17.89	4.84	18.59	4.92	19.29	5.01	19.99	5.08	20.69	5.16
17.90	4.84	18.60	4.93	19.30	5.01	20.00	5.09	20.70	5.16
17.91	4.84	18.61	4.93	19.31	5.01	20.01	5.09	20.71	5.16
17.92	4.84	18.62	4.93	19.32	5.01	20.02	5.09	20.72	5.16
17.93	4.85	18.63	4.93	19.33	5.01	20.03	5.09	20.73	5.17
17.94	4.85	18.64	4.93	19.34	5.01	20.04	5.09	20.74	5.17
17.95	4.85	18.65	4.93	19.35	5.01	20.05	5.09	20.75	5.17
17.96	4.85	18.66	4.93	19.36	5.01	20.06	5.09	20.76	5.17
17.97	4.85	18.67	4.93	19.37	5.01	20.07	5.09	20.77	5.17
17.98	4.85	18.68	4.93	19.38	5.02	20.08	5.09	20.78	5.17
17.99	4.85	18.69	4.94	19.39	5.02	20.09	5.10	20.79	5.17
18.00	4.85	18.70	4.94	19.40	5.02	20.10	5.10	20.80	5.17
18.01	4.86	18.71	4.94	19.41	5.02	20.11	5.10	20.81	5.17
18.02	4.86	18.72	4.94	19.42	5.02	20.12	5.10	20.82	5.18
18.03	4.86	18.73	4.94	19.43	5.02	20.13	5.10	20.83	5.18
18.04	4.86	18.74	4.94	19.44	5.02	20.14	5.10	20.84	5.18
18.05	4.86	18.75	4.94	19.45	5.02	20.15	5.10	20.85	5.18
18.06	4.86	18.76	4.94	19.46	5.02	20.16	5.10	20.86	5.18
18.07	4.86	18.77	4.95	19.47	5.03	20.17	5.10	20.87	5.18
18.08	4.86	18.78	4.95	19.48	5.03	20.18	5.11	20.88	5.18
18.09	4.86	18.79	4.95	19.49	5.03	20.19	5.11	20.89	5.18
18.10	4.87	18.80	4.95	19.50	5.03	20.20	5.11	20.90	5.18
18.11	4.87	18.81	4.95	19.51	5.03	20.21	5.11	20.91	5.18
18.12	4.87	18.82	4.95	19.52	5.03	20.22	5.11	20.92	5.19
18.13	4.87	18.83	4.95	19.53	5.03	20.23	5.11	20.93	5.19
18.14	4.87	18.84	4.95	19.54	5.03	20.24	5.11	20.94	5.19
18.15	4.87	18.85	4.95	19.55	5.03	20.25	5.11	20.95	5.19
18.16	4.87	18.86	4.96	19.56	5.04	20.26	5.11	20.96	5.19
18.17	4.87	18.87	4.96	19.57	5.04	20.27	5.11	20.97	5.19
18.18	4.88	18.88	4.96	19.58	5.04	20.28	5.12	20.98	5.19
18.19	4.88	18.89	4.96	19.59	5.04	20.29	5.12	20.99	5.19
18.20	4.88	18.90	4.96	19.60	5.04	20.30	5.12	21.00	5.19

TABLE 1 *Continued*

Y	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*
21.01	5.20	21.71	5.27	22.41	5.34	23.11	5.42	23.81	5.49
21.02	5.20	21.72	5.27	22.42	5.35	23.12	5.42	23.82	5.49
21.03	5.20	21.73	5.27	22.43	5.35	23.13	5.42	23.83	5.49
21.04	5.20	21.74	5.27	22.44	5.35	23.14	5.42	23.84	5.49
21.05	5.20	21.75	5.27	22.45	5.35	23.15	5.42	23.85	5.49
21.06	5.20	21.76	5.28	22.46	5.35	23.16	5.42	23.86	5.49
21.07	5.20	21.77	5.28	22.47	5.35	23.17	5.42	23.87	5.49
21.08	5.20	21.78	5.28	22.48	5.35	23.18	5.42	23.88	5.49
21.09	5.20	21.79	5.28	22.49	5.35	23.19	5.42	23.89	5.49
21.10	5.21	21.80	5.28	22.50	5.35	23.20	5.43	23.90	5.50
21.11	5.21	21.81	5.28	22.51	5.35	23.21	5.43	23.91	5.50
21.12	5.21	21.82	5.28	22.52	5.36	23.22	5.43	23.92	5.50
21.13	5.21	21.83	5.28	22.53	5.36	23.23	5.43	23.93	5.50
21.14	5.21	21.84	5.28	22.54	5.36	23.24	5.43	23.94	5.50
21.15	5.21	21.85	5.29	22.55	5.36	23.25	5.43	23.95	5.50
21.16	5.21	21.86	5.29	22.56	5.36	23.26	5.43	23.96	5.50
21.17	5.21	21.87	5.29	22.57	5.36	23.27	5.43	23.97	5.50
21.18	5.21	21.88	5.29	22.58	5.36	23.28	5.43	23.98	5.50
21.19	5.21	21.89	5.29	22.59	5.36	23.29	5.43	23.99	5.50
21.20	5.22	21.90	5.29	22.60	5.36	23.30	5.44	24.0	5.51
21.21	5.22	21.91	5.29	22.61	5.36	23.31	5.44	24.1	5.52
21.22	5.22	21.92	5.29	22.62	5.37	23.32	5.44	24.2	5.53
21.23	5.22	21.93	5.29	22.63	5.37	23.33	5.44	24.3	5.54
21.24	5.22	21.94	5.29	22.64	5.37	23.34	5.44	24.4	5.55
21.25	5.22	21.95	5.30	22.65	5.37	23.35	5.44	24.5	5.55
21.26	5.22	21.96	5.30	22.66	5.37	23.36	5.44	24.6	5.56
21.27	5.22	21.97	5.30	22.67	5.37	23.37	5.44	24.7	5.57
21.28	5.22	21.98	5.30	22.68	5.37	23.38	5.44	24.8	5.58
21.29	5.23	21.99	5.30	22.69	5.37	23.39	5.44	24.9	5.59
21.30	5.23	22.00	5.30	22.70	5.37	23.40	5.45	25.0	5.60
21.31	5.23	22.01	5.30	22.71	5.38	23.41	5.45	25.1	5.61
21.32	5.23	22.02	5.30	22.72	5.38	23.42	5.45	25.2	5.62
21.33	5.23	22.03	5.30	22.73	5.38	23.43	5.45	25.3	5.63
21.34	5.23	22.04	5.31	22.74	5.38	23.44	5.45	25.4	5.64
21.35	5.23	22.05	5.31	22.75	5.38	23.45	5.45	25.5	5.65
21.36	5.23	22.06	5.31	22.76	5.38	23.46	5.45	25.6	5.66
21.37	5.23	22.07	5.31	22.77	5.38	23.47	5.45	25.7	5.67
21.38	5.24	22.08	5.31	22.78	5.38	23.48	5.45	25.8	5.68
21.39	5.24	22.09	5.31	22.79	5.38	23.49	5.45	25.9	5.69
21.40	5.24	22.10	5.31	22.80	5.38	23.50	5.46	26.0	5.70
21.41	5.24	22.11	5.31	22.81	5.39	23.51	5.46	26.1	5.71
21.42	5.24	22.12	5.31	22.82	5.39	23.52	5.46	26.2	5.72
21.43	5.24	22.13	5.31	22.83	5.39	23.53	5.46	26.3	5.73
21.44	5.24	22.14	5.32	22.84	5.39	23.54	5.46	26.4	5.74
21.45	5.24	22.15	5.32	22.85	5.39	23.55	5.46	26.5	5.75
21.46	5.24	22.16	5.32	22.86	5.39	23.56	5.46	26.6	5.75
21.47	5.24	22.17	5.32	22.87	5.39	23.57	5.46	26.7	5.76
21.48	5.25	22.18	5.32	22.88	5.39	23.58	5.46	26.8	5.77
21.49	5.25	22.19	5.32	22.89	5.39	23.59	5.46	26.9	5.78
21.50	5.25	22.20	5.32	22.90	5.39	23.60	5.47	27.0	5.79
21.51	5.25	22.21	5.32	22.91	5.40	23.61	5.47	27.1	5.80
21.52	5.25	22.22	5.32	22.92	5.40	23.62	5.47	27.2	5.81
21.53	5.25	22.23	5.33	22.93	5.40	23.63	5.47	27.3	5.82
21.54	5.25	22.24	5.33	22.94	5.40	23.64	5.47	27.4	5.83
21.55	5.25	22.25	5.33	22.95	5.40	23.65	5.47	27.5	5.84
21.56	5.25	22.26	5.33	22.96	5.40	23.66	5.47	27.6	5.85
21.57	5.26	22.27	5.33	22.97	5.40	23.67	5.47	27.7	5.86
21.58	5.26	22.28	5.33	22.98	5.40	23.68	5.47	27.8	5.87
21.59	5.26	22.29	5.33	22.99	5.40	23.69	5.47	27.9	5.87
21.60	5.26	22.30	5.33	23.00	5.40	23.70	5.48	28.0	5.88
21.61	5.26	22.31	5.33	23.01	5.41	23.71	5.48	28.1	5.89
21.62	5.26	22.32	5.33	23.02	5.41	23.72	5.48	28.2	5.90
21.63	5.26	22.33	5.34	23.03	5.41	23.73	5.48	28.3	5.91
21.64	5.26	22.34	5.34	23.04	5.41	23.74	5.48	28.4	5.92
21.65	5.26	22.35	5.34	23.05	5.41	23.75	5.48	28.5	5.93
21.66	5.27	22.36	5.34	23.06	5.41	23.76	5.48	28.6	5.94
21.67	5.27	22.37	5.34	23.07	5.41	23.77	5.48	28.7	5.95
21.68	5.27	22.38	5.34	23.08	5.41	23.78	5.48	28.8	5.96
21.69	5.27	22.39	5.34	23.09	5.41	23.79	5.48	28.9	5.96
21.70	5.27	22.40	5.34	23.10	5.42	23.80	5.49	29.0	5.97

TABLE 1 *Continued*

Y	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*
29.1	5.98	36.1	6.56	43.1	7.08	50.1	7.55	57.1	7.97
29.2	5.99	36.2	6.57	43.2	7.09	50.2	7.55	57.2	7.98
29.3	6.00	36.3	6.58	43.3	7.10	50.3	7.56	57.3	7.98
29.4	6.01	36.4	6.59	43.4	7.10	50.4	7.57	57.4	7.99
29.5	6.02	36.5	6.60	43.5	7.11	50.5	7.57	57.5	7.99
29.6	6.03	36.6	6.60	43.6	7.12	50.6	7.58	57.6	8.00
29.7	6.03	36.7	6.61	43.7	7.12	50.7	7.59	57.7	8.01
29.8	6.04	36.8	6.62	43.8	7.13	50.8	7.59	57.8	8.01
29.9	6.05	36.9	6.63	43.9	7.14	50.9	7.60	57.9	8.02
30.0	6.06	37.0	6.63	44.0	7.14	51.0	7.60	58.0	8.02
30.1	6.07	37.1	6.64	44.1	7.15	51.1	7.61	58.1	8.03
30.2	6.08	37.2	6.65	44.2	7.16	51.2	7.62	58.2	8.03
30.3	6.09	37.3	6.66	44.3	7.16	51.3	7.62	58.3	8.04
30.4	6.10	37.4	6.67	44.4	7.17	51.4	7.63	58.4	8.05
30.5	6.10	37.5	6.67	44.5	7.18	51.5	7.64	58.5	8.05
30.6	6.11	37.6	6.68	44.6	7.19	51.6	7.64	58.6	8.06
30.7	6.12	37.7	6.69	44.7	7.19	51.7	7.65	58.7	8.06
30.8	6.13	37.8	6.70	44.8	7.20	51.8	7.65	58.8	8.07
30.9	6.14	37.9	6.70	44.9	7.21	51.9	7.66	58.9	8.07
31.0	6.15	38.0	6.71	45.0	7.21	52.0	7.67	59.0	8.08
31.1	6.16	38.1	6.72	45.1	7.22	52.1	7.67	59.1	8.09
31.2	6.16	38.2	6.73	45.2	7.23	52.2	7.68	59.2	8.09
31.3	6.17	38.3	6.73	45.3	7.23	52.3	7.69	59.3	8.10
31.4	6.18	38.4	6.74	45.4	7.24	52.4	7.69	59.4	8.10
31.5	6.19	38.5	6.75	45.5	7.25	52.5	7.70	59.5	8.11
31.6	6.20	38.6	6.76	45.6	7.25	52.6	7.70	59.6	8.11
31.7	6.21	38.7	6.76	45.7	7.26	52.7	7.71	59.7	8.12
31.8	6.21	38.8	6.77	45.8	7.27	52.8	7.72	59.8	8.13
31.9	6.22	38.9	6.78	45.9	7.27	52.9	7.72	59.9	8.13
32.0	6.23	39.0	6.79	46.0	7.28	53.0	7.73	60.0	8.14
32.1	6.24	39.1	6.79	46.1	7.29	53.1	7.73	60.1	8.14
32.2	6.25	39.2	6.80	46.2	7.29	53.2	7.74	60.2	8.15
32.3	6.26	39.3	6.81	46.3	7.30	53.3	7.75	60.3	8.15
32.4	6.27	39.4	6.82	46.4	7.31	53.4	7.75	60.4	8.16
32.5	6.27	39.5	6.82	46.5	7.31	53.5	7.76	60.5	8.16
32.6	6.28	39.6	6.83	46.6	7.32	53.6	7.76	60.6	8.17
32.7	6.29	39.7	6.84	46.7	7.33	53.7	7.77	60.7	8.18
32.8	6.30	39.8	6.85	46.8	7.33	53.8	7.78	60.8	8.18
32.9	6.31	39.9	6.85	46.9	7.34	53.9	7.78	60.9	8.19
33.0	6.32	40.0	6.86	47.0	7.35	54.0	7.79	61.0	8.19
33.1	6.32	40.1	6.87	47.1	7.35	54.1	7.79	61.1	8.20
33.2	6.33	40.2	6.87	47.2	7.36	54.2	7.80	61.2	8.20
33.3	6.34	40.3	6.88	47.3	7.37	54.3	7.81	61.3	8.21
33.4	6.35	40.4	6.89	47.4	7.37	54.4	7.81	61.4	8.21
33.5	6.36	40.5	6.90	47.5	7.38	54.5	7.82	61.5	8.22
33.6	6.36	40.6	6.90	47.6	7.39	54.6	7.82	61.6	8.23
33.7	6.37	40.7	6.91	47.7	7.39	54.7	7.83	61.7	8.23
33.8	6.38	40.8	6.92	47.8	7.40	54.8	7.84	61.8	8.24
33.9	6.39	40.9	6.93	47.9	7.41	54.9	7.84	61.9	8.24
34.0	6.40	41.0	6.93	48.0	7.41	55.0	7.85	62.0	8.25
34.1	6.41	41.1	6.94	48.1	7.42	55.1	7.85	62.1	8.25
34.2	6.41	41.2	6.95	48.2	7.43	55.2	7.86	62.2	8.26
34.3	6.42	41.3	6.95	48.3	7.43	55.3	7.87	62.3	8.26
34.4	6.43	41.4	6.96	48.4	7.44	55.4	7.87	62.4	8.27
34.5	6.44	41.5	6.97	48.5	7.44	55.5	7.88	62.5	8.27
34.6	6.45	41.6	6.98	48.6	7.45	55.6	7.88	62.6	8.28
34.7	6.45	41.7	6.98	48.7	7.46	55.7	7.89	62.7	8.29
34.8	6.46	41.8	6.99	48.8	7.46	55.8	7.90	62.8	8.29
34.9	6.47	41.9	7.00	48.9	7.47	55.9	7.90	62.9	8.30
35.0	6.48	42.0	7.00	49.0	7.48	56.0	7.91	63.0	8.30
35.1	6.49	42.1	7.01	49.1	7.48	56.1	7.91	63.1	8.31
35.2	6.49	42.2	7.02	49.2	7.49	56.2	7.92	63.2	8.31
35.3	6.50	42.3	7.03	49.3	7.50	56.3	7.92	63.3	8.32
35.4	6.51	42.4	7.03	49.4	7.50	56.4	7.93	63.4	8.32
35.5	6.52	42.5	7.04	49.5	7.51	56.5	7.94	63.5	8.33
35.6	6.52	42.6	7.05	49.6	7.52	56.6	7.94	63.6	8.33
35.7	6.53	42.7	7.05	49.7	7.52	56.7	7.95	63.7	8.34
35.8	6.54	42.8	7.06	49.8	7.53	56.8	7.95	63.8	8.34
35.9	6.55	42.9	7.07	49.9	7.53	56.9	7.96	63.9	8.35
36.0	6.56	43.0	7.07	50.0	7.54	57.0	7.97	64.0	8.36

TABLE 1 *Continued*

Y	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*	*Y*	*V*
64.1	8.36	71.3	8.73	78.5	9.08	85.7	9.41	92.9	9.72
64.2	8.37	71.4	8.74	78.6	9.09	85.8	9.41	93.0	9.72
64.3	8.37	71.5	8.74	78.7	9.09	85.9	9.42	93.1	9.72
64.4	8.38	71.6	8.75	78.8	9.10	86.0	9.42	93.2	9.73
64.5	8.38	71.7	8.75	78.9	9.10	86.1	9.43	93.3	9.73
64.6	8.39	71.8	8.76	79.0	9.10	86.2	9.43	93.4	9.74
64.7	8.39	71.9	8.76	79.1	9.11	86.3	9.43	93.5	9.74
64.8	8.40	72.0	8.77	79.2	9.11	86.4	9.44	93.6	9.74
64.9	8.40	72.1	8.77	79.3	9.12	86.5	9.44	93.7	9.75
65.0	8.41	72.2	8.78	79.4	9.12	86.6	9.45	93.8	9.75
65.1	8.41	72.3	8.78	79.5	9.13	86.7	9.45	93.9	9.76
65.2	8.42	72.4	8.79	79.6	9.13	86.8	9.46	94.0	9.76
65.3	8.42	72.5	8.79	79.7	9.14	86.9	9.46	94.1	9.76
65.4	8.43	72.6	8.80	79.8	9.14	87.0	9.47	94.2	9.77
65.5	8.44	72.7	8.80	79.9	9.15	87.1	9.47	94.3	9.77
65.6	8.44	72.8	8.81	80.0	9.15	87.2	9.47	94.4	9.78
65.7	8.45	72.9	8.81	80.1	9.16	87.3	9.48	94.5	9.78
65.8	8.45	73.0	8.82	80.2	9.16	87.4	9.48	94.6	9.79
65.9	8.46	73.1	8.82	80.3	9.17	87.5	9.49	94.7	9.79
66.0	8.46	73.2	8.83	80.4	9.17	87.6	9.49	94.8	9.79
66.1	8.47	73.3	8.83	80.5	9.17	87.7	9.50	94.9	9.80
66.2	8.47	73.4	8.84	80.6	9.18	87.8	9.50	95.0	9.80
66.3	8.48	73.5	8.84	80.7	9.18	87.9	9.50	95.1	9.81
66.4	8.48	73.6	8.85	80.8	9.19	88.0	9.51	95.2	9.81
66.5	8.49	73.7	8.85	80.9	9.19	88.1	9.51	95.3	9.81
66.6	8.49	73.8	8.86	81.0	9.20	88.2	9.52	95.4	9.82
66.7	8.50	73.9	8.86	81.1	9.20	88.3	9.52	95.5	9.82
66.8	8.50	74.0	8.87	81.2	9.21	88.4	9.53	95.6	9.83
66.9	8.51	74.1	8.87	81.3	9.21	88.5	9.53	95.7	9.83
67.0	8.51	74.2	8.88	81.4	9.22	88.6	9.53	95.8	9.83
67.1	8.52	74.3	8.88	81.5	9.22	88.7	9.54	95.9	9.84
67.2	8.53	74.4	8.89	81.6	9.22	88.8	9.54	96.0	9.84
67.3	8.53	74.5	8.89	81.7	9.23	88.9	9.55	96.1	9.85
67.4	8.54	74.6	8.90	81.8	9.23	89.0	9.55	96.2	9.85
67.5	8.54	74.7	8.90	81.9	9.24	89.1	9.56	96.3	9.85
67.6	8.55	74.8	8.91	82.0	9.24	89.2	9.56	96.4	9.86
67.7	8.55	74.9	8.91	82.1	9.25	89.3	9.56	96.5	9.86
67.8	8.56	75.0	8.92	82.2	9.25	89.4	9.57	96.6	9.87
67.9	8.56	75.1	8.92	82.3	9.26	89.5	9.57	96.7	9.87
68.0	8.57	75.2	8.93	82.4	9.26	89.6	9.58	96.8	9.87
68.1	8.57	75.3	8.93	82.5	9.27	89.7	9.58	96.9	9.88
68.2	8.58	75.4	8.93	82.6	9.27	89.8	9.59	97.0	9.88
68.3	8.58	75.5	8.94	82.7	9.27	89.9	9.59	97.1	9.89
68.4	8.59	75.6	8.94	82.8	9.28	90.0	9.59	97.2	9.89
68.5	8.59	75.7	8.95	82.9	9.28	90.1	9.60	97.3	9.89
68.6	8.60	75.8	8.95	83.0	9.29	90.2	9.60	97.4	9.90
68.7	8.60	75.9	8.96	83.1	9.29	90.3	9.61	97.5	9.90
68.8	8.61	76.0	8.96	83.2	9.30	90.4	9.61	97.6	9.91
68.9	8.61	76.1	8.97	83.3	9.30	90.5	9.62	97.7	9.91
69.0	8.62	76.2	8.97	83.4	9.31	90.6	9.62	97.8	9.91
69.1	8.62	76.3	8.98	83.5	9.31	90.7	9.62	97.9	9.92
69.2	8.63	76.4	8.98	83.6	9.32	90.8	9.63	98.0	9.92
69.3	8.63	76.5	8.99	83.7	9.32	90.9	9.63	98.1	9.93
69.4	8.64	76.6	8.99	83.8	9.32	91.0	9.64	98.2	9.93
69.5	8.64	76.7	9.00	83.9	9.33	91.1	9.64	98.3	9.93
69.6	8.65	76.8	9.00	84.0	9.33	91.2	9.64	98.4	9.94
69.7	8.65	76.9	9.01	84.1	9.34	91.3	9.65	98.5	9.94
69.8	8.66	77.0	9.01	84.2	9.34	91.4	9.65	98.6	9.95
69.9	8.66	77.1	9.02	84.3	9.35	91.5	9.66	98.7	9.95
70.0	8.67	77.2	9.02	84.4	9.35	91.6	9.66	98.8	9.95
70.1	8.67	77.3	9.03	84.5	9.36	91.7	9.67	98.9	9.96
70.2	8.68	77.4	9.03	84.6	9.36	91.8	9.67	99.0	9.96
70.3	8.68	77.5	9.03	84.7	9.36	91.9	9.67	99.1	9.97
70.4	8.69	77.6	9.04	84.8	9.37	92.0	9.68	99.2	9.97
70.5	8.69	77.7	9.04	84.9	9.37	92.1	9.68	99.3	9.97
70.6	8.70	77.8	9.05	85.0	9.38	92.2	9.69	99.4	9.98
70.7	8.70	77.9	9.05	85.1	9.38	92.3	9.69	99.5	9.98
70.8	8.71	78.0	9.06	85.2	9.39	92.4	9.69	99.6	9.99
70.9	8.71	78.1	9.06	85.3	9.39	92.5	9.70	99.7	9.99
71.0	8.72	78.2	9.07	85.4	9.40	92.6	9.70	99.8	9.99
71.1	8.72	78.3	9.07	85.5	9.40	92.7	9.71	99.9	10.00
71.2	8.73	78.4	9.08	85.6	9.40	92.8	9.71	100.0	10.00

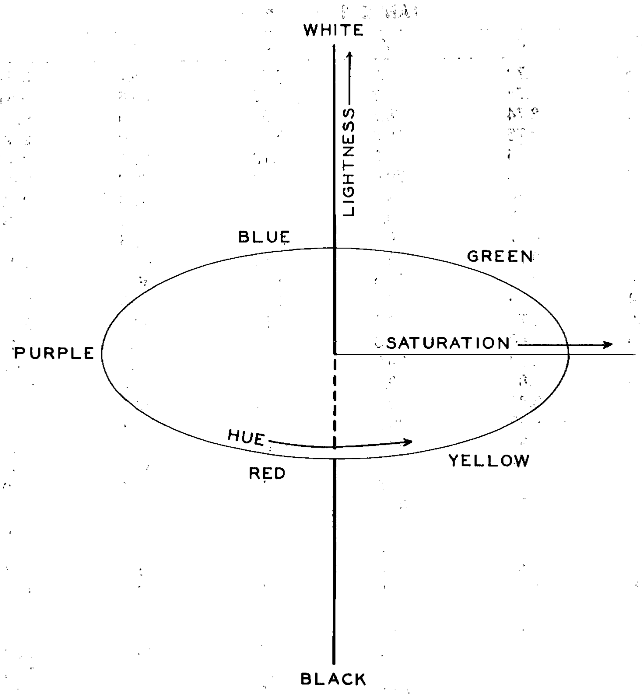


FIG. 1 Dimensions of the Surface-Color-Perception Solid

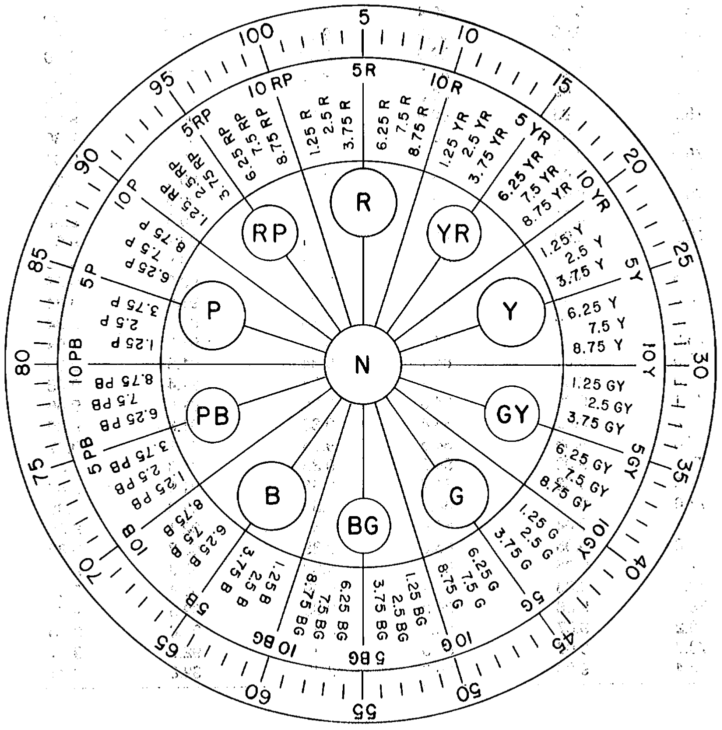


FIG. 2 Designation Systems for Munsell Hue

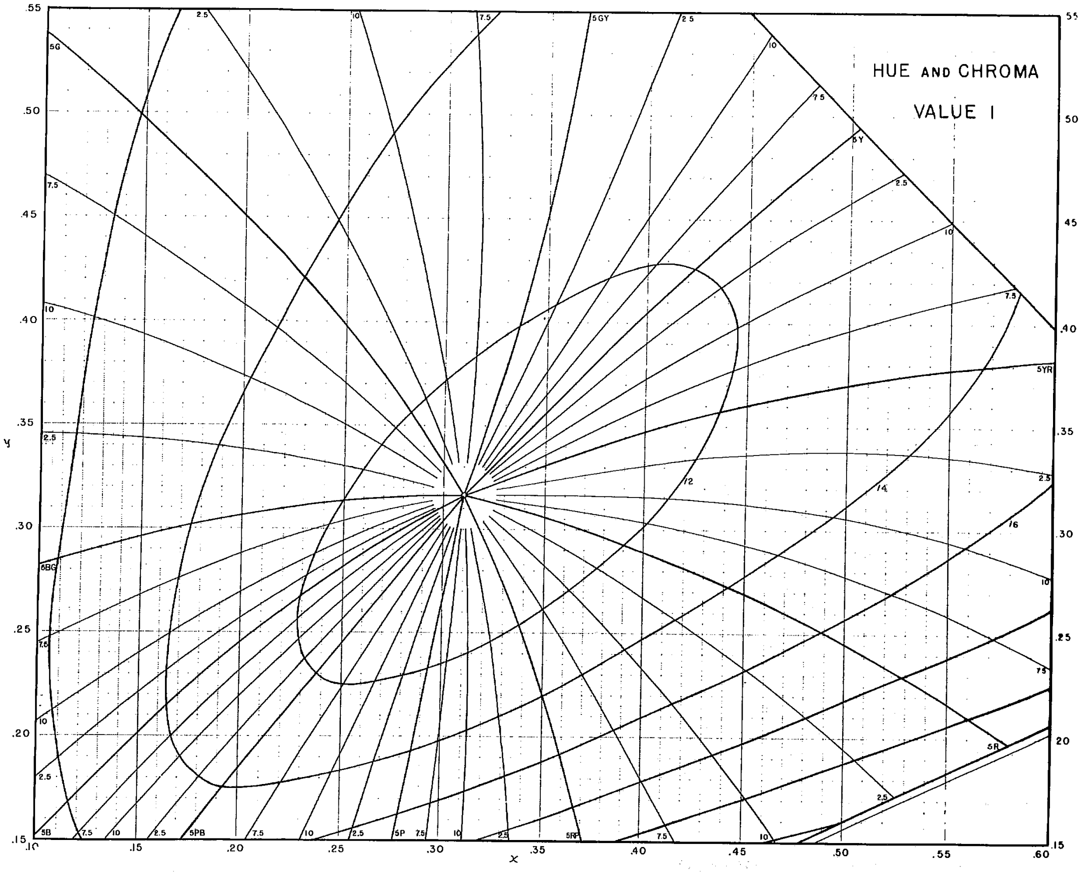


FIG. 3 Munsell Value 1—Loci of Constant Hue and Constant Chroma in CIE (*x*,*y*) Coordinates

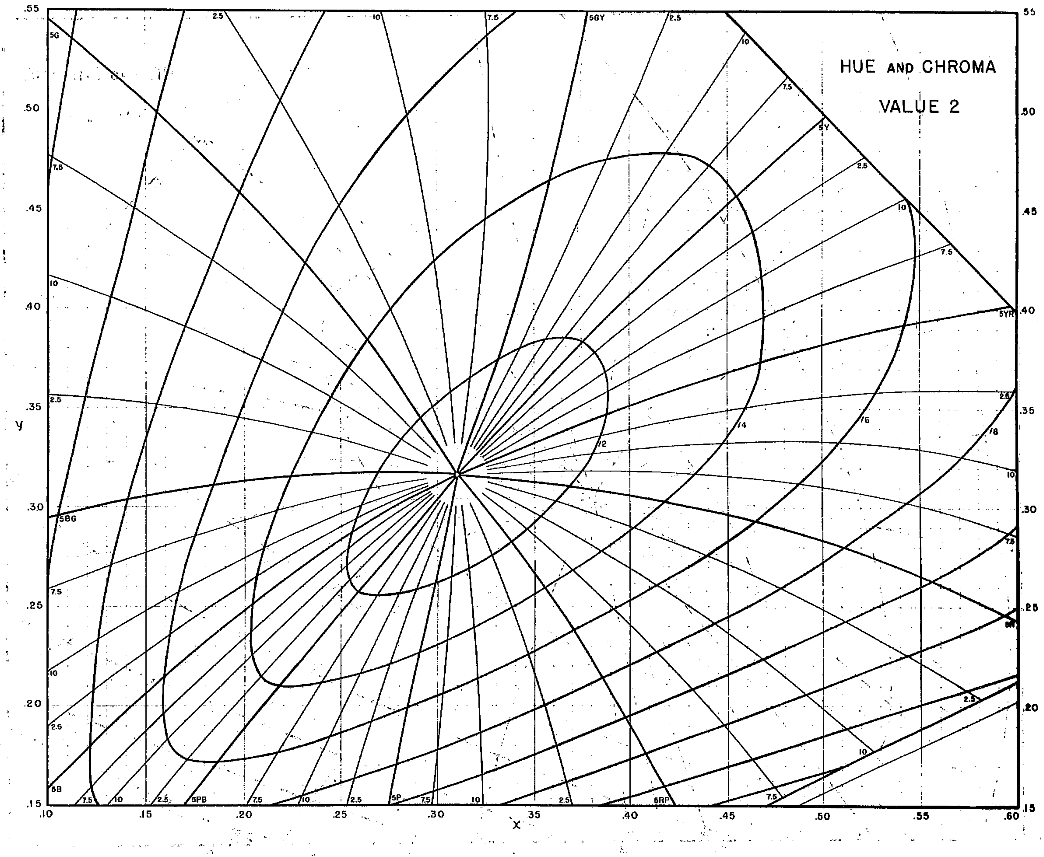


FIG. 4 Munsell Value 2—Loci of Constant Hue and Constant Chroma in CIE (*x*,*y*) Coordinates

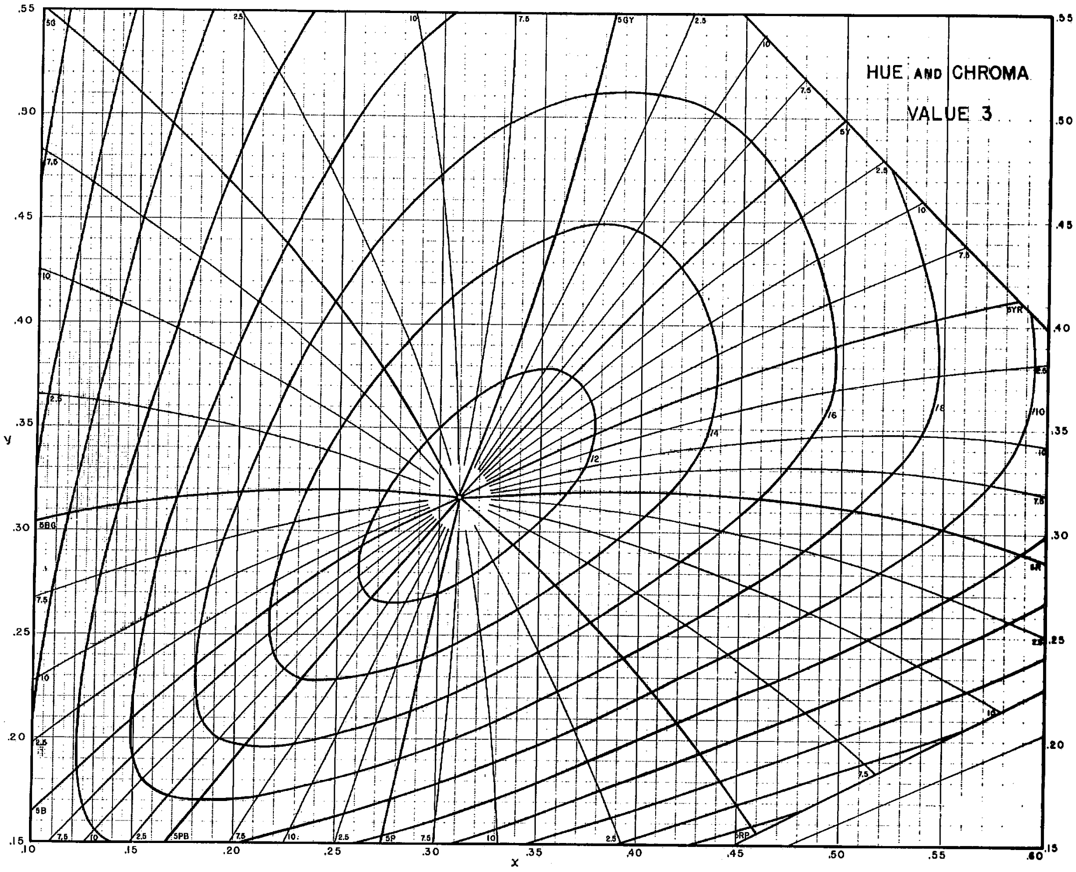


FIG. 5 Munsell Value 3—Loci of Constant Hue and Constant Chroma in CIE (x,y) Coordinates

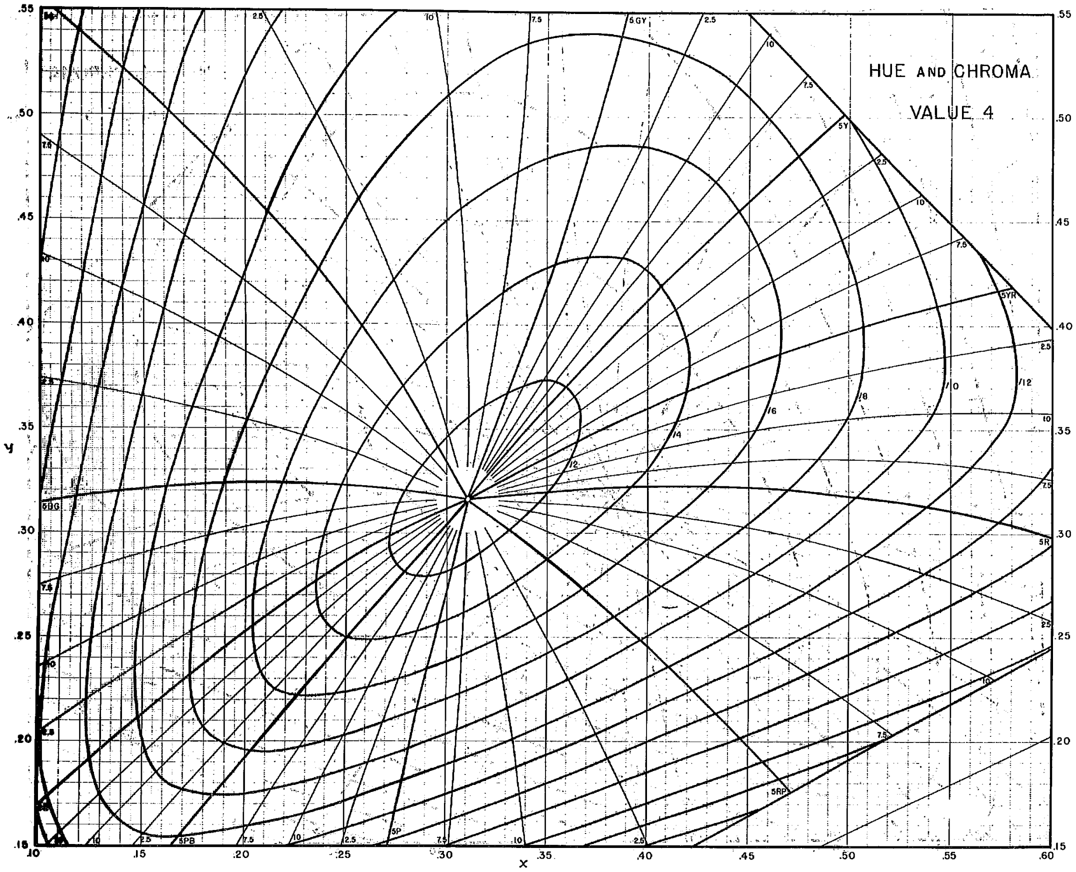


FIG. 6 Munsell Value 4—Loci of Constant Hue and Constant Chroma in CIE (x,y) Coordinates

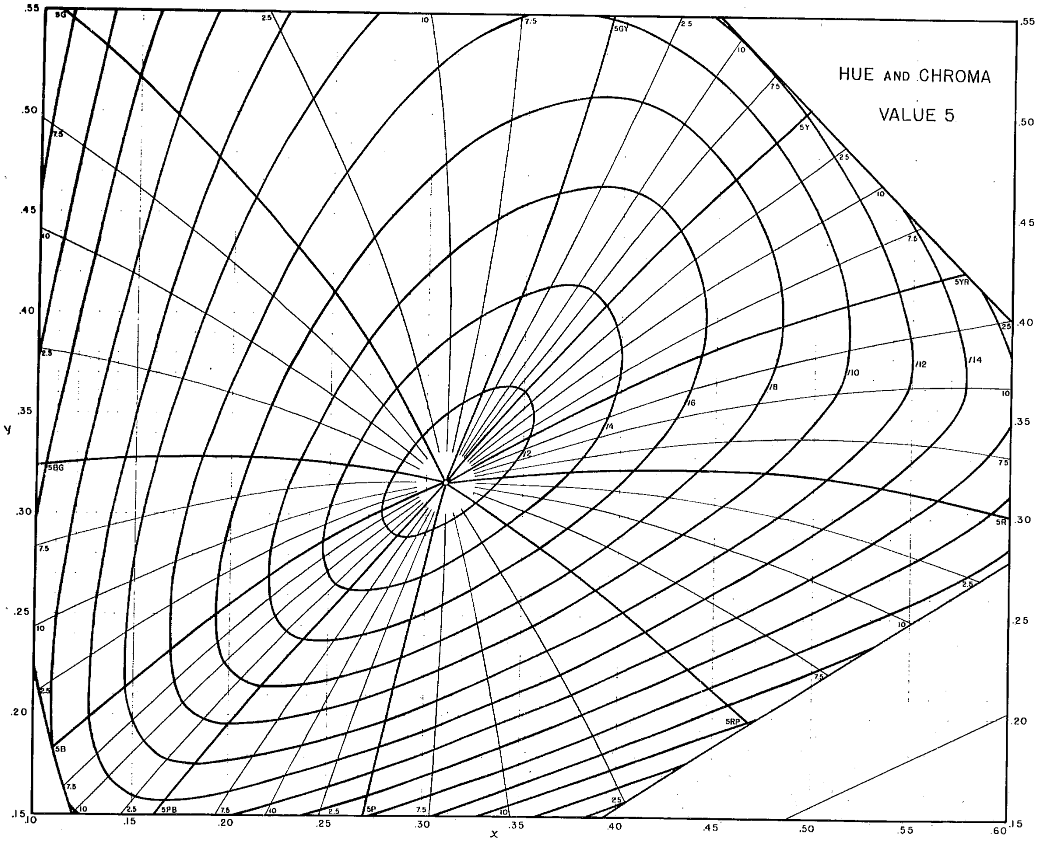


FIG. 7 Munsell Value 5—Loci of Constant Chroma in CIE (*x*,*y*) Coordinates

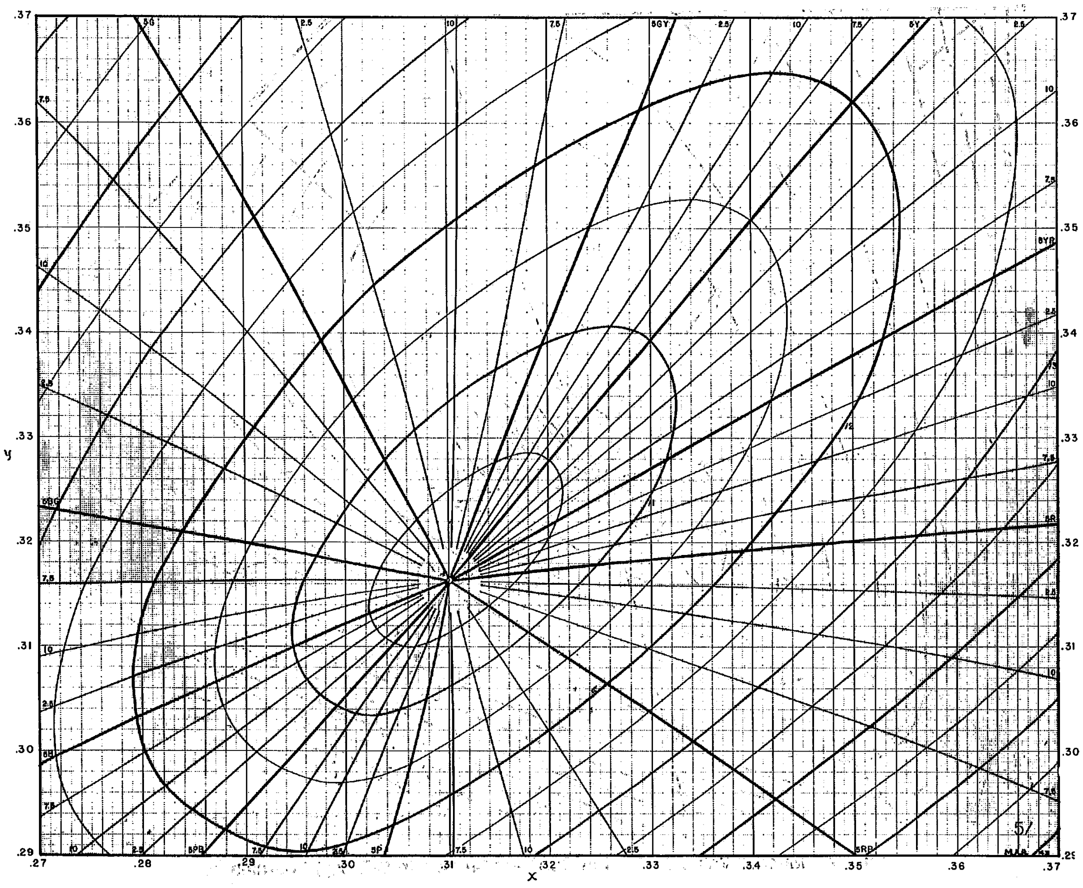


FIG. 8 Munsell Value 5—Loci of Constant Hue and Constant Chroma, Near Gray, in CIE (*x*,*y*) Coordinates

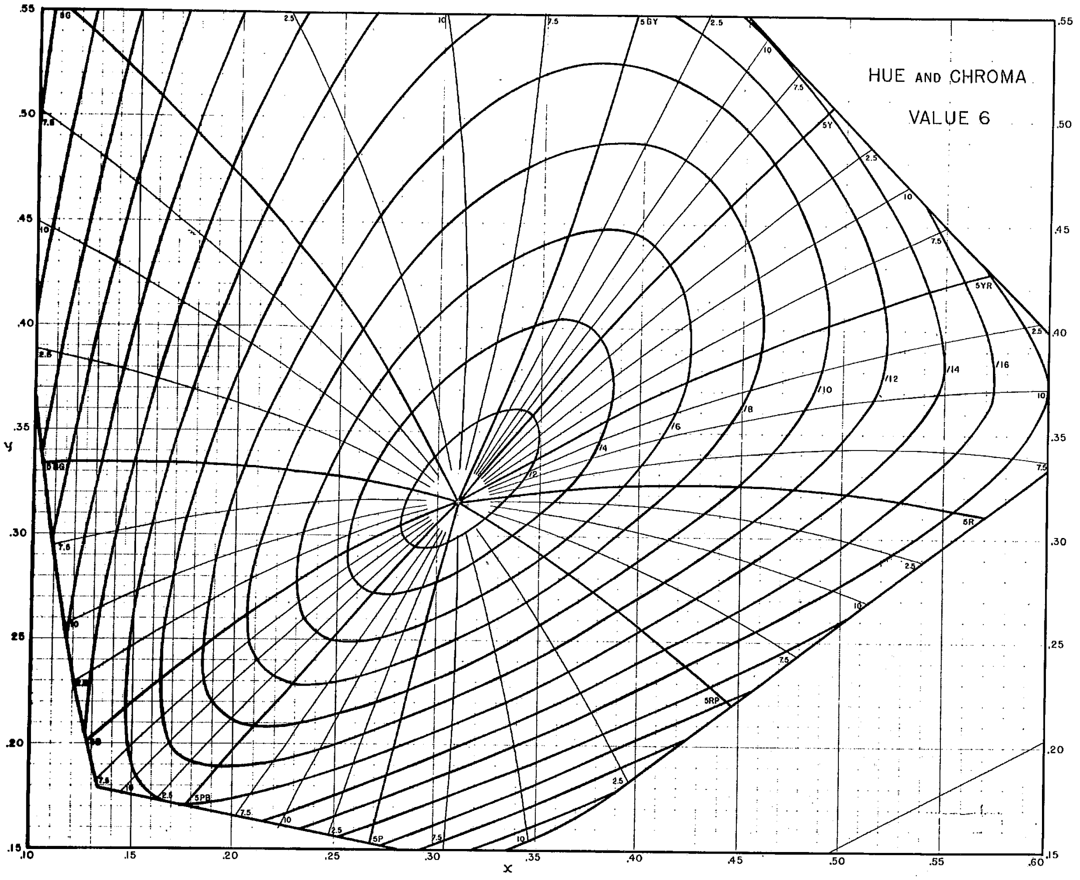


FIG. 9 Munsell Value 6—Loci of Constant Hue and Constant Chroma in CIE (*x*,*y*) Coordinates

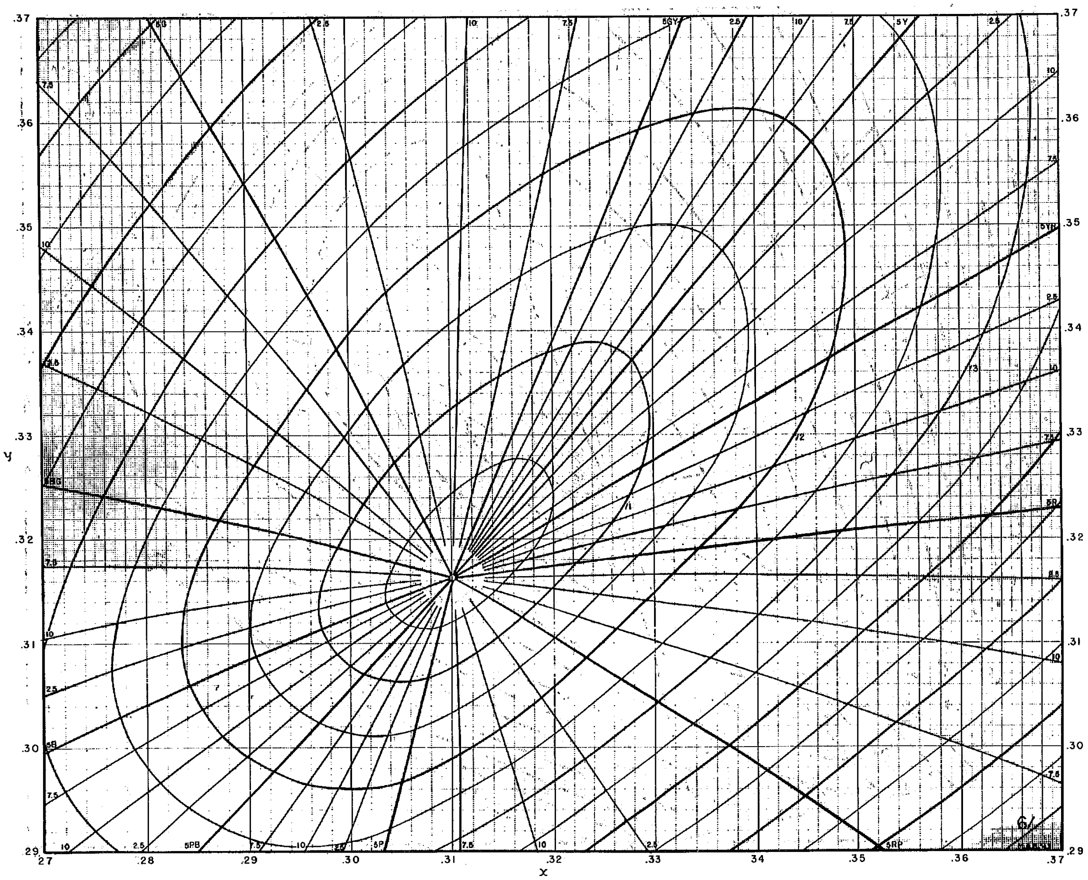


FIG. 10 Munsell Value 6—Loci of Constant Hue and Constant Chroma, Near Gray, in CIE (*x*,*y*) Coordinates

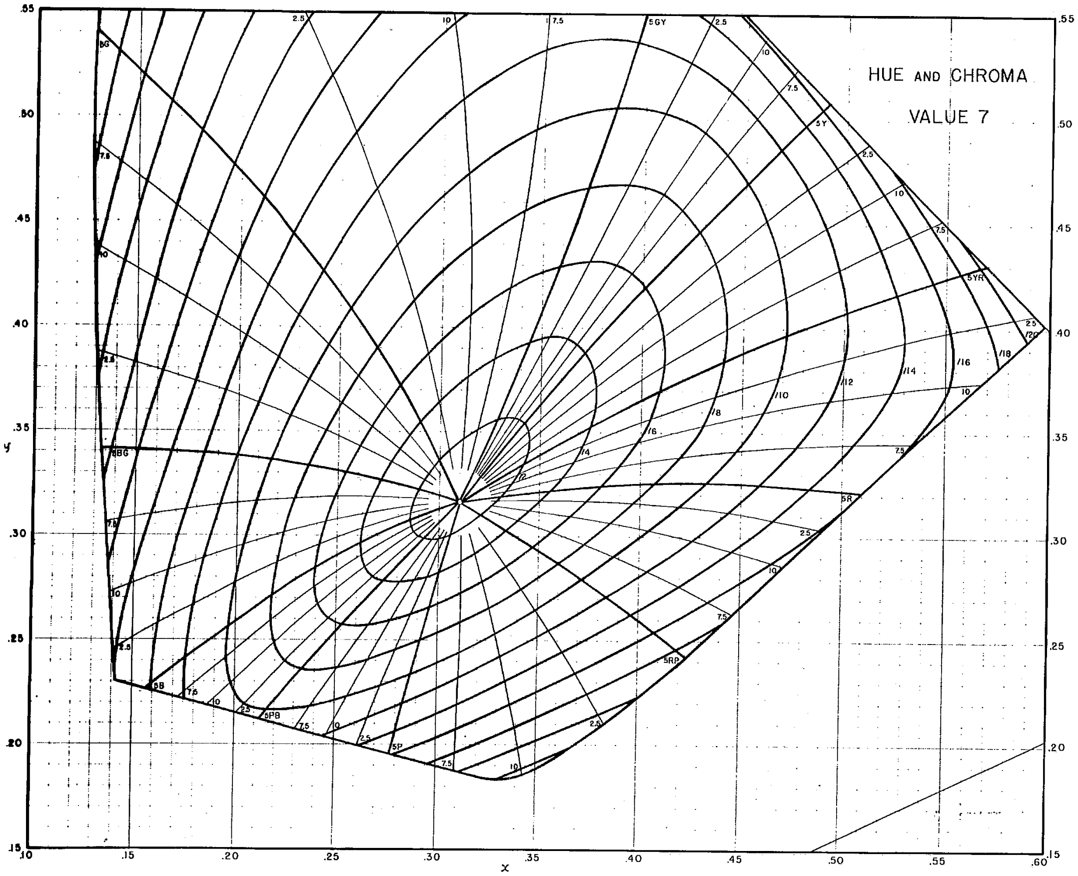


FIG. 11 Munsell Value 7—Loci of Constant Hue and Constant Chroma in CIE (*x*,*y*) Coordinates

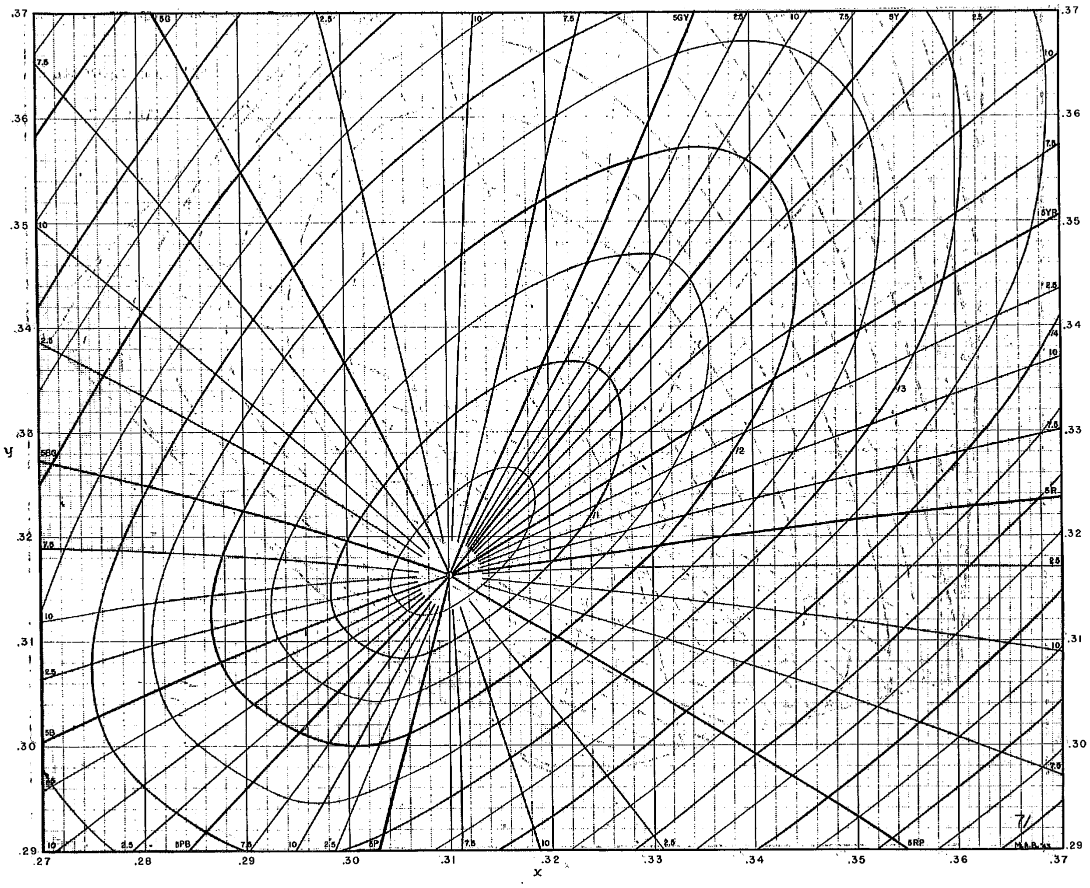


FIG. 12 Munsell Value 7—Loci of Constant Hue and Constant Chroma, Near Gray, in CIE (x,y) Coordinates

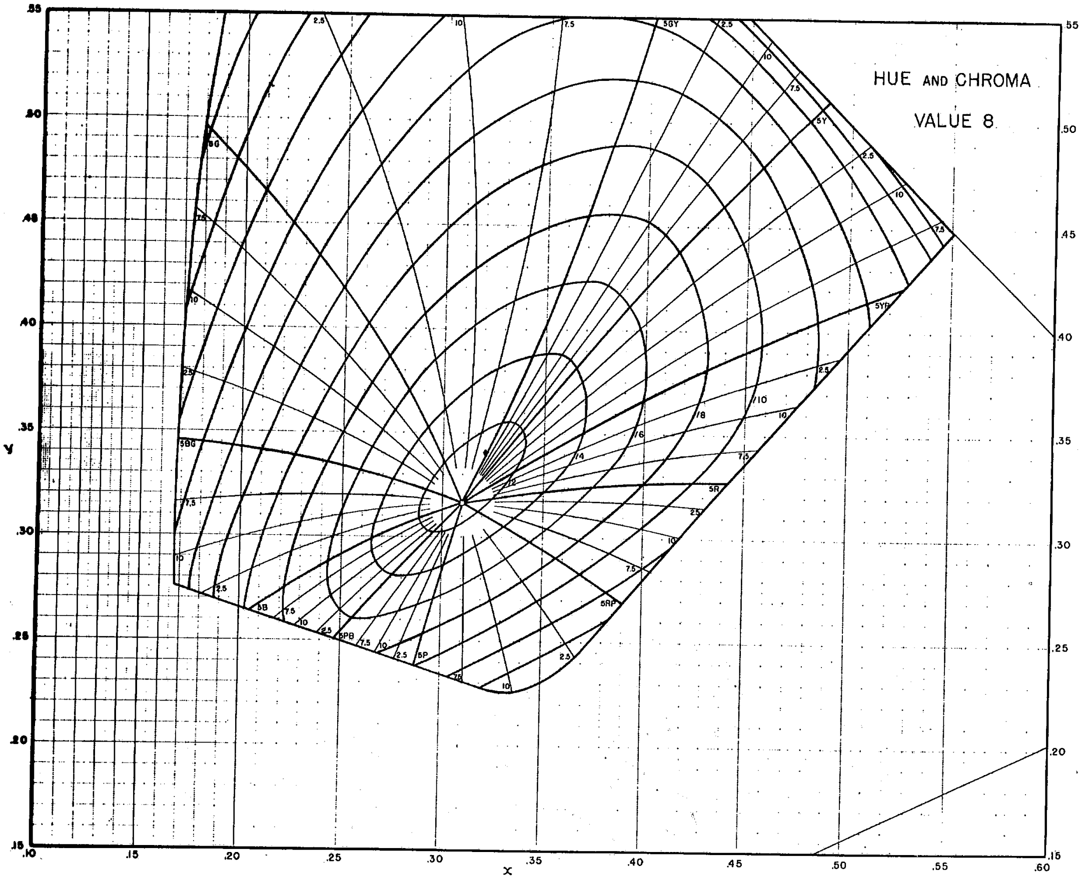


FIG. 13 Munsell Value 8—Loci of Constant Hue and Constant Chroma in CIE (*x*,*y*) Coordinates

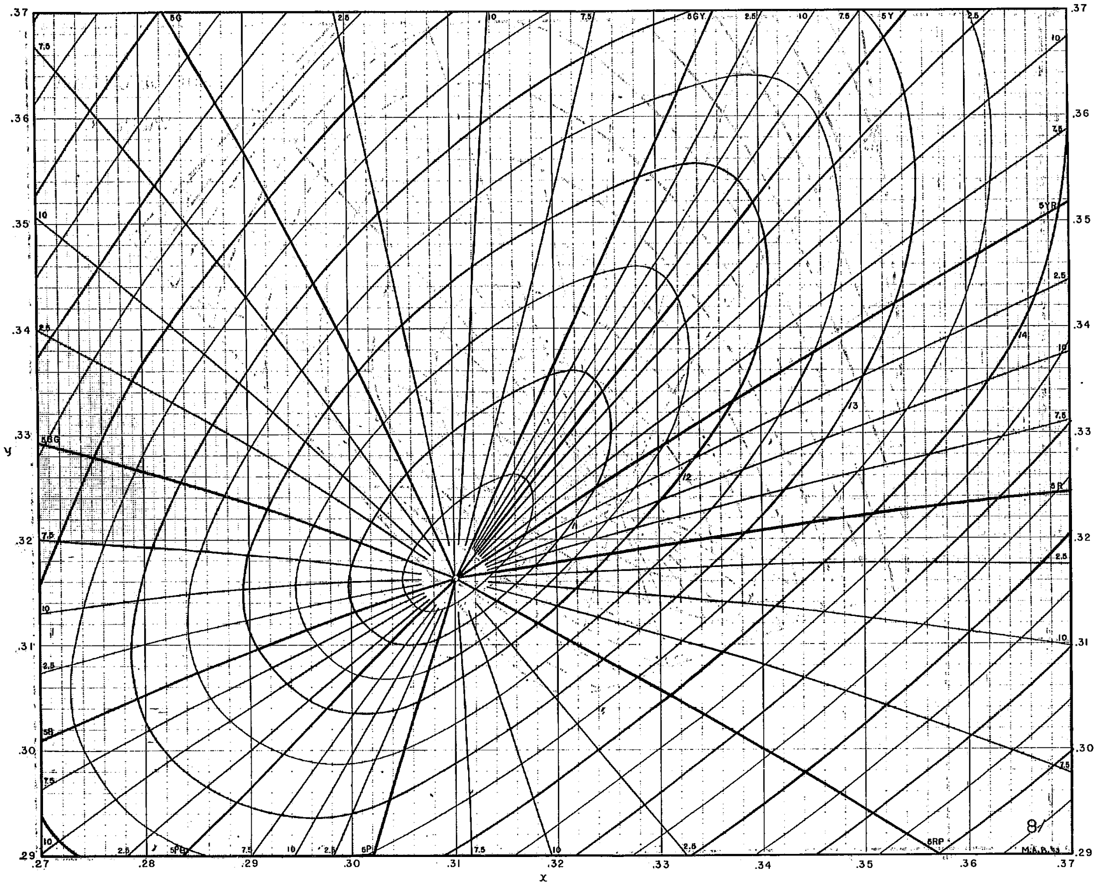


FIG. 14 Munsell Value 8—Loci of Constant Hue and Constant Chroma, Near White, in CIE (x,y) Coordinates

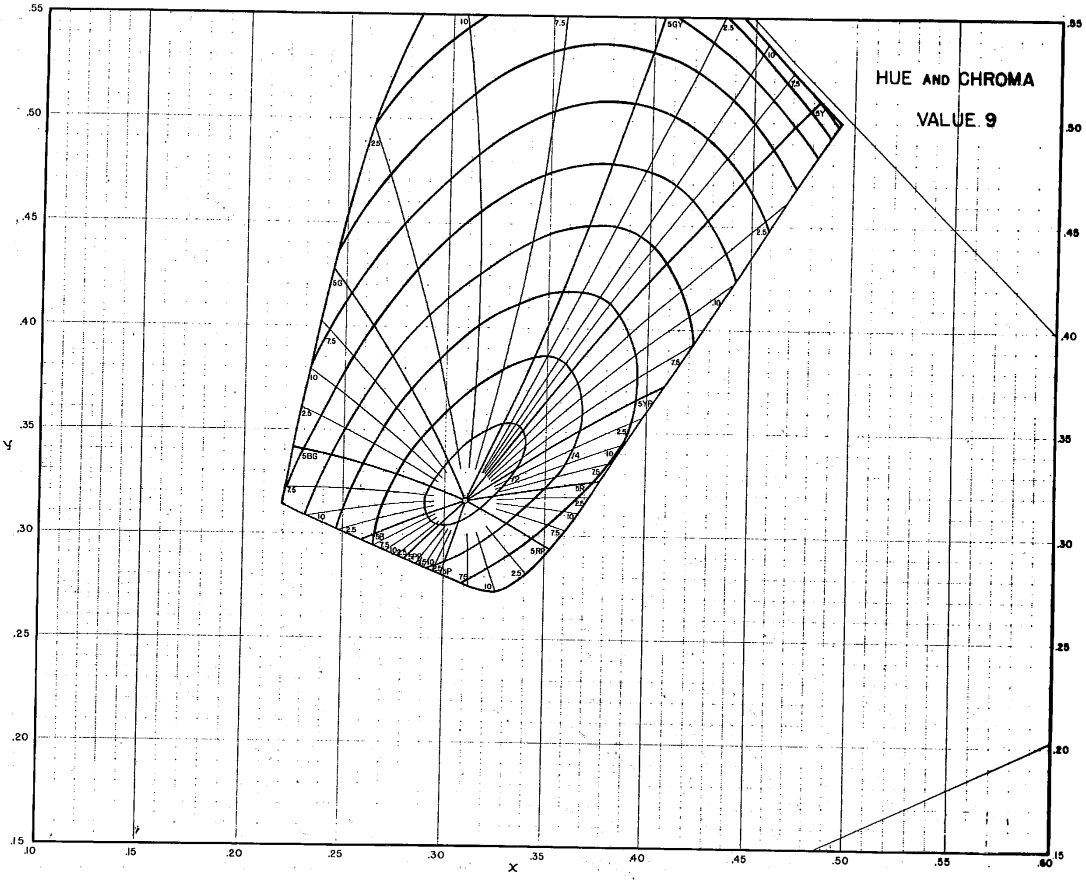


FIG. 15 Munsell Value 9—Loci of Constant Hue and Constant Chroma in CIE (*x*,*y*) Coordinates

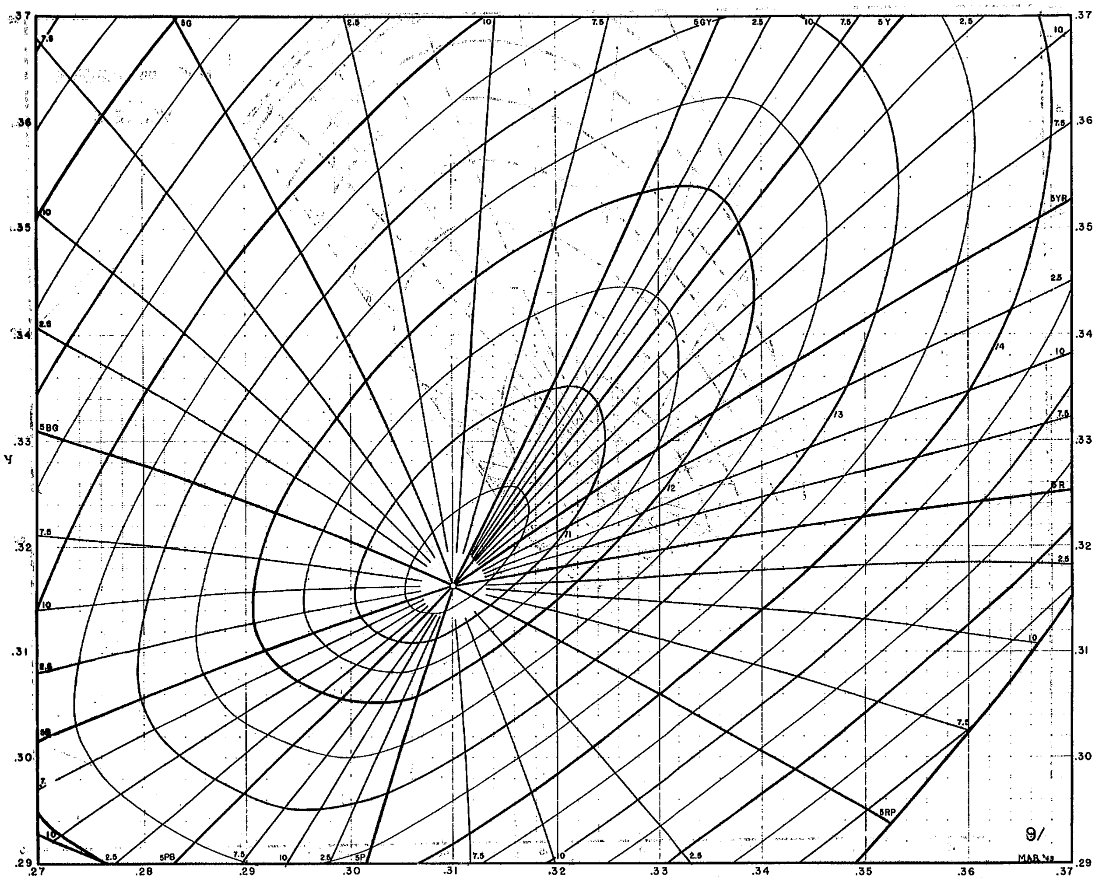


FIG. 16 Munsell Value 9—Loci of Constant Hue and Constant Chroma, Near White, in CIE (x,y) Coordinates

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM D1552: Standard Test Method for Sulfur in Petroleum Products (High-Temperature Method)

CFR Section(s): 40 CFR 60, Appendix A-7

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 1552 – 95

An American National Standard

Standard Test Method for Sulfur in Petroleum Products (High-Temperature Method)[1]

This standard is issued under the fixed designation D 1552; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This test method has been approved for use by agencies of the Department of Defense and for listing in the DOD Index of Specifications and Standards.

1. Scope

1.1 This test method covers three procedures for the determination of total sulfur in petroleum products including lubricating oils containing additives, and in additive concentrates. This test method is applicable to samples boiling above 177°C (350°F) and containing not less than 0.06 mass % sulfur. Two of the three procedures use iodate detection; one employing an induction furnace for pyrolysis, the other a resistance furnace. The third procedure uses IR detection following pyrolysis in a resistance furnace.

1.2 Petroleum coke containing up to 8 mass % sulfur can be analyzed.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1266 Test Method for Sulfur in Petroleum Products (Lamp Method)[3]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[4]

3. Summary of Test Method

3.1 *Iodate Detection System*—The sample is burned in a stream of oxygen at a sufficiently high temperature to convert about 97 % of the sulfur to sulfur dioxide. A standardization factor is employed to obtain accurate results. The combustion products are passed into an absorber containing an acid solution of potassium iodide and starch indicator. A faint blue color is developed in the absorber solution by the addition of standard potassium iodate solution. As combustion proceeds, bleaching the blue color, more iodate is added. The amount of standard iodate consumed during the combustion is a measure of the sulfur content of the sample.

3.2 *IR Detection System*—The sample is weighed into a special ceramic boat which is then placed into a combustion furnace at 1371°C (2500°F) in an oxygen atmosphere. Most sulfur present is combusted to SO_2 which is then measured with an infrared detector after moisture and dust are removed by traps. A microprocessor calculates the mass percent sulfur from the sample weight, the integrated detector signal and a predetermined calibration factor. Both the sample identification number and mass percent sulfur are then printed out. The calibration factor is determined using standards approximating the material to be analyzed.

4. Significance and Use

4.1 This test method provides a means of monitoring the sulfur level of various petroleum products and additives. This knowledge can be used to predict performance, handling, or processing properties. In some cases the presence of sulfur compounds is beneficial to the product and monitoring the depletion of sulfur can provide useful information. In other cases the presence of sulfur compounds is detrimental to the processing or use of the product.

5. Interferences

5.1 For the iodate systems, chlorine in concentrations less than 1 mass % does not interfere. The IR system can tolerate somewhat higher concentrations. Nitrogen when present in excess of 0.1 mass % may interfere with the iodate systems; the extent of such interference may be dependent on the type of nitrogen compound as well as the combustion conditions. Nitrogen does not interfere with the IR system. The alkali and alkaline earth metals, as well as zinc, phosphorus, and lead, do not interfere with either system.

6. Apparatus

6.1 *Combustion and Iodate Detection System*

6.1.1 *Furnaces*—Two major types are available, the primary difference being the manner in which the necessary high temperatures are obtained. These two types are as follows:

6.1.1.1 *Induction Type*, which depends upon the high-frequency electrical induction method of heating. This assembly shall be capable of attaining a temperature of at least 1482°C (2700°F) in the sample combustion zone, under the conditions set forth in Section 10 and shall be equipped with an additional induction coil located above the combustion zone, substantially as shown in Fig. 1.

6.1.1.2 The furnace work coil should have a minimum output of 500 W; the minimum input rating of the furnace must be 1000 W. With the correct amount of iron chips,

---

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

Current edition approved Aug. 15, 1995. Published October 1995. Originally published as D 1552 – 58 T. Last previous edition D 1552 – 90.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 05.02.

weighed to ±0.05 g, the maximum plate current will be between 350 and 450 mA.

NOTE 1: **Warning**—This type of furnace is capable of inflicting high-frequency burns and high-voltage shocks. In addition to other precautions, maintain all guards properly. **Precaution**—Disconnect the furnace from the power line whenever electrical repairs or adjustments are made.

6.1.1.3 *Resistance Type*, capable of maintaining a temperature of at least 1371°C (2500°F).

6.1.2 *Absorber*, as described in Test Method D 1266.

NOTE 2—Also suitable for use with either type of furnace is an automatic titrator, specifically designed for iodometry. This combines the functions of absorption and titration to a predetermined end point.

6.1.3 *Buret*, standard 25-mL or automatic types available from the manufacturers of the specific combustion units, are suitable (Note 2).

6.2 *Combustion and IR Detection System*, comprised of automatic balance, oxygen flow controls, drying tubes, combustion furnace, infrared detector and microprocessor. The furnace shall be capable of maintaining a nominal operating temperature of 1350°C (2460°F).[5]

6.3 *Miscellaneous Apparatus*—Specific combustion assemblies require additional equipment such as crucibles, combustion boats, crucible lids, boat pushers, separator disks, combustion tubes, sample inserters, oxygen flow indicator, and oxygen drying trains. The additional equipment required is dependent on the type of furnace used and is available from the manufacturer of the specific combustion unit. To attain the lower sulfur concentration given in Section 1, the ceramics used with the induction furnace assembly shall be ignited in a muffle furnace at 1371°C (2500°F) for at least 4 h before use.

6.4 *Sieve*, 60-mesh (250-mm).

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II or III of Specification D 1193.

7.3 *Alundum* (Al_2O_3) or *Magnesium Oxide* (Com-Aid).

7.4 *Anhydrone (Magnesium Perchlorate)*.

NOTE 3: **Precaution**—In addition to other precautions, handle magnesium perchlorate with care. Avoid contacting it with acid and organic materials. Reactions with fuel may be violent.

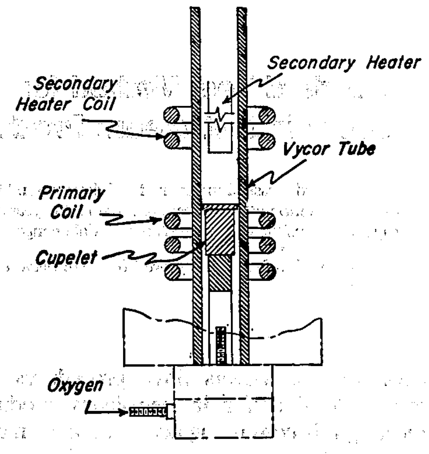


FIG. 1 Combustion Tube

7.5 *Hydrochloric Acid* (3 + 197)—Dilute 30 mL of concentrated hydrochloric acid (HCl, relative density 1.19) to 2 L with water.

NOTE 4: **Warning**—Poison. Corrosive. May be fatal if swallowed. Liquid and vapor cause severe burns.

7.6 *Oxygen (Extra Dry)*—The oxygen shall be at least 99.5 % pure and show no detectable sulfur by blank determination.

NOTE 5: **Warning**—Oxygen vigorously accelerates combustion.

7.7 *Phosphorus Pentoxide* (P_2O_5).

7.8 *Potassium Alum (Aluminum Potassium Sulfate)*.

7.9 *Potassium Iodate, Standard Solution* (0.06238 *M*, 1 mL = 1 mg S)—Dissolve 2.225 g of potassium iodate (KIO_3) that has been dried at about 180°C to constant weight, in water and dilute to 1 L. Thoroughly mix the solution.

7.10 *Potassium Iodate, Standard Solution* (0.006238 *M*, 1 mL = 0.1 mg S)—Measure exactly 100 mL of KIO_3 solution (0.06238 *M*, 1 mL = 1 mg S) into a 1-L volumetric flask, and dilute to volume with water. Thoroughly mix the solution.

7.11 *Potassium Iodate, Standard Solution* (0.01248 *M*, 1 mL = 0.2 mg S)—Measure exactly 200 mL of KIO_3 solution (0.06238 *M*, 1 mL = 1 mg S) into a 1-L volumetric flask and dilute to volume with water. Thoroughly mix the solution.

7.12 *Ascarite*, 8 to 20 mesh.

7.13 *Special Materials for Induction-Type Furnaces:*

7.13.1 *Tin* (20 to 30-mesh).

7.13.2 *Iron-Chip Accelerator* having a sulfur content of not more than 0.005 mass %.

7.14 *Standard Sample*—Potassium alum ($AlK(SO_4)_2 \cdot 12H_2O$).

7.15 *Starch-Iodide Solution*—Make a paste by adding 9 g of soluble starch to 15 mL of water. Add this mixture, with stirring, to 500 mL of boiling water. Cool the mixture, add 15 g of potassium iodide (KI), and dilute to 1 L with water.

7.16 *Sulfuric Acid* (relative density 1.84)—Concentrated sulfuric acid (H_2SO_4).

NOTE 6: **Warning**—Poison. Corrosive. Strong oxidizer.

7.17 *Vanadium Pentoxide*, anhydrous, powdered V_2O_5.

8. Sampling

8.1 Take samples in accordance with the instructions in Practice D 4057.

---

[5] The Models SC32, or SC132, manufactured by LECO Corporation, 3800 Lakeview Avenue, St. Joseph, MI 49085-2396, have been found satisfactory for this purpose.

[6] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

9. Preparation of Apparatus

9.1 *Induction-Type Furnace*—Assemble the apparatus according to the instructions furnished by the manufacturer. Purify the oxygen by passing it through (*1*) H_2SO_4 (relative density 1.84), (*2*) Ascarite, and (*3*) magnesium perchlorate ($Mg(ClO_4)_2$) or phosphorus pentoxide (P_2O_5) (**Precaution**—see Note 3). Connect a rotameter between the purifying train and the furnace. Insert a small glass-wool plug in the upper end of the glass tubing connecting the furnace with the absorber to catch oxides of tin. Connect the exit end of the combustion tube to the absorber with glass tubing, using gum rubber tubing to make connections. Position the absorber so as to make this delivery line as short as possible. Figure 2 illustrates schematically the assembled apparatus. Adjust the oxygen flow to 1 ± 0.05 L/min. Add 65 mL of HCl (3 + 197) and 2 mL of starch-iodide solution to the absorber. Add a sufficient amount of the appropriate standard KIO_3 solution (Table 1) to produce a faint blue color. This color will serve as the end point for the titration. Adjust the buret to zero. Turn on the furnace filament switch and allow at least 1 min warm-up before running samples (**Precaution**—see Note 3).

9.2 *Resistance-Type Furnace*—Assemble the apparatus according to the instructions furnished by the manufacturer. Purify the oxygen by passing it through (*1*) H_2SO_4 (relative density 1.84), (*2*) Ascarite, and (*3*) $Mg(ClO_4)_2$ or P_2O_5 (**Precaution**—see Note 3). Connect a rotameter between the purifying train and the furnace. Figure 3 illustrates schematically the assembled apparatus. Turn on the current and adjust the furnace control to maintain a constant temperature of 1316 ± 14°C (2400 ± 25°F). Adjust the oxygen flow rate to 2 ± 0.1 L/min. Add 65 mL of HCl (3 ± 197) and 2 mL of starch-iodide solution to the absorber. Add a few drops of the appropriate standard KIO_3 solution (Table 2) to produce a faint blue color. Adjust the buret to zero.

9.3 *Resistance-Type Furnace-IR Detection*—Assemble and adjust apparatus according to manufacturer's instructions. Initialize microprocessor, check power supplies, set oxygen pressure and flows and set furnace temperature to 1371°C (2500°F).

9.3.1 Condition a fresh anhydrone scrubber with four coal samples.

9.3.2 Calibrate the automatic balance according to manufacturer's instructions.

10. Standardization

10.1 *For Iodate Methods:*

10.1.1 *Determination of Alum Factor:*

10.1.1.1 Because these rapid combustion methods involve the reversible reaction $2SO_2 + O_2 = 2SO_3$, it is not possible to evolve all the sulfur as SO_2. The equilibrium of the reaction is temperature dependent and, in an oxygen atmosphere above 1316°C, about 97 % of the sulfur is present as SO_2. To assure that the furnace is in proper adjustment and that its operation produces acceptably high temperature, potassium alum is employed for standardizing the apparatus. Depending on the type of combustion equipment used, proceed as described in Sections 10 to 13 to determine the alum factor. Use 15 mg weighed to ±0.1 mg of potassium alum for this determination. Use the same materials in the determination of the alum and standardization factors as for the unknown samples. For example, V_2O_5 has a definite effect and should be included if used for unknowns as recommended in the procedure with the resistance-type furnace (Note 10).

TABLE 1 Sample Weight for Induction Furnace

Sulfur Content, %	Weight of Sample to be Taken, mg	Normality of Standard KIO_3 solution for Titration
0 to 2	90^A	0.006238
2 to 4	50 to 90	0.006238
4 to 10	50 to 90	0.01248
Over 10	12.1.1	(Note 7)

^{A} Approximate.

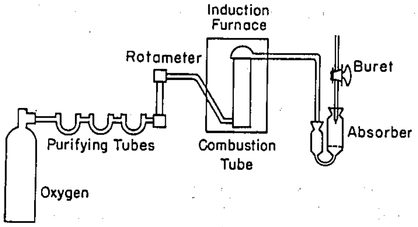


FIG. 2 Schematic Illustration of Induction-Type Furnace

10.1.1.2 Calculate the alum factor as follows:

$$\text{Alum factor } (AF) = (S_A \times W_A)/(100(V_a - V_b) \times C_1) \quad (1)$$

where:

S_A = mass percent sulfur in potassium alum used,

W_A = milligrams of potassium alum used,

V_a = millilitres of standard KIO_3 solution used in determining the alum factor,

V_b = millilitres of standard KIO_3 solution used in the blank determination, and

C_1 = sulfur equivalent of the standard KIO_3 solution used in determining the alum factor, mg/mL.

10.1.1.3 The alum factor should be in the range from 1.02 to 1.08. If values smaller than 1.02 are observed, confirm independently the sulfur content of the alum and the sulfur equivalent of the KIO_3 solution before repeating the alum factor determination. If values larger than 1.08 are observed, make adjustments in the equipment in accordance with the manufacturer's recommendation and repeat the alum factor determination.

10.1.2 *Determination of Standardization Factor:*

10.1.2.1 Because effects such as sample volatility can also affect the relative recovery as SO_2 of the sulfur originally present in the sample, it is necessary to determine a standardization factor. Proceed as described in Sections 10 to 13, using an oil sample of similar type to the unknown sample and of accurately known sulfur content.[7]

10.1.2.2 For IR detection, determine and load the microprocessor with the calibration factor for the particular type of sample to be analyzed (lubricating oil, petroleum coke, residual fuel) as recommended by the manufacturer.

10.1.2.3 Calculate the standardization factor as follows:

$$\text{Standardization factor } (F_s) = (S_s \times W_s)/(100\ (V_s - V_b) \times C) \quad (2)$$

---

[7] Residual fuel oil Standard Reference Materials may be obtained from the National Institute of Standards and Technology or other sources.

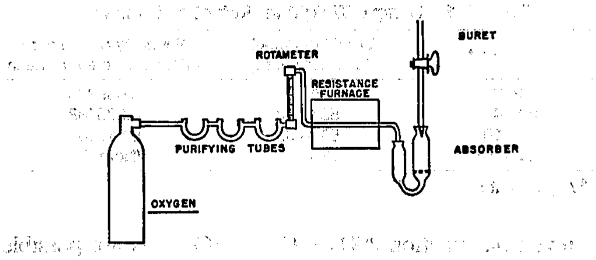


FIG. 3 Schematic Illustration of Resistance-Type Furnace

where:

S_s = mass percent sulfur in standardization sample used,
W_s = milligrams of standardization sample used,
V_b = millilitres of standard KIO_3 solution used in the blank determination,
V_s = millilitres of standard KIO_3 solution used in determining the standardization factor, and
C = sulfur equivalent of the standard KIO_3 solution used in determining the standardization factor, mg/mL.

10.1.3 *Quality Control*—Run a suitable analytical quality control sample several times daily. When the observed value lies between acceptable limits on a quality control chart, proceed with sample determinations.

11. Preparation of Coke

11.1 It is assumed that a representative sample has been received for analysis.

11.2 Grind and sieve the sample received so as to pass a 60-mesh (250-mm) sieve.

11.3 Dry the sieved material to constant weight at 105 to 110°C.

12. Procedure with Induction-Type Furnace

12.1 *Sample Preparation*—Add a 3.2 to 4.8-mm (1/8 to 3/16-in.) layer of alundum or magnesium oxide to a sample crucible. Make a depression in the bed with the end of a stirring rod. Weigh the crucible to 0.1 mg. Weigh into the depression the proper amount of sample according to Table 1 (12.1.1) (Note 7). Cover the sample with a separator disk (Note 8). Place on the separator disk the predetermined amount of iron chips necessary to obtain the required temperature (6.1.1.2). This is usually between 1.2 and 2.0 g, but should be held constant with ±0.05 g. Sprinkle about 0.1 g of tin on the iron. Cover the crucible with a lid and place on the furnace pedestal.

12.1.1 Under no conditions shall an organic sample larger than 100 mg be burned in an induction-type furnace.

NOTE 7—More concentrated KIO_3 solutions, such as the 0.06238 *N* solution, may be found more convenient for samples containing more than 10 % sulfur. The sample size and KIO_3 concentration should be chosen so that not more than 25 mL of titrant are needed.

NOTE 8—The use of the separator disk is optional.

12.2 *Combustion and Titration*—Turn on the plate current switch. After about 1 min for warm-up, raise the pedestal and lock into position. The plate current will fluctuate for a few seconds and should gradually rise to a maximum value. Add the appropriate standard KIO_3 solution (Table 1) to the absorber to maintain the blue color. Should the absorber solution become completely colorless, discard the determination. Make KIO_3 additions as the rate of evolution of SO_2 diminishes such that, when combustion is completed, the intensity of the blue color is the same as the initial intensity. Combustion is complete when this color remains for at least 1 min and the plate current has dropped considerably. Record the volume of KIO_3 solution required to titrate the SO_2 evolved.

12.3 *Blank Determination*—Make a blank determination whenever a new supply of crucibles, materials, or reagents is used. Follow the preceding procedure, but omit the sample.

TABLE 2 Sample Weight for Resistance Furnace

Sulfur Content, %	Weight of Sample to be Taken, mg	Normality of Standard KIO_3 solution for Titration
0 to 2	100 to 200	0.006238
2 to 5	100 to 200	0.01248
5 to 10	100 to 200	0.06238
Over 10	(Note 7)	(Note 7)

13. Procedure with Resistance-Type Furnace

13.1 *Sample Preparation*—Weigh into a combustion boat the proper amount of sample according to Table 2 (Footnote 8). Add 100 ± 5 mg of vanadium pentoxide and completely cover the mixture with Alundum.

13.2 *Combustion and Titration*—Place the boat in the cool portion of the combustion tube, near the entrance. To proceed with the combustion, push the boat containing the sample progressively into the hotter zone of the combustion tube using the equipment provided by the manufacturers. The boat should be advanced as rapidly as possible consistent with the rate of evolution of SO_2. Add the appropriate standard KIO_3 solution (Table 2) to the absorber to maintain the blue color. Should the absorber solution become completely colorless, discard the determination. Make KIO_3 additions as the rate of evolution of SO_2 diminishes such that, when combustion is completed, the intensity of the blue color is the same as the initial intensity. Combustion is complete when this color remains for at least 1 min. Record the volume of KIO_3 solution required to titrate the SO_2 evolved.

13.3 *Blank Determination*—Make a blank determination whenever a new supply of combustion boats, materials, or reagents is used. Follow the above procedure, but omit the sample.

14. Procedure with Resistance Furnace–IR Detection

14.1 Allow the system to warm up and the furnace to reach operating temperature.

14.2 After homogeneity of the sample is assured, select the sample size as follows: for liquid samples, take up to 0.13 g for analysis and for solid samples, take up to 0.4 g for analysis. In each case mass percent sulfur times weight of sample must be less than or equal to four in the case of the SC32 instrument, and two in the case of the SC132 instrument. For other instruments, consult the manufacturer's instructions.

14.3 Determine and store the system blank value.

---

[8] Precision for the IR detection method was determined in a 1985 cooperative study (RR: D02-1231) which involved fourteen laboratories and ten samples. No statistically significant bias between the iodate and IR detector procedures was found.

14.4 Weigh the samples into combustion boats and record the net weights. It is possible to weigh and store several weights in the microprocessor before beginning a series of burns.

14.4.1 Fill the combustion boat to one-third capacity with evenly spread MgO powder.

14.4.2 Form a slight trench in the MgO powder with a scoop.

14.4.3 Place the combustion boat on the balance and weigh an appropriate amount of the sample into the trench in the MgO powder. Enter the weight.

14.4.4 Remove the combustion boat from the balance and add MgO powder until the combustion boat is filled to two-thirds capacity.

NOTE 9—If unacceptable repeatability is encountered for particular oil samples, combustion promoter such as V_2O_5 or the LECO product *Com-Aid* can be substituted for the MgO.

NOTE 10—**Caution**—V_2O_5 can cause deterioration of the furnace ceramics so use it with care.

14.5 Initiate oxygen flow and load boat into furnace.

14.6 When the analysis is complete, read the result from the microprocessor.

14.7 Remove the expended combustion boat from the furnace.

14.8 Make repeated runs until two results differ by less than the repeatability of the method.

15. Calculation

15.1 *Calculation for Iodate Detection*—Calculate the sulfur content of the sample as follows:

$$\text{Sulfur, mass \%} = (100\,(V - V_b) \times F_s \times C)/W \quad (3)$$

where:

V = standard KIO_3 solution, mL, used in the analysis,
V_b = standard KIO_3 solution, mL, used in the blank determination,
F_s = standardization factor (see 10.1.2),
C = sulfur equivalent of the standard KIO_3 solution used in the analysis, mg/mL, and
W = milligrams of sample used in the analysis.

15.2 *Calculation for IR Detection:*

15.2.1 Report all results using the microprocessor.

15.2.2 Report the average of two results.

16. Report

16.1 In the range from 0.05 to 5.00 mass % sulfur, report to the nearest 0.01 mass %. In the range from 5 to 30 mass % sulfur, report to the nearest 0.1 mass %.

17. Precision and Bias

17.1 *For Petroleum Products by Iodate and IR Methods*—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

17.1.1 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

	Repeatability	
Sulfur, mass-% Range	Iodate	IR[8]
0.0 to 0.5	0.05	0.04
0.5 to 1.0	0.07	0.07
1.0 to 2.0	0.10	0.09
2.0 to 3.0	0.16	0.12
3.0 to 4.0	0.22	0.13
4.0 to 5.0	0.24	0.16

17.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

	Reproducibility	
Sulfur, mass-% Range	Iodate	IR[8]
0.0 to 0.5	0.08	0.13
0.5 to 1.0	0.11	0.21
1.0 to 2.0	0.17	0.27
2.0 to 3.0	0.26	0.38
3.0 to 4.0	0.40	0.44
4.0 to 5.0	0.54	0.49

17.2 *For Petroleum Cokes by Iodate and IR Methods*—The precision of the test method as determined by statistical examination of interlaboratory results is as follows:

17.2.1 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

$$r = 0.05X$$

where X is the average of the two test results.

17.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material could, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

$$R = 0.22X$$

where X is the average of the two test results.

17.3 *Bias*—The bias of the procedure in this test method is being determined.

18. Keywords

18.1 furnace; high temperature; induction furnace; iodate titration; IR detection; petroleum; resistance; sulfur; titration

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM D1688: Standard Test Method for Copper in Water

CFR Section(s): 40 CFR 141.23(k)(1)

Standards Body: American Society for Testing and Materials

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1688 – 95

Standard Test Methods for Copper in Water[1]

This standard is issued under the fixed designation D 1688; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue that has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover the determination of copper in water by atomic absorption spectrophotometry. Three test methods are included as follows:

	Concentration Range	Sections
Test Method A—Atomic Absorption, Direct	0.05 to 5 mg/L	7 to 15
Test Method B—Atomic Absorption, Chelation-Extraction	50 to 500 µg/L	16 to 24
Test Method C—Atomic Absorption, Graphite Furnace	5 to 100 µg/L	25 to 33

1.2 Either dissolved or total recoverable copper may be determined. Determination of dissolved copper requires filtration through a 0.45-µm (No. 325) membrane filter at the time of collection. In-line membrane filtration is preferable.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Notes 3, 5, 8, and 13.

1.5 Three former photometric test methods were discontinued. Refer to Appendix X1 for historical information.

2. Referenced Documents

2.1 *ASTM Standards:*

D 858 Test Methods for Manganese in Water[2]
D 1066 Practice for Sampling Steam[2]
D 1068 Test Methods for Iron in Water[2]
D 1129 Terminology Relating to Water[2]
D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits[2]
D 1193 Specification for Reagent Water[2]
D 1687 Test Methods for Chromium in Water[2]
D 1691 Test Methods for Zinc in Water[2]
D 1886 Test Methods for Nickel in Water[2]
D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[2]
D 3370 Practices for Sampling Water from Closed Conduits[2]
D 3557 Test Methods for Cadmium in Water[2]
D 3558 Test Methods for Cobalt in Water[2]
D 3559 Test Methods for Lead in Water[2]
D 3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry[2]
D 4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1129.

4. Significance and Use

4.1 Copper is found in naturally occurring minerals principally as a sulfide, oxide, or carbonate. It makes up approximately 0.01 % of the earth's crust and is obtained commercially from such ores as chalcopyrite ($CuFeS_2$). Copper is also found in biological complexes such as hemocyanin.

4.2 Copper enters water supplies through the natural process of dissolution of minerals, through industrial effluents, through its use, as copper sulfate, to control biological growth in some reservoirs and distribution systems, and through corrosion of copper alloy water pipes. Industries whose wastewaters may contain significant concentrations of copper include mining, ammunition production, and most metal plating and finishing operations. It may occur in simple ionic form or in one of many complexes with such groups as cyanide, chloride, ammonia, or organic ligands.

4.3 Although its salts, particularly copper sulfate, inhibit biological growth such as some algae and bacteria, copper is considered essential to human nutrition and is not considered a toxic chemical at concentrations normally found in water supplies.

5. Purity of Reagents

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be

---

[1] These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved Feb. 15, 1995. Published April 1995. Originally published as D 1688 – 59 T. Last previous edition D 1688 – 90.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type I. Other reagent water types may be used, provided it is first ascertained that the water is of sufficiently high purity to permit its use without lessening the bias and precision of the determination. Type II water was specified at the time of round-robin testing of this test method.

6. Sampling

6.1 Collect the sample in accordance with Practices D 1066, Specification D 1192, and Practices D 3370, as applicable.

6.2 Samples shall be preserved with nitric acid (HNO_3, sp gr 1.42) to a pH of 2 or less immediately at the time of collection, normally about 2 mL/L. If only dissolved copper is to be determined, the sample shall be filtered through a 0.45-µm (No. 325) membrane filter before acidification. The holding time for samples may be calculated in accordance with Practice D 4841.

TEST METHOD A—ATOMIC ABSORPTION, DIRECT

7. Scope

7.1 This test method covers the determination of dissolved and total recoverable copper in most waters and waste waters.

7.2 This test method is applicable in the range from 0.05 to 5 mg/L of copper. The range may be extended to concentrations greater than 5 mg/L by dilution of the sample.

7.3 Collaborative test data were obtained on reagent water, river water, tap water, ground water, lake water, refinery primary treated effluent, and two untreated waste waters. The information on precision and bias may not apply to other waters.

8. Summary of Test Method

8.1 Copper is determined by atomic absorption spectrophotometry. Dissolved copper in the filtered sample is aspirated directly with no pretreatment. Total recoverable copper in the sample is aspirated following hydrochloric-nitric acid digestion and filtration. The same digestion procedure may be used to determine total recoverable cadmium (Test Methods D 3557), chromium (Test Methods D 1687), cobalt (Test Methods D 3558), iron (Test Methods D 1068), lead (Test Methods D 3559), manganese (Test Methods D 858), nickel (Test Methods D 1886), and zinc (Test Methods D 1691).

9. Interferences

9.1 Sodium, potassium, sulfate, and chloride (8000 mg/L each), calcium and magnesium (5000 mg/L each), nitrate (2000 mg/L), iron (1000 mg/L), and cadmium, lead, nickel, zinc, cobalt, manganese, and chromium (10 mg/L each) do not interfere.

9.2 Background correction or a chelation-extraction procedure (see Test Method B) may be necessary to determine low levels of copper in some waters.

NOTE 1—Instrument manufacturers' instructions for use of the specific correction technique should be followed.

10. Apparatus

10.1 *Atomic Absorption Spectrophotometer*, for use at 324.7 nm.

NOTE 2—The manufacturer's instructions should be followed for all instrumental parameters. A wavelength other than 324.7 nm may be used if it has been determined to be equally suitable.

10.1.1 *Copper Hollow-Cathode Lamp*—Multielement hollow-cathode lamps are available and have been found satisfactory.

10.2 *Oxidant*—See 11.6.

10.3 *Fuel*—See 11.7.

10.4 *Pressure-Reducing Valves*—The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

11. Reagents and Materials

11.1 *Copper Solution, Stock* (1.0 mL = 1.0 mg Cu)—Dissolve 1.000 g of electrolytic copper contained in a 250-mL beaker in a mixture of 15 mL of HNO_3 (sp gr 1.42) and 15 mL of water. Slowly add 4 mL of H_2SO_4 (1+1) and heat until SO_3 fumes evolve. Cool, wash down the beaker with water, and dilute to 1 L with water.

11.2 *Copper Solution, Standard* (1.0 mL = 0.1 mg Cu)—Dilute 100.0 mL of copper stock solution to 1 L with water.

11.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

NOTE 3—If a high reagent blank is obtained, distill the HCl or use a spectrograde acid. **Caution**—When HCl is distilled an azeotropic mixture is obtained (approximately 6 *N* HCl). Therefore, whenever concentrated HCl is specified for the preparation of a reagent or in the procedure, use double the volume specified if distilled HCl is used.

11.4 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

NOTE 4—If a high reagent blank is obtained, distill the HNO_3 or use a spectrograde acid.

11.5 *Nitric Acid* (1+499)—Add 1 volume of HNO_3 (sp gr 1.42) to 499 volumes of water.

11.6 *Oxidant:*

11.6.1 *Air*, which has been passed through a suitable filter to remove oil, water, and other foreign substances, is the usual oxidant.

11.7 *Fuel:*

11.7.1 *Acetylene*—Standard, commercially available acetylene is the usual fuel. Acetone, always present in acetylene cylinders, can affect analytical results. The cylinder should be replaced at 50 psig (345 kPa).

NOTE 5: **Precaution**—"Purified" grade acetylene containing a special proprietary solvent rather than acetone should not be used with poly(vinyl chloride) tubing as weakening of the tubing walls can cause a potentially hazardous situation.

12. Standardization

12.1 Prepare 100 mL each of a blank and at least four standard solutions to bracket the expected copper concentration range of the samples to be analyzed by diluting the standard copper solution (11.2) with HNO_3 (1+499). Prepare the standards each time the test is to be performed.

12.2 When determining total recoverable copper add 0.5 mL of HNO_3 (sp gr 1.42) and proceed as directed in 13.2 through 13.4. When determining dissolved copper proceed with 13.5.

12.3 Aspirate the blank and standards and record the instrument readings. Aspirate HNO_3 (1+499) between each standard.

12.4 Prepare an analytical curve by plotting on linear graph paper the absorbance versus standard concentration for each standard. Alternatively, read directly in concentration if this capability is provided with the instrument.

13. Procedure

13.1 Measure 100.0 mL of a well-mixed acidified sample into a 125-mL beaker or flask.

NOTE 6—If only dissolved copper is to be determined, start with 13.5.

13.2 Add 5 mL of HCl (sp gr 1.19) to each sample.

13.3 Heat the samples on a steam bath or hotplate in a well-ventilated hood until the volume has been reduced to 15 to 20 mL, making certain that the samples do not boil.

NOTE 7—When analyzing samples containing appreciable amounts of suspended matter, the amount of reduction in volume is left to the discretion of the analyst.

13.4 Cool and filter the samples through a suitable filter, such as fine-textured, acid washed, ashless paper, into 100-mL volumetric flasks. Wash the filter paper two or three times with water and adjust to volume.

13.5 Aspirate each filtered and acidified sample and determine its absorbance or concentration at 324.7 nm. Aspirate HNO_3 (1+499) between each sample.

14. Calculation

14.1 Calculate the concentration of copper in each sample, in milligrams per litre, using an analytical curve or alternatively, read directly in concentration (see 12.4).

15. Precision and Bias[4]

15.1 The collaborative test of this test method was performed by ten laboratories, five of which supplied two operators each. Each of the 15 operators made determinations at three levels on three different days in samples of reagent water and water of choice for a total of 270 determinations.

15.2 These collaborative test data were obtained on reagent grade water, river water, tap water, ground water, lake water, refinery primary treated effluent, and two untreated waste waters. For other matrices, these data may not apply.

15.3 *Precision*—The single-operator and overall precision of this test method within its designated range may be expressed as follows:

In reagent water, Type II:

$$S_O = 0.020X + 0.035$$
$$S_T = 0.052X + 0.123$$

In water or waste water:

$$S_O = 0.016X + 0.033$$
$$S_T = 0.060X + 0.039$$

where:

S_O = single-operator precision,
S_T = overall precision, and
X = determined concentration of copper, mg/L.

TABLE 1 Determination of Bias for Test Method A

Amount Added, mg Cu/L	Amount Found, mg Cu/L	Bias, %	Statistically Significant, 95 % Level
	Reagent Water		
4.0	4.11	+2.75	no
2.0	2.06	+3.0	no
0.4	0.46	+15.0	yes
	Water or Waste Water		
4.0	4.03	+0.75	no
2.0	2.02	+1.0	no
0.4	0.41	+2.5	no

15.4 *Bias*—Recoveries of known amounts of copper were as shown in Table 1.

TEST METHOD B—ATOMIC ABSORPTION, CHELATION-EXTRACTION

16. Scope

16.1 This test method covers the determination of dissolved and total recoverable copper in most waters and brines.

16.2 This test method is applicable in the range from 50 to 500 µg/L of copper. The range may be extended to concentrations greater than 500 µg/L by dilution of the sample.

16.3 Collaborative test data were obtained on reagent water, river water, tap water, 50 % artificial sea water, and synthetic NaCl brine (50 000 mg/L). The information on precision and bias may not apply to other waters.

17. Summary of Test Method

17.1 Copper is determined by atomic absorption spectrophotometry. The element, either dissolved or total recoverable, is chelated with pyrrolidine dithiocarbamic acid and extracted with chloroform. The extract is evaporated to dryness, treated with hot nitric acid to destroy organic matter, dissolved in hydrochloric acid, and diluted to a specified volume with water. A portion of the resulting solution is then aspirated into the air-acetylene flame of the spectrophotometer. The digestion procedure summarized in 8.1 is used for total recoverable copper. The same chelation-extraction procedure is used to determine cadmium (Test Methods D 3557), cobalt (Test Methods D 3558), iron (Test Methods D 1068), lead (Test Methods D 3559), nickel (Test Methods D 1886), and zinc (Test Methods D 1691).

18. Interferences

18.1 See Section 9.

19. Apparatus

19.1 All apparatus described in Section 10 are required.

20. Reagents and Materials

20.1 *Bromphenol Blue Indicator Solution* (1 g/L)—

---

[4] Supporting data are available from ASTM Headquarters. Request RR: D19-1037.

Dissolve 0.1 g of bromphenol blue in 100 mL of 50 % ethanol or isopropanol.

20.2 *Chloroform* ($CHCl_3$).

20.3 *Copper Solution, Stock* (1.0 mL = 1.0 mg Cu)—Dissolve 1.000 g of electrolytic copper contained in a 250-mL beaker in a mixture of 15 mL of HNO_3 (sp gr 1.42) and 15 mL of water. Slowly add 4 mL of H_2SO_4 (1+1) and heat until SO_3 fumes evolve. Cool, wash down the beaker with water, and dilute to 1 L with water.

20.4 *Copper Solution, Intermediate* (1.0 mL = 10 µg Cu)—Dilute 10.0 mL of copper stock solution and 1 mL of HNO_3 (sp gr 1.42) to 1 L with water.

20.5 *Copper Solution, Standard* (1.0 mL = 1.0 µg Cu)—Immediately before use, dilute 10.0 mL of copper intermediate solution to 100 mL with water. This standard is used to prepare working standards at the time of analysis.

20.6 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl) (see Note 4).

20.7 *Hydrochloric Acid* (1+2)—Add 1 volume of HCl (sp gr 1.19) to 2 volumes of water.

20.8 *Hydrochloric Acid* (1+49)—Add 1 volume of HCl (sp gr 1.19) to 49 volumes of water.

20.9 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3) (see Note 4).

20.10 *Pyrrolidine Dithiocarbamic Acid-Chloroform Reagent*—Add 36 mL of pyrrolidine to 1 L of $CHCl_3$. Cool the solution and add 30 mL of CS_2 in small portions, swirling between additions. Dilute to 2 L with $CHCl_3$. The reagent can be used for several months if stored in a cool, dark place.

NOTE 8: **Warning**—All components of this reagent are highly toxic. Carbon disulfide is also highly flammable. **Precaution**—Prepare and use in a well-ventilated hood.

20.11 *Sodium Hydroxide Solution* (100 g/L)—Dissolve 100 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

20.12 *Oxidant*—See 11.6.

20.13 *Fuel*—See 11.7.

21. Standardization

21.1 Prepare a blank and sufficient standards containing from 0.0 to 50.0 µg of copper by diluting 0.0 to 50.0-mL portions of standard copper solution (20.5) to 100 mL with water.

21.2 When determining total recoverable copper, use 125-mL beakers or flasks, add 0.5 mL of HNO_3 (sp gr 1.42) and proceed as directed in 22.2 through 22.15. When determining dissolved copper, use 250-mL separatory funnels and proceed as directed in 22.5 through 22.15.

21.3 Construct an analytical curve by plotting the absorbances of standards versus micrograms of copper. Alternatively, read directly in concentration if this capability is provided with the instrument.

22. Procedure

22.1 Measure a volume of a well-mixed acidified sample containing less than 50.0 µg of copper (100 mL maximum) into a 125-mL beaker or flask and adjust the volume to 100 mL with water.

NOTE 9—If only dissolved copper is to be determined measure a volume of filtered and acidified sample containing less than 50.0 µg of copper (100-mL maximum) into a 250-mL separatory funnel, and begin with 22.5.

22.2 Add 5 mL of HCl (sp gr 1.19) to each sample.

22.3 Heat the samples on a steam bath or hotplate in a well-ventilated hood until the volume has been reduced to 15 to 20 mL, making certain that the samples do not boil.

NOTE 10—When analyzing brine samples and samples containing appreciable amounts of suspended matter, the amount of reduction in volume is left to the discretion of the analyst.

22.4 Cool and filter the samples through a suitable filter, such as fine-textured, acid-washed, ashless paper, into 250-mL separatory funnels. Wash the filter paper two or three times with water and adjust the volume to approximately 100 mL.

22.5 Add 2 drops of bromphenol blue indicator solution and mix.

22.6 Adjust the pH by addition of NaOH (100 g/L) solution until a blue color persists. Add HCl (1+49) by drops until the blue color just disappears; then add 2.5 mL of HCl (1+49) in excess. The pH at this point should be 2.3.

NOTE 11—The pH adjustment in 22.6 may be made with a pH meter instead of using an indicator.

22.7 Add 10 mL of pyrrolidine dithiocarbamic acid-chloroform reagent and shake vigorously for 2 min. **Warning**—See Note 8.

22.8 Plug the tip of the separatory funnel with cotton, allow the phases to separate, and drain the $CHCl_3$ phase into a 100-mL beaker.

22.9 Repeat the extraction with 10 mL of $CHCl_3$ and drain the $CHCl_3$ layer into the same beaker.

NOTE 12—If color still remains in the $CHCl_3$ extract, reextract the aqueous phase until the $CHCl_3$ layer is colorless.

22.10 Place the beaker on a hot plate set at low heat or on a steam bath, and evaporate to near dryness. Remove beaker from heat and allow residual solvent to evaporate without further heating.

NOTE 13: **Precaution**—Perform in a well-ventilated hood.

22.11 Hold the beaker at a 45° angle, and slowly add dropwise 2 mL of HNO_3 (sp gr 1.42), rotating the beaker to effect thorough contact of the acid with the residue.

22.11.1 If acid is added to the beaker in a vertical position, a violent reaction will occur accompanied by high heat and spattering.

22.12 Place the beaker on a hotplate set at low heat or on a steam bath and evaporate to near dryness. Remove beaker from heat and allow residual solvent to evaporate without further heating.

22.13 Add 2 mL of HCl (1+2) to the beaker, and heat, while swirling, for 1 min.

22.14 Cool and quantitatively transfer the solution to a 10-mL volumetric flask and adjust to volume with water.

22.15 Aspirate each sample and record the scale reading or concentration at 324.7 nm.

23. Calculation

23.1 Determine the weight of copper in micrograms in each sample by referring to the analytical curve or, alternatively, by multiplying the direct read-out concentration of copper by 10 mL. (See 21.3.) Calculate the concentration of

TABLE 2 Determination of Bias for Test Method B

Amount Added, µg Cu/L	Amount Found, µg Cu/L	Bias, %	Statistically Significant, 95 % Level
	Reagent Water		
300	290	−3.3	no
100	112	+12.0	no
20	65	+225	yes
	Water or Brine		
300	234	−22.0	no
100	93	−7.0	no
20	49	+145	no

copper in the original sample in micrograms per litre using Eq 1:

$$\text{Copper, µg/L} = \frac{1000 \times B}{A} \qquad (1)$$

where:
A = volume of original sample, mL, and
B = weight of copper in sample, µg.

24. Precision and Bias[4]

24.1 The collaborative test of this test method was performed by six laboratories, two of which supplied two operators each. Each operator performed the test at three levels. A total of 120 determinations were made.

24.2 These collaborative test data were obtained on reagent grade water, river water, tap water, 50 % artificial seawater, and synthetic NaCl brine (50 000 mg/L). For other matrices, these data may not apply.

24.3 *Precision*—The single-operator and overall precision of this test method within its designated range may be expressed as follows:

In reagent water, Type II:

$$S_O = 0.119X + 9$$
$$S_T = 0.247X + 47$$

In water or brine:

$$S_O = 27$$
$$S_T = 0.270X + 42$$

where:
S_O = single-operator precision, µg/L,
S_T = overall precision, µg/L, and
X = concentration of copper, µg/L.

24.4 *Bias*—Recoveries of known amounts of copper were as shown in Table 2.

TEST METHOD C—ATOMIC ABSORPTION, GRAPHITE FURNACE

25. Scope

25.1 This test method covers the determination of dissolved and total recoverable copper in most waters and wastewaters.

25.2 This test method is applicable in the range from 5 to 100 µg/L of copper. The range can be increased or decreased by varying the volume of sample injected or the instrumental settings. High concentrations may be diluted but preferably should be analyzed by direct aspiration atomic absorption spectrophotometry (see Test Method A).

25.3 This test method has been used successfully with reagent grade water, filtered tap water, condensate from a medium Btu coal gasification process, river water, lake water, well water, and production plant process waters. It is the user's responsibility to assure the validity of this test method in other matrices.

26. Summary of Test Method

26.1 Copper is determined by an atomic absorption spectrophotometer used in conjunction with a graphite furnace. A sample is placed in a graphite tube, evaporated to dryness, charred (pyrolyzed or ashed) and atomized. Since the graphite furnace uses the sample much more efficiently than flame atomization, the detection of low concentrations of elements in small sample volumes is possible. The absorption signal generated during atomization is recorded and compared to standards. A general guide for the application of the graphite furnace is given in Practice D 3919.

26.2 Dissolved copper is determined on a filtered sample with no pretreatment.

26.3 Total recoverable copper is determined following acid digestion and filtration. Because chlorides interfere with furnace procedures for some metals, the use of hydrochloric acid in any digestion or solubilization step is to be avoided. If suspended material is not present, this digestion and filtration may be omitted.

27. Interferences

27.1 For a complete discussion on general interferences with furnace procedures, the analyst is referred to Practice D 3919.

28. Apparatus

28.1 *Atomic Absorption Spectrophotometer*, for use at 324.7 nm with background correction.

NOTE 14—A wavelength other than 324.7 nm may be used if it has been determined to be suitable. Greater linearity may be obtained at high concentrations by using a less sensitive wavelength.

NOTE 15—The manufacturer's instructions should be followed for all instrumental parameters.

28.2 *Copper Hollow Cathode Lamp*, a single element lamp is preferred, but multielement lamps may be used.

28.3 *Graphite Furnace*, capable of reaching temperatures sufficient to atomize the element of interest.

28.4 *Graphite Tubes*, compatible with furnace device. Standard graphite tubes are preferred unless extreme sensitivity is required. In this instance and to eliminate the possible formation of carbides, pyrolytically coated graphite tubes are recommended.

28.5 *Pipets*, microlitre with disposable tips. Sizes may range from 1 µL to 100 µL, as required.

28.6 *Data Storage and Reduction Devices, Computer- and Microprocessor-Controlled Devices, or Strip Chart Recorders* shall be utilized for collection, storage, reduction, and problem recognition (such as drift, incomplete atomization, changes in sensitivity, etc.). Strip chart recorders shall have a full scale deflection time of 0.2 s or less to ensure accuracy.

28.7 *Automatic Sampling* is recommended if available.

29. Reagents and Materials

29.1 *Copper Solution, Stock* (1.0 mL = 1.0 mg Cu)—See 20.3.

29.2 *Copper Solution, Intermediate* (1.0 mL = 10 µg Cu)—See 20.4.

29.3 *Copper Solution, Standard* (1.0 mL = 0.10 µg Cu)—Dilute 10.0 mL of copper intermediate solution (29.2) and 1 mL of HNO_3 (sp gr 1.42) to 1 L with water. This standard is used to prepare working standards at the time of the analysis.

29.4 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3). (See Note 5.)

29.5 *Argon,* standard, welders grade, commercially available. Nitrogen may also be used if recommended by the instrument manufacturer.

30. Standardization

30.1 Initially, set the instrument according to the manufacturer's specifications. Follow the general instructions as provided in Practice D 3919.

31. Procedure

31.1 Clean all glassware to be used for preparation of standard solutions or in the digestion step, or both, by rinsing first with HNO_3 (1+1) and then with water. Alternatively, soaking the glassware overnight in HNO_3 (1+1) is useful for low levels.

31.2 Measure 100.0 mL of each standard and well-mixed sample into 125-mL beakers or flasks.

31.3 For total recoverable copper add HNO_3 (sp gr 1.42) to each standard and sample at a rate of 5 mL/L and proceed as directed in 31.4 through 31.6. If only dissolved copper is to be determined, filter the sample through a 0.45-µm membrane filter prior to acidification, add HNO_3 (sp gr 1.42) to each standard and sample at a rate of 5 mL/L, and proceed to 31.6.

31.4 Heat the samples at 95°C on a steam bath or hotplate in a well-ventilated fume hood until the volume has been reduced to 15 to 20 mL making certain that the samples do not boil. (See Note 7.)

31.5 Cool and filter the sample through a suitable filter (such as fine-textured, acid-washed, ashless paper) into a 100-mL volumetric flask. Wash the filter paper 2 or 3 times with water and bring to volume (see Note 16). The acid concentration at this point should be 0.5 % HNO_3.

NOTE 16—If suspended material is not present, this filtration may be omitted, but the sample must still be diluted to 100 mL.

31.6 Inject a measured aliquot of sample into the furnace device following the directions as provided by the particular instrument manufacturer. Refer to Practice D 3919.

TABLE 3 Determination of Bias and Overall Precision for Test Method C

Amount Added, µg Cu/L	Amount Found, µg Cu/L	S_T	± Bias	Bias, ± %	Statistically Significant, 95 % Confidence Level
		Reagent Water			
32	31.3	4.54	−0.7	−2.2	No
11	11.7	1.33	+0.7	+6.4	No
5	5.6	1.65	+0.6	+12.0	No
		Waters of Choice			
32	36.3	9.15	+4.3	+13.4	No
11	12.0	2.57	+1.0	+9.1	No
5	9.0	6.96	+4.0	+80.0	No

32. Calculation

32.1 Determine the concentration of copper in each sample by referring to Practice D 3919.

33. Precision and Bias[5]

33.1 The precision and bias of this test method were tested in reagent water by 16 laboratories. Thirteen laboratories also tested this test method in either boiler blowdown water, lake water, tap water, filtered tap water, condensate, well water, or production plant process waters as a water of choice. One laboratory reported data for two operators. Although multiple injections may have been made, the report sheets provided allowed only for reporting single values. Thus, no single operator precision data can be calculated. Two sets of laboratory data were rejected from both the reagent water series and the water of choice series because of either the laboratory ranking test or the individual outlier test. Bias data and overall precision data are given in Table 3.

33.2 These data may not apply to waters of other matrices, therefore, it is the responsibility of the analyst to assure the validity of this test method in a particular matrix.

34. Keywords

34.1 atomic absorption; chelation; copper; flame; graphite furnace; water

---

[5] Supporting data are available from ASTM Headquarters. Request RR: D19-1098.

APPENDIX

(Nonmandatory Information)

X1. RATIONALE FOR DISCONTINUATION OF TEST METHODS

X1.1 Colorimetric Test Methods for Determination of Copper in Water:

X1.1.1 These test methods were discontinued in 1988. They were last published in their entirety in the 1988 *Annual Book of ASTM Standards*, Vol 11.01.

X1.1.2 Former Test Method A, Necuproine (for concentrations of copper in the range from 0.05 to 5 mg/L):

X1.1.2.1 This test method is applicable to the determination of copper in water and waste water containing 0.05 mg/L of copper or more.

X1.1.2.2 (*a*) This test method is based on the measurement of the intensity of the yellow color of the cuprous complex of 2,9-dimethyl-1, 10-phenanthroline (neocuproine). Full development of the color takes place over the pH range from 2.3 to 9.0. However, a buffer solution is used to produce an aqueous phase with a pH of 4.0 to 6.0.

(*b*) The copper is reduced with hydroxylamine hydrochloride and the pH of the solution is adjusted with a sodium citrate solution. The cuprous ion is then reacted with 2,9-dimethyl-1, 10-phenanthroline and the yellow complex extracted with chloroform. Any of the usual photometric or visual methods may be used for measuring or comparing the color. The test method follows Beer's law up to a concentration of 5 mg/L of copper. The maximum absorption occurs at 457 nm.

X1.1.3 Former Test Method B, Necuproine (for concentrations of copper in the range from 2 to 100 µg/L):

X1.1.3.1 This test method is applicable to the determination of copper in waters such as steam condensate and deionized water. It is specifically applicable to concentrations of copper from 2 to 1000 µg/L.

X1.1.3.2 This test method is the same as former Test Method A (for high-level neocuproine), except that a choice between chloroform and isoamyl alcohol is given as the organic solvent used for extraction. The maximum absorption occurs at 457 nm when chloroform is the extractant and at 454 nm when isoamyl alcohol is the extractant.

X1.1.4 Former Test Method C, Cuprethol (for concentrations of copper in the range from 0.05 to 4 mg/L):

X1.1.4.1 This test method is applicable to the determination of copper in water containing 0.05 mg/L of copper or more. Former Test Method C is preferred for relatively unpolluted waters since it does not involve an organic extraction step, and allows for a rapid determination.

X1.1.4.2 Cupric ions form a yellow-colored chelate with cuprethol, the trivial name for the reagent, bis(2-hydroxyethyl)-dithiocarbamate. The colored compound formed at a pH between 5 and 6 is soluble. The maximum absorption occurs at 435 nm and Beer's law is valid up to a copper concentration of 2 mg/L. Any of the usual photoelectric or visual methods may be used for measuring or comparing the color.

X1.1.5 These test methods were discontinued because there were insufficient laboratories interested in participating in a collaborative study to obtain the necessary precision and bias data as required by Practice D 2777.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1785: Standard Specification for Poly (Vinyl Chloride)(PVC) Plastic Pipe, Schedules 40, 80, and 120

CFR Section(s): 46 CFR 56.01-2

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 1785 – 86

Standard Specification for Poly(Vinyl Chloride) (PVC) Plastic Pipe, Schedules 40, 80, and 120[1]

This standard is issued under the fixed designation D 1785; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This specification has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This specification covers poly(vinyl chloride) (PVC) pipe made in Schedule 40, 80, and 120 sizes and pressure-rated for water (see Appendix). Included are criteria for classifying PVC plastic pipe materials and PVC plastic pipe, a system of nomenclature for PVC plastic pipe, and requirements and test methods for materials, workmanship, dimensions, sustained pressure, burst pressure, flattening, and extrusion quality. Methods of marking are also given.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The following precautionary caveat pertains only to the test method portion, Section 7, of this specification. *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* A specific precautionary statement is given in Note 6.

NOTE 1—CPVC plastic pipes, Schedules 40 and 80, which were formerly included in this standard, are now covered by Specification F 441, Chlorinated Poly(Vinyl Chloride) (CPVC) Plastic Pipe, Schedules 40 and 80.[2]

NOTE 2—The sustained and burst pressure test requirements, and the pressure ratings in the Appendix, are calculated from stress values obtained from tests made on pipe 4 in. (100 mm) and smaller. However, tests conducted on pipe as large as 24 in. (600 mm) diameter have shown these stress values to be valid for larger diameter PVC pipe.

NOTE 3—PVC pipe made to this specification is often belled for use as line pipe. For details of the solvent cement belled pipe, see Specification D 2672 and for details of belled elastomeric joints, see Specifications D 3139 and D 3212.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Methods of Conditioning Plastics and Electrical Insulating Materials for Testing[3]

D 1598 Test Method for Time-To-Failure of Plastic Pipe Under Constant Internal Pressure[2]

D 1599 Test Method for Short-Time Hydraulic Failure Pressure of Plastic Pipe, Tubing, and Fittings[2]

D 1600 Abbreviations of Terms Relating to Plastics[4]

D 1784 Specification for Rigid Poly(Vinyl Chloride) (PVC) Compounds and Chlorinated Poly(Vinyl Chloride) (CPVC) Compounds[5]

D 2122 Method of Determining Dimensions of Thermoplastic Pipe and Fittings[2]

D 2152 Test Method for Degree of Fusion of Extruded Poly(Vinyl Chloride) Pipe and Molding Fittings by Acetone Immersion[2]

D 2672 Specification for Solvent Cement Joint Sockets on Belled PVC Pressure Pipe[2]

D 2837 Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials[2]

D 3139 Specification for Joints for Plastic Pressure Pipes Using Flexible Elastomeric Seals[2]

D 3212 Specification for Joints for Drain and Sewer Plastic Pipes Using Flexible Elastomeric Seals[2]

F 412 Definitions of Terms Relating to Plastic Piping Systems[2]

2.2 *Federal Standard:*[6]

Fed. Std. No. 123 Marking for Shipment (Civil Agencies)

2.3 *Military Standard:*[6]

MIL-STD-129 Marking for Shipment and Storage

3. Terminology

3.1 *Definitions:*

3.1.1 *General*—Definitions are in accordance with Definitions F 412 and abbreviations are in accordance with Abbreviations D 1600, unless otherwise indicated. The abbreviation for poly(vinyl chloride) plastic is PVC.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *hydrostatic design stress*—the estimated maximum tensile stress in the wall of the pipe in the circumferential orientation due to internal hydrostatic water pressure that can be applied continuously with a high degree of certainty that failure of the pipe will not occur.

3.2.2 *pressure rating (PR)*—the estimated maximum pressure that water in the pipe can exert continuously with a high degree of certainty that failure of the pipe will not occur.

3.2.3 *relation between dimensions, design stress, and pressure rating*—the following expression, commonly known as

---

[1] This specification is under the jurisdiction of ASTM Committee F-17 on Plastic Piping Systems and is the direct responsibility of Subcommittee F17.25 on Vinyl Based Pipe.

Current edition approved Oct. 31, 1986. Published December 1986. Originally published as D 1785 – 60. Last previous edition D 1785 – $83^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 08.04.

[3] *Annual Book of ASTM Standards*, Vol 08.01.

[4] *Annual Book of ASTM Standards*, Vols 08.01 and 08.04.

[5] *Annual Book of ASTM Standards*, Vols 08.02 and 08.04.

[6] Available from Naval Publications and Forms Center, 5801 Tabor Ave., Philadelphia, PA 19120.

the ISO equation,[7] is used in this specification to relate dimensions, hydrostatic design stress, and pressure rating:

$$2S/P = (D_0/t) - 1$$

where:

S = hydrostatic design stress, psi (or MPa),
P = pressure rating, psi (or MPa),
D_0 = average outside diameter, in. (or mm), and
t = minimal wall thickness, in. (or mm).

3.2.4 *standard thermoplastic pipe materials designation code*—the pipe materials designation code shall consist of the abbreviation PVC for the type of plastic, followed by the ASTM type and grade in Arabic numerals and the design stress in units of 100 psi (0.7 MPa) with any decimal figures dropped. When the design stress code contains less than two figures, a cipher shall be used before the number. Thus a complete material code shall consist of three letters and four figures for PVC plastic pipe materials (see Section 5).

4. Classification

4.1 *General*—This specification covers PVC pipe made to and marked with one of six type/grade/design stress designations (see Appendix X1.2) in Schedule 40, 80, and 120 wall sizes.

4.2 *Hydrostatic Design Stresses*—This specification covers pipe made from PVC plastics as defined by four hydrostatic design stresses which have been developed on the basis of long-term tests (Appendix).

5. Materials and Manufacturer

5.1 *General*—Poly(vinyl chloride) plastics used to make pipe meeting the requirements of this specification are categorized by means of two criteria, namely, (*1*) short-term strength tests and (*2*) long-term strength tests.

NOTE 4—PVC pipe that is intended for use in the transport of potable water should be evaluated for this purpose by a laboratory recognized by the public health profession and by the regulatory bodies having jurisdiction. Many public health authorities recognize National Sanitation Foundation Standard No. 14 for Plastic Piping System Components and Related Materials, as a suitable standard to evaluate materials for potable water service. The seal or mark of the laboratory making the evaluation should be included in the marking on pipe that is intended for the transport of potable water. A laboratory that makes evaluations of pipe for transport of potable water is the National Sanitation Foundation Testing Laboratories, Inc., NSF Bldg., Ann Arbor, MI 48106. Names of other recognized laboratories will be added when they are brought to the attention of ASTM.

5.2 *Basic Materials*—This specification covers pipe made from PVC plastics having certain physical and chemical properties as described in Specification D 1784.

5.3 *Compound*—The PVC compounds used for this pipe shall equal or exceed the following classes described in Specification D 1784; PVC 12454-B, 12454-C, or 14333-D.

5.4 *Rework Material*—Clean, rework material of the same type and grade (cell classification), generated from the manufacturer's own pipe production, may be used by the same manufacturer, as long as the pipe produced meets all the requirements of this specification.

6. Requirements

6.1 *Dimensions and Tolerances:*

6.1.1 Dimensions and tolerances shall be as shown in Tables 1 and 2 when measured in accordance with Method D 2122. The tolerances for out-of-roundness shall apply only to pipe prior to shipment.

6.2 *Sustained Pressure*—The pipe shall not fail, balloon, burst, or weep as defined in Test Method D 1598, at the test pressures given in Tables 3, 4, or 5 when tested in accordance with 8.4.

6.2.1 *Accelerated Regression Test*—At the option of the manufacturer, an accelerated regression test may be substituted for the sustained pressure test. The test shall be conducted in accordance with 8.4.1. The pipe shall demonstrate a hydrostatic design basis projection at the 100 000 h intercept that meets the hydrostatic design basis category requirement (see Table 1 and Method D 2837) for the PVC

TABLE 1 Outside Diameters and Tolerances for PVC Plastic Pipe Schedules 40, 80, and 120, in. (mm)

Nominal Pipe Size	Outside Diameter	Tolerances		
		Average	For Maximum and Minimum Diameter (Out-of-Roundness)	
			Schedule 40 sizes 3½ in. and over; Schedule 80 sizes 8 in. and over	Schedule 40 sizes 3 in. and less; Schedule 80 sizes 6 in. and less; Schedule 120 sizes all
⅛	0.405 (10.29)	±0.004 (±0.10)	. . .	±0.008 (±0.20)
¼	0.540 (13.72)	±0.004 (±0.10)	. . .	±0.008 (±0.20)
⅜	0.675 (17.14)	±0.004 (±0.10)	. . .	±0.008 (±0.20)
½	0.840 (21.34)	±0.004 (±0.10)	. . .	±0.008 (±0.20)
¾	1.050 (26.67)	±0.004 (±0.10)	. . .	±0.010 (±0.25)
1	1.315 (33.40)	±0.005 (±0.13)	. . .	±0.010 (±0.25)
1¼	1.660 (42.16)	±0.005 (±0.13)	. . .	±0.012 (±0.30)
1½	1.900 (48.26)	±0.006 (±0.15)	. . .	±0.012 (±0.30)
2	2.375 (60.32)	±0.006 (±0.15)	. . .	±0.012 (±0.30)
2½	2.875 (73.02)	±0.007 (±0.18)	. . .	±0.015 (±0.38)
3	3.500 (88.90)	±0.008 (±0.20)	. . .	±0.015 (±0.38)
3½	4.000 (101.60)	±0.008 (±0.20)	±0.050 (±1.27)	±0.015 (±0.38)
4	4.500 (114.30)	±0.009 (±0.23)	±0.050 (±1.27)	±0.015 (±0.38)
5	5.563 (141.30)	±0.010 (±0.25)	±0.050 (±1.27)	±0.030 (±0.76)
6	6.625 (168.28)	±0.011 (±0.28)	±0.050 (±1.27)	±0.035 (±0.89)
8	8.625 (219.08)	±0.015 (±0.38)	±0.075 (±1.90)	±0.045 (±1.14)
10	10.750 (273.05)	±0.015 (±0.38)	±0.075 (±1.90)	±0.050 (±1.27)
12	12.750 (323.85)	±0.015 (±0.38)	±0.075 (±1.90)	±0.060 (±1.52)

---

[7] ISO R161-1960, Pipes of Plastics Materials for the Transport of Fluids (Outside Diameters and Nominal Pressures) Part 1, Metric Series.

material used in its manufacture. (*Example:* PVC 1120 pipe must have a minimum 100 000 h projection of 3830 psi (26.40 MPa) and 85 % lower confidence limit (LCL).

6.3 *Burst Pressure*—The minimum burst pressures for PVC plastic pipe shall be as given in Table 6, when determined in accordance with Test Method D 1599.

6.4 *Flattening*—There shall be no evidence of splitting, cracking, or breaking when the pipe is tested in accordance with 8.5.

6.5 *Extrusion Quality*—The pipe shall not flake or disintegrate when tested in accordance with Test Method D 2152.

7. Workmanship, Finish, and Appearance

7.1 The pipe shall be homogeneous throughout and free of visible cracks, holes, foreign inclusions, or other defects. The pipe shall be as uniform as commercially practicable in color, opacity, density, and other physical properties.

NOTE 5—Color and transparency or opacity should be specified in the contract or purchase order.

8. Test Methods

8.1 *Conditioning*—Condition the test specimens at 73.4 ± 3.6°F (23 ± 2°C) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Methods D 618, for those tests where conditioning is required.

8.2 *Test Conditions*—Conduct tests in the Standard Laboratory Atmosphere of 73.4 ± 3.6°F (23 ± 2°C) and 50 ± 5 % relative humidity, unless otherwise specified in the test methods or in this specification.

8.3 *Sampling*—The selection of the sample or samples of pipe shall be as agreed upon by the purchaser and seller. In case of no prior agreement, any sample selected by the testing laboratory shall be deemed adequate.

8.3.1 *Test Specimens*—Not less than 50 % of the test specimens required for any pressure test shall have at least a part of the marking in their central sections. The central section is that portion of pipe which is at least one pipe diameter away from an end closure.

8.4 *Sustained Pressure Test*—Select the test specimens at random. Test individually with water at the internal pressures given in Tables 3, 4, and 5, six specimens of pipe, each specimen at least ten times the nominal diameter in length, but not less than 10 in. (250 mm) or more than 3 ft (1 m) between end closures and bearing the permanent marking on the pipe. Maintain the specimens at the pressure indicated for a period of 1000 h. Hold the pressure as closely as possible, but within ±10 psi (±70 kPa). Condition the specimens at the test temperature of 73.4°F (23°C) to within 3.6°F (±2°C). Test in accordance with Test Method D 1598, except maintain the pressure at the values given in Tables 3, 4, or 5 for 1000 h. Failure of one of the six specimens tested is cause for retest of six additional specimens. Failure of one of the six specimens tested in retest shall constitute failure in the test. Evidence of failure of the pipe shall be as defined in Test Method D 1598.

8.4.1 *Accelerated Regression Test*—Test in accordance with procedures in Test Method D 1598, except that restrained-end fittings may be employed. A minimum of six samples will be tested at pressures selected to yield data points as follows:

0.010 to 0.099 h (36 s to 6 min)
0.10 to 0.999 h (6 min to 1 h)
1.00 to 9.999 h
10.0 to 99.999 h
100 to 100+ h
0 to 100+ h (random point)

Additional points may be added if necessary to improve projection or LCL, or both. No points shall be excluded unless an obvious defect is detected in the failure area of the test sample. Characterize the results using the least squares extrapolation described in Method D 2837.

NOTE 6—**Caution:** Since rupture of the test specimen is expected in quick burst and high stress regression testing, well shielded test equip-

TABLE 2 Wall Thicknesses and Tolerances for PVC Plastic Pipe, Schedules 40, 80, and 120,[A,B] in. (mm)

Nominal Pipe Size	Wall Thickness[A]					
	Schedule 40		Schedule 80		Schedule 120	
	Minimum	Tolerance	Minimum	Tolerance	Minimum	Tolerance
⅛	0.068 (1.73)	+0.020 (+0.51)	0.095 (2.41)	+0.020 (+0.51)	...	...
¼	0.088 (2.24)	+0.020 (+0.51)	0.119 (3.02)	+0.020 (+0.51)	...	...
⅜	0.091 (2.31)	+0.020 (+0.51)	0.126 (3.20)	+0.020 (+0.51)	...	...
½	0.109 (2.77)	+0.020 (+0.51)	0.147 (3.73)	+0.020 (+0.51)	0.170 (4.32)	+0.020 (+0.51)
¾	0.113 (2.87)	+0.020 (+0.51)	0.154 (3.91)	+0.020 (+0.51)	0.170 (4.32)	+0.020 (+0.51)
1	0.133 (3.38)	+0.020 (+0.51)	0.179 (4.55)	+0.021 (+0.53)	0.200 (5.08)	+0.024 (+0.61)
1¼	0.140 (3.56)	+0.020 (+0.51)	0.191 (4.85)	+0.023 (+0.58)	0.215 (5.46)	+0.026 (+0.66)
1½	0.145 (3.68)	+0.020 (+0.51)	0.200 (5.08)	+0.024 (+0.61)	0.225 (5.72)	+0.027 (+0.68)
2	0.154 (3.91)	+0.020 (+0.51)	0.218 (5.54)	+0.026 (+0.66)	0.250 (6.35)	+0.030 (+0.76)
2½	0.203 (5.16)	+0.024 (+0.61)	0.276 (7.01)	+0.033 (+0.84)	0.300 (7.62)	+0.036 (+0.91)
3	0.216 (5.49)	+0.026 (+0.66)	0.300 (7.62)	+0.036 (+0.91)	0.350 (8.89)	+0.042 (+1.07)
3½	0.226 (5.74)	+0.027 (+0.68)	0.318 (8.08)	+0.038 (+0.96)	0.350 (8.89)	+0.042 (+1.07)
4	0.237 (6.02)	+0.028 (+0.71)	0.337 (8.56)	+0.040 (+1.02)	0.437 (11.10)	+0.052 (+1.32)
5	0.258 (6.55)	+0.031 (+0.79)	0.375 (9.52)	+0.045 (+1.14)	0.500 (12.70)	+0.060 (+1.52)
6	0.280 (7.11)	+0.034 (+0.86)	0.432 (10.97)	+0.052 (+1.32)	0.562 (14.27)	+0.067 (+1.70)
8	0.322 (8.18)	+0.039 (+0.99)	0.500 (12.70)	+0.060 (+1.52)	0.718 (18.24)	+0.086 (+2.18)
10	0.365 (9.27)	+0.044 (+1.12)	0.593 (15.06)	+0.071 (+1.80)	0.843 (21.41)	+0.101 (+2.56)
12	0.406 (10.31)	+0.049 (+1.24)	0.687 (17.45)	+0.082 (+2.08)	1.000 (25.40)	+0.120 (+3.05)

[A] The minimum is the lowest wall thickness of the pipe at any cross section. The maximum permitted wall thickness, at any cross section, is the minimum wall thickness plus the stated tolerance. All tolerances are on the plus side of the minimum requirement.

[B] These dimensions conform to nominal IPS dimensions, with the exception that Schedule 120 wall thickness for pipe sizes ½ to 3½ in. (12.5 to 87.5 mm), inclusive, are special PVC plastic pipe sizes.

TABLE 3 Sustained Pressure Test Conditions for Water at 73°F (23°C) for PVC Plastic Pipe, Schedule 40

Nominal Pipe Size	Pressure Required for Test[A]			
	PVC1120 PVC1220 PVC2120	PVC2116	PVC2112	PVC2110
in.	psi			
⅛	1690	1360	1130	930
¼	1640	1310	1090	900
⅜	1310	1050	870	720
½	1250	1000	840	690
¾	1010	810	680	550
1	950	760	630	520
1¼	770	620	520	420
1½	690	560	460	380
2	580	470	390	320
2½	640	510	430	350
3	590	440	370	300
3½	500	400	340	280
4	470	370	310	260
5	410	330	270	220
6	370	300	250	200
8	330	260	220	180
10	300	240	200	160
12	280	220	180	150
in.	MPa			
⅛	11.65	9.38	7.79	6.41
¼	11.31	9.03	7.52	6.21
⅜	9.03	7.24	6.00	4.96
½	8.62	6.89	5.79	4.76
¾	6.96	5.58	4.69	3.79
1	6.55	5.24	4.34	3.59
1¼	5.31	4.27	3.59	2.90
1½	4.76	3.86	3.17	2.62
2	4.00	3.24	2.69	2.21
2½	4.41	3.52	2.96	2.41
3	4.07	3.03	2.55	2.07
3½	3.45	2.76	2.34	1.93
4	3.24	2.55	2.14	1.79
5	2.83	2.28	1.86	1.52
6	2.55	2.07	1.72	1.38
8	2.28	1.79	1.52	1.24
10	2.07	1.65	1.38	1.10
12	1.93	1.52	1.24	1.03

[A] The fiber stresses used to derive these test pressures are as follows:

	psi	MPa
PVC1120	4200	29.0
PVC1220	4200	29.0
PVC2120	4200	29.0
PVC2116	3360	23.2
PVC2112	2800	19.3
PVC2110	2300	15.9

TABLE 4 Sustained Pressure Test Conditions for Water at 73°F (23°C) for PVC Plastic Pipe, Schedule 80

Nominal Pipe Size	Pressure Required for Test[A]			
	PVC1120 PVC1220 PVC2120	PVC2116	PVC2112	PVC2110
in.	psi			
⅛	2570	2060	1720	1410
¼	2370	1900	1580	1300
⅜	1930	1540	1290	1060
½	1780	1430	1190	980
¾	1440	1160	960	790
1	1320	1060	880	720
1¼	1090	870	730	600
1½	990	790	660	540
2	850	680	570	460
2½	890	710	590	490
3	790	630	520	430
3½	730	580	480	400
4	680	540	450	370
5	610	490	400	330
6	590	470	390	320
8	520	410	340	280
10	490	390	330	270
12	480	380	320	260
in.	MPa			
⅛	17.72	14.21	11.86	9.72
¼	16.34	13.10	10.90	8.96
⅜	13.31	10.62	8.89	7.31
½	12.27	9.86	8.20	6.76
¾	9.93	8.00	6.62	5.45
1	9.10	7.31	6.07	4.96
1¼	7.52	6.00	5.03	4.14
1½	6.83	4.96	4.55	3.72
2	5.86	4.69	3.93	3.17
2½	6.14	4.90	4.07	3.38
3	5.45	4.34	3.59	2.96
3½	5.03	4.00	3.31	2.76
4	4.69	3.72	3.10	2.55
5	4.21	3.38	2.76	2.28
6	4.07	3.24	2.69	2.21
8	3.59	2.83	2.34	1.93
10	3.38	2.69	2.28	1.86
12	3.31	2.62	2.21	1.79

[A] The fiber stresses used to derive these test pressures are as follows:

	psi	MPa
PVC1120	4200	29.0
PVC1220	4200	29.0
PVC2120	4200	29.0
PVC2116	3360	23.2
PVC2112	2800	19.3
PVC2110	2300	15.9

ment and protective personal equipment should be used when conducting the tests.

8.5 *Burst Pressure*—Determine the minimum burst pressure with at least five specimens in accordance with Test Method D 1599. The time of testing of each specimen shall be between 60 and 70 s.

8.6 *Flattening*—Flatten three specimens of the pipe, 2 in. (50 mm) long, between parallel plates in a suitable press until the distance between the plates is 40 % of the outside diameter of the pipe or the walls of the pipe touch, whichever occurs first. The rate of loading shall be uniform and such that the compression is completed within 2 to 5 min. On removal of the load examine the specimens for evidence of splitting, cracking, or breaking.

9. Retest and Rejection

9.1 If any failure occurs, the materials may be retested to establish conformity in accordance with agreement between the purchaser and the seller.

10. Certification

10.1 The seal of the National Sanitation Foundation Testing Laboratory, Inc. indicates that the product is tested under the NSF certification program.

10.2 Certification and labeling, by other independent laboratories, may be accepted if approved by the code authority having jurisdiction.

11. Product Marking

11.1 *Quality of Marking*—The marking shall be applied to the pipe in such a manner that it remains legible (easily read) after installation and inspection.

11.2 *Content of Marking:*

11.2.1 Marking on the pipe shall include the following, spaced at intervals of not more than 5 ft (1.5 m):

11.2.1.1 Nominal pipe size (for example, 2 in. (50 mm)),

11.2.1.2 Type of plastic pipe material in accordance with the designation code prescribed in 4.5, for example, PVC1120,

11.2.1.3 Schedule (40, 80, or 120, whichever is applicable) and the pressure rating in pounds per square inch (megapascals) for water at 73°F (23°C) shown as the number followed by psi (for example, 200 psi (1.4 MPa)). When the indicated pressure rating is lower than that calculated in accordance with 3.4 (see Appendix), this shall be indicated by placing a star after the pressure rating,

11.2.1.4 ASTM designation D 1785, with which the pipe complies,

11.2.1.5 Manufacturer's name (or trademark) and code (see Note 3), and

TABLE 5 Sustained Pressure Test Conditions for Water at 73°F (23°C) for PVC Plastic Pipe, Schedule 120

Nominal Pipe Size	Pressure Required for Test[A]			
	PVC1120 PVC1220 PVC2120	PVC2116	PVC2112	PVC2110
in.	psi			
½	2130	1710	1420	1170
¾	1620	1300	1080	890
1	1510	1200	1000	830
1¼	1250	1000	830	680
1½	1130	900	750	620
2	990	790	660	540
2½	980	780	650	540
3	930	750	620	510
3½	810	640	540	440
4	900	720	600	490
5	830	660	550	450
6	780	620	520	430
8	760	610	510	420
10	770	620	510	420
12	710	570	480	390

TABLE 5 *Continued*

Nominal Pipe Size	Pressure Required for Test[A]			
	PVC1120 PVC1220 PVC2120	PVC2116	PVC2112	PVC2110
in.	MPa			
½	14.69	11.79	9.79	8.07
¾	11.17	8.96	7.45	6.14
1	10.41	8.27	6.89	5.72
1¼	8.62	6.89	5.72	4.69
1½	7.79	6.21	5.17	4.27
2	6.83	5.45	4.55	3.72
2½	6.76	5.38	4.48	3.72
3	6.41	5.17	4.27	3.52
3½	5.58	4.41	3.72	3.03
4	6.21	4.96	4.14	3.38
5	5.72	4.55	3.79	3.10
6	5.38	4.27	3.59	2.96
8	5.24	4.21	3.52	2.90
10	5.31	4.27	3.52	2.90
12	4.90	3.93	3.31	2.69

[A] The fiber stresses used to derive these test pressures are as follows:

	psi	MPa
PVC1120	4200	29.0
PVC1220	4200	29.0
PVC2120	4200	29.0
PVC2116	3360	23.2
PVC2112	2800	19.3
PVC2110	2300	15.9

11.2.1.6 Pipe intended for the transport of potable water shall also include the seal or mark of the laboratory making the evaluation for this purpose, spaced at intervals specified by the laboratory.

NOTE 7—Manufacturers using the seal or mark of a laboratory must obtain prior authorization from the laboratory concerned.

NOTE 8—It is common practice to dual mark Schedule 40 piping for potable water and DWV usage in which compliance with each applicable standard is met.

12. Quality Assurance

12.1 *Quality Assurance*—When the product is marked with this designation "ASTM D 1785", the manufacturer affirms that this product was inspected, sampled, and tested in accordance with this specification and has been found to meet the requirements of this specification.

TABLE 6 Burst Pressure Requirements for Water at 73°F (23°C) for PVC Plastic Pipe, Schedules 40, 80, and 120

Nominal Pipe Size	Minimum Burst Pressures[A]					
	Schedule 40		Schedule 80		Schedule 120	
	PVC1120 PVC1220 PVC2120	PVC2112 PVC2116 PVC2110	PVC1120 PVC1220 PVC2120	PVC2112 PVC2116 PVC2110	PVC1120 PVC1220 PVC2120	PVC2112 PVC2116 PVC2110
in.			psi			
⅛	2580	2020	3920	3060	...	...
¼	2490	1950	3620	2830	...	...
⅜	1990	1560	2940	2300	...	...
½	1910	1490	2720	2120	3250	2540
¾	1540	1210	2200	1720	2470	1930
1	1440	1130	2020	1580	2300	1790
1¼	1180	920	1660	1300	1900	1490
1½	1060	830	1510	1180	1720	1340
2	890	690	1290	1010	1510	1180
2½	970	760	1360	1060	1490	1170
3	840	660	1200	940	1420	1110
3½	770	600	1110	860	1230	960
4	710	560	1040	810	1380	1080
5	620	390	930	720	1260	990
6	560	440	890	700	1190	930
8	500	390	790	620	1160	910
10	450	350	750	580	1170	920
12	420	330	730	570	1090	850
in.			MPa			
⅛	17.79	13.93	27.03	21.10	...	...
¼	17.17	13.45	24.96	19.52	...	...
⅜	13.72	10.76	20.27	15.86	...	...
½	13.17	10.27	18.76	14.62	22.41	17.52
¾	10.62	8.34	15.17	11.86	17.03	13.31
1	9.93	7.79	13.93	10.89	15.86	12.34
1¼	8.14	6.34	11.45	8.96	13.10	10.27
1½	7.31	5.72	10.41	8.14	11.86	9.24
2	6.14	4.76	8.89	6.96	10.41	8.14
2½	6.69	5.24	9.38	7.31	10.27	8.07
3	5.79	4.55	8.27	6.48	9.79	7.65
3½	5.31	4.14	7.65	5.93	8.48	6.62
4	4.90	3.86	7.17	5.58	9.51	7.45
5	4.27	2.69	6.41	4.96	8.69	6.83
6	3.86	3.03	6.14	4.83	8.20	6.41
8	3.45	2.69	5.45	4.27	8.00	6.27
10	3.10	2.41	5.17	4.00	8.07	6.34
12	2.90	2.28	5.03	3.93	7.52	5.86

[A] The fiber stresses used to derive these test pressures are as follows:

	psi	MPa
PVC1120	6400	44.1
PVC1220	6400	44.1
PVC2120	6400	44.1
PVC2116	5000	34.5
PVC2112	5000	34.5
PVC2110	5000	34.5

GOVERNMENT/MILITARY PROCUREMENT

These requirements apply *only* to Federal/Military procurement, not domestic sales or transfers.

S1. *Responsibility for Inspection*—Unless otherwise specified in the contract or purchase order, the producer is responsible for the performance of all inspection and test requirements specified herein. The producer may use his own or any other suitable facilities for the performance of the inspection and test requirements specified herein, unless the purchaser disapproves. The purchaser shall have the right to perform any of the inspections and tests set forth in this specification where such inspections are deemed necessary to ensure that material conforms to prescribed requirements.

NOTE S1—In U.S. Federal contracts, the contractor is responsible for inspection.

S2. *Packaging and Marking for U.S. Government Procurement:*

S2.1 *Packaging*—Unless otherwise specified in the contract, the materials shall be packaged in accordance with the supplier's standard practice in a manner ensuring arrival at destination in satisfactory condition and which will be acceptable to the carrier at lowest rates. Containers and packing shall comply with Uniform Freight Classification rules or National Motor Freight Classification rules.

S2.2 *Marking*—Marking for shipment shall be in accordance with Fed. Std. No. 123 for military agencies.

NOTE S2—The inclusion of U.S. Government procurement requirements should not be construed as an indication that the U.S. Government uses or endorses the products described in this document.

APPENDIX

(Nonmandatory Information)

X1. SOURCE OF HYDROSTATIC DESIGN STRESSES

TABLE X1.1 Water Pressure Ratings at 73°F (23°C) for Schedule 40 PVC Plastic Pipe

Nominal Pipe Size	Pressure Ratings[A]			
	PVC1120[B] PVC1220 PVC2120	PVC2116	PVC2112[B]	PVC2110[B]
in.	psi			
⅛	810	650	500	400
¼	780	620	490	390
⅜	620	500	390	310
½	600	480	370	300
¾	480	390	300	240
1	450	360	280	220
1¼	370	290	230	180
1½	330	260	210	170
2	280	220	170	140
2½	300	240	190	150
3	260	210	160	130
3½	240	190	150	120
4	220	180	140	110
5	190	160	120	100
6	180	140	110	90
8	160	120	100	80
10	140	110	90	70
12	130	110	80	70
in.	MPa (bar)[C]			
⅛	5.58 (56)	4.48 (45)	3.45 (34)	2.76 (28)
¼	5.38 (54)	4.27 (43)	3.38 (33)	2.69 (27)
⅜	4.27 (43)	3.45 (34)	2.69 (27)	2.14 (21)
½	4.14 (41)	3.31 (33)	2.55 (25)	2.07 (21)
¾	3.31 (33)	2.69 (27)	2.07 (21)	1.65 (16)
1	3.10 (31)	2.48 (25)	1.93 (19)	1.52 (15)
1¼	2.55 (25)	2.04 (20)	1.59 (16)	1.24 (12)
1½	2.28 (23)	1.79 (18)	1.45 (14)	1.17 (12)
2	1.93 (19)	1.52 (15)	1.17 (12)	0.97 (9.7)
2½	2.07 (21)	1.65 (16)	1.31 (13)	1.03 (10)
3	1.79 (28)	1.45 (14)	1.10 (11)	0.90 (9.0)
3½	1.65 (16)	1.31 (13)	1.03 (10)	0.83 (8.3)
4	1.52 (15)	1.24 (12)	0.97 (9.7)	0.76 (7.6)
5	1.31 (13)	1.10 (11)	0.83 (8.3)	0.69 (6.9)
6	1.24 (12)	0.97 (9.7)	0.76 (7.6)	0.62 (6.2)
8	1.10 (11)	0.83 (8.3)	0.69 (6.9)	0.55 (5.5)
10	0.97 (9.7)	0.76 (7.6)	0.62 (6.2)	0.48 (4.8)
12	0.90 (9.0)	0.76 (7.6)	0.55 (5.5)	0.48 (4.8)

[A] These pressure ratings apply only to unthreaded pipe. The industry does not recommend threading PVC plastic pipe in Schedule 40 dimensions in nominal pipe sizes 6 in. (150 mm) and smaller.

[B] See Appendix for code designation.

[C] 1 bar = 14.504 psi.

X1.1 The hydrostatic design stresses recommended by the Plastics Pipe Institute are used to pressure rate PVC plastic pipe. These hydrostatic design stresses are 2000 psi (14 MPa), 1600 psi (11.2 MPa), 1250 psi (8.7 MPa, and 1000 psi (7 MPa) for water at 73°F (23°C). These hydrostatic design stresses apply only to pipe meeting all the requirements of this specification.

X1.2 Six PVC pipe materials are included based on the requirements of Specification D 1784 and the PPI-recommended hydrostatic design stresses as follows:

X1.2.1 Type I, Grade 1 (12454-B), with a hydrostatic design stress of 2000 psi (14 MPa), designated as PVC1120.

X1.2.2 Type I, Grade 2 (12454-C), with a hydrostatic design stress of 2000 psi (14 MPa), designated as PVC1220.

X1.2.3 Type II, Grade 1 (14333-D), with a hydrostatic design stress of 2000 psi (14 MPa), designated as PVC2120.

X1.2.4 Type II, Grade I (14333-D), with a hydrostatic design stress of 1600 psi (11.2 MPa), designated as PVC2116.

X1.2.5 Type II, Grade 1 (14333-D), with a hydrostatic design stress of 1250 psi (8.7 MPa), designated as PVC2112.

X1.2.6 Type II, Grade 1 (14333-D), with a hydrostatic design stress of 1000 psi (7.0 MPa), designated as PVC2110.

X1.3 The standard method for obtaining hydrostatic basis for thermoplastic pipe materials is Method D 2837. Additional information regarding the method of test and other criteria used in developing these hydrostatic design stresses may be obtained from the Plastics Pipe Institute, a division of The Society of the Plastics Industry, 355 Lexington Ave., New York, NY 10017. These hydrostatic design stresses may not be suitable for materials that show a wide departure from a straight-line plot of log stress versus log time to failure. All the data available to date on PVC pipe materials made in the United States exhibit a straight-line plot under these plotting conditions.

X1.4 The pipe is rated for use with water at 73°F (23°C) at the maximum internal pressures shown in Tables X1.1, X1.2, and X1.3. Lower pressure ratings than those calculated in accordance with 3.4 may be recommended, at the option of the pipe manufacturer, in which case the SDR shall be included in the marking. Experience of the industry indicates that PVC plastic pipe meeting the requirements of this specification gives satisfactory service under normal conditions for a long period at these pressure ratings. The sustained pressure requirements are related to these ratings through the slopes of the strength-time plots of these materials in pipe form.

X1.5 The hydrostatic design stresses recommended by the Plastics Pipe Institute are based on tests made on pipe ranging in size from ½ to 2½ in. (12.5 to 63.5 mm).

TABLE X1.2 Water Pressure Ratings at 73°F (23°C) for Schedule 80 PVC Plastic Pipe

Nominal Pipe Size, in.	psi							
	PVC1120, PVC1220, PVC2120		PVC2116		PVC2112		PVC2110	
	Unthreaded	Threaded	Unthreaded	Threaded	Unthreaded	Threaded	Unthreaded	Threaded
⅛	1230	610	980	490	770	380	610	310
¼	1130	570	900	450	710	350	570	280
⅜	920	460	730	370	570	290	460	230
½	850	420	680	340	530	260	420	210
¾	690	340	550	280	430	210	340	170
1	630	320	500	250	390	200	320	160
1¼	520	260	420	210	320	160	260	130
1½	470	240	380	190	290	150	240	120
2	400	200	320	160	250	130	200	100
2½	420	210	340	170	260	130	210	110
3	370	190	300	150	230	120	190	90
3½	350	170	280	140	220	110	170	90
4	320	160	260	130	200	100	160	80
5	290	140	230	120	180	90	140	70
6	280	140	220	110	170	90	140	70
8	250	120	200	100	150	80	120	60
10	230	120	190	90	150	70	120	60
12	230	110	180	90	140	70	110	60

Nominal Pipe Size, in.	MPa (bar)							
	PVC1120, PVC1220, PVC2120		PVC2116		PVC2112		PVC2110	
	Unthreaded	Threaded	Unthreaded	Threaded	Unthreaded	Threaded	Unthreaded	Threaded
⅛	8.48 (85)	4.21 (42)	6.76 (68)	3.38 (39)	5.31 (53)	2.62 (26)	4.21 (42)	2.14 (21)
¼	7.79 (80)	3.93 (39)	6.21 (62)	3.10 (31)	4.90 (49)	2.41 (24)	3.93 (40)	1.93 (19)
⅜	6.34 (63)	3.17 (32)	5.03 (50)	2.55 (25)	3.93 (39)	2.00 (20)	3.17 (32)	1.59 (16)
½	5.86 (59)	2.90 (29)	4.69 (47)	2.34 (23)	3.65 (36)	1.79 (18)	2.90 (29)	1.45 (14)
¾	4.76 (48)	2.34 (23)	3.79 (38)	1.93 (19)	2.96 (29)	1.45 (14)	2.34 (23)	1.17 (12)
1	4.34 (43)	2.21 (22)	3.45 (34)	1.72 (17)	2.69 (27)	1.38 (13)	2.21 (22)	1.10 (11)
1¼	3.59 (36)	1.79 (18)	2.90 (29)	1.45 (14)	2.21 (22)	1.10 (11)	1.79 (18)	0.90 (9.0)
1½	3.24 (32)	1.65 (16)	2.62 (26)	1.31 (13)	2.0 (20)	1.03 (10)	1.65 (16)	0.83 (8.3)
2	2.76 (28)	1.38 (14)	2.21 (22)	1.10 (11)	1.72 (17)	0.90 (9.0)	1.38 (14)	0.69 (6.9)
2½	2.90 (29)	1.45 (15)	2.34 (23)	1.17 (12)	1.79 (18)	0.90 (9.0)	1.45 (14)	0.76 (7.6)
3	2.55 (25)	1.31 (13)	2.07 (21)	1.03 (10)	1.59 (16)	0.83 (8.3)	1.31 (13)	0.62 (6.2)
3½	2.41 (24)	1.17 (12)	1.93 (19)	0.97 (9.7)	1.52 (15)	0.76 (7.6)	1.17 (12)	0.62 (6.2)
4	2.21 (22)	1.10 (11)	1.79 (18)	0.90 (9.0)	1.38 (14)	0.69 (6.9)	1.10 (11)	0.55 (5.5)
5	2.00 (20)	0.97 (9.7)	1.59 (16)	0.83 (8.3)	1.24 (12)	0.62 (6.2)	0.97 (9.7)	0.48 (4.8)
6	1.93 (19)	0.97 (9.7)	1.52 (15)	0.76 (7.6)	1.17 (11)	0.62 (6.2)	0.97 (9.7)	0.48 (4.8)
8	1.72 (17)	0.83 (8.3)	1.38 (14)	0.69 (6.9)	1.03 (10)	0.55 (5.5)	0.83 (8.3)	0.41 (4.1)
10	1.59 (16)	0.83 (8.3)	1.31 (13)	0.62 (6.2)	1.03 (10)	0.48 (4.8)	0.83 (8.3)	0.41 (4.1)
12	1.59 (16)	0.76 (7.6)	1.24 (12)	0.62 (6.2)	0.97 (9.7)	0.48 (4.8)	0.76 (7.6)	0.41 (4.1)

TABLE X1.3 Water Pressure Ratings at 73°F (23°C) for Schedule 120 PVC Plastic Pipe

Nominal Pipe Size, in.	psi							
	PVC1120, PVC1220, PVC2120		PVC2116		PVC2112		PVC2110	
	Untreaded	Threaded	Untreaded	Threaded	Unthreaded	Threaded	Unthreaded	Threaded
½	1010	510	810	410	630	320	510	250
¾	770	390	620	310	480	240	390	190
1	720	360	570	290	450	220	360	180
1¼	600	300	480	240	370	190	300	150
1½	540	270	430	210	340	170	270	130
2	470	240	380	190	290	150	240	120
2½	470	230	370	190	290	150	230	120
3	440	220	360	180	280	140	220	110
3½	380	190	310	150	240	120	190	100
4	430	220	340	170	270	130	220	110
5	400	200	320	160	250	120	200	100
6	370	190	300	150	230	120	190	90
8	380	180	290	140	230	110	180	90
10	370	180	290	140	230	110	180	90
12	340	170	270	140	210	110	170	80

Nominal Pipe Size, in.	MPa (bar)							
	PVC1120, PVC1220, PVC2120		PVC2116		PVC2112		PVC2110	
	Unthreaded	Threaded	Unthreaded	Threaded	Unthreaded	Threaded	Unthreaded	Threaded
½	6.96 (70)	3.52 (35)	5.58 (56)	2.83 (28)	4.34 (43)	2.21 (22)	3.52 (35)	1.72 (17)
¾	5.31 (53)	2.69 (27)	4.27 (43)	2.14 (21)	3.31 (33)	1.65 (16)	2.69 (27)	1.31 (13)
1	4.96 (50)	2.48 (25)	3.93 (39)	2.00 (20)	3.10 (31)	1.52 (15)	2.48 (25)	1.24 (12)
1¼	4.14 (41)	2.07 (21)	3.31 (33)	1.65 (16)	2.55 (25)	1.31 (13)	2.07 (21)	1.03 (10)
1½	3.72 (37)	1.86 (19)	2.96 (30)	1.45 (14)	2.34 (23)	1.17 (12)	1.86 (18)	0.90 (9.0)
2	3.24 (32)	1.65 (17)	2.62 (26)	1.31 (13)	2.00 (20)	1.03 (10)	1.65 (16)	0.83 (8.3)
2½	3.24 (32)	1.59 (16)	2.55 (25)	1.31 (13)	2.00 (20)	1.03 (10)	1.59 (16)	0.83 (8.3)
3	3.03 (30)	1.52 (15)	2.48 (24)	1.24 (12)	1.93 (19)	0.97 (9.7)	1.52 (15)	0.76 (7.6)
3½	2.62 (26)	1.31 (13)	2.14 (21)	1.03 (10)	1.65 (16)	0.83 (8.3)	1.31 (13)	0.69 (6.9)
4	2.96 (29)	1.52 (15)	2.34 (23)	1.17 (12)	1.86 (18)	0.90 (9.0)	1.52 (15)	0.76 (7.6)
5	2.76 (27)	1.38 (14)	2.21 (22)	1.10 (11)	1.72 (17)	0.83 (8.3)	1.38 (14)	0.69 (6.9)
6	2.55 (25)	1.31 (13)	2.07 (21)	1.03 (10)	1.59 (16)	0.83 (8.3)	1.31 (13)	0.62 (6.2)
8	2.62 (26)	1.24 (12)	2.00 (20)	0.97 (9.7)	1.59 (16)	0.76 (7.6)	1.24 (12)	0.62 (6.2)
10	2.55 (25)	1.24 (12)	2.00 (20)	0.97 (9.7)	1.59 (16)	0.76 (7.6)	1.24 (12)	0.62 (6.2)
12	2.34 (23)	1.17 (11)	1.86 (18)	0.97 (9.7)	1.45 (14)	0.76 (7.6)	1.17 (11)	0.55 (5.5)

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By Public.Resource.Org, Inc., a California Nonprofit Organization.

Not Affiliated Or Authorized by ASTM or by the United States Government

Document Name: ASTM D1835: Standard Specification for Liquefied Petroleum (LP) Gases

CFR Section(s): 49 CFR 180.209(e)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

ASTM Logo Removed

Designation: D 1835 – 97

An American National Standard

Standard Specification for Liquefied Petroleum (LP) Gases[1]

This standard is issued under the fixed designation D 1835; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers those products commonly referred to as liquefied petroleum gases.

1.2 This specification is applicable to products intended for use as domestic, commercial, industrial, and engine fuels.

1.3 This specification is for use in formulating specifications for required properties of liquefied petroleum gases at the time of delivery in bulk.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1265 Practice for Sampling Liquefied Petroleum (LP) Gases (Manual Method)[2]

D 1267 Test Method for Gage Vapor Pressure of Liquefied Petroleum (LP) Gases (LP-Gas Method)[2]

D 1657 Test Method for Density or Relative Density of Light Hydrocarbons by Pressure Thermohydrometer[3]

D 1837 Test Method for Volatility of Liquefied Petroleum (LP) Gases[2]

D 1838 Test Method for Copper Strip Corrosion by Liquefied Petroleum (LP) Gases[2]

D 2158 Test Method for Residues in Liquefied Petroleum (LP) Gases[2]

D 2163 Test Method for Analysis of Liquefied Petroleum (LP) Gases and Propene Concentrates by Gas Chromatography[2]

D 2420 Test Method for Hydrogen Sulfide in Liquefied Petroleum (LP) Gases (Lead Acetate Method)[2]

D 2598 Practice for Calculation of Certain Physical Properties of Liquefied Petroleum (LP) Gases from Compositional Analysis[3]

D 2713 Test Method for Dryness of Propane (Valve Freeze Method)[3]

D 2784 Test Method for Sulfur in Liquefied Petroleum Gases (Oxy-Hydrogen Burner or Lamp)[3]

D 3700 Practice for Containing Hydrocarbon Fluid Samples Using a Floating Piston Cylinder[3]

2.2 *Other Document:*

GPA Standard 2140[4]

3. Types

3.1 Four basic types of liquefied petroleum gases are provided to cover the common use applications, as follows:

3.1.1 *Commercial Propane*—A hydrocarbon product for use where high volatility is required. Commercial propane is suitable for certain low severity internal combustion engine applications.

3.1.2 *Commercial Butane*—A hydrocarbon product for use where low volatility is required.

3.1.3 *Commercial PB Mixtures*—Mixtures of propane and butane for use where intermediate volatility is required.

3.1.4 *Special-Duty Propane*—A high-quality product composed chiefly of propane, which exhibits superior antiknock characteristics when used as an internal combustion engine fuel.

4. Detail Requirements

4.1 The four types of liquefied petroleum gases shall conform to the requirements prescribed in Table 1.

5. Sampling

5.1 Proper sampling of liquefied gases is extremely important if the tests are to be significant. Obtain samples for compositional analysis in accordance with Practice D 3700. Samples for other required tests should be obtained in accordance with Practice D 1265.

---

[1] This specification is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.H on Liquefied Petroleum Gas.

Current edition approved Nov. 10, 1997. Published June 1998. Originally published as D1835 – 61 T. Last previous edition D1835 – 91.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] Available from Gas Processors Assn., 6526 E. 60th St., Tulsa, OK 74145.

TABLE 1 Detail Requirements for Liquefied Petroleum Gases

	Product Designation				
	Commercial Propane	Commercial Butane	Commercial PB Mixtures	Special-Duty Propane[A]	ASTM Test Methods (see Section 2)
Vapor pressure at 100°F (37.8°C), max, psig	208	70	[B]	208	D 1267 or
kPa	1434	483		1434	D 2598[C]
Volatile residue:					
evaporated temperature, 95 %, max, °F	−37	36	36	−37	
°C	−38.3	2.2	2.2	−38.3	D 1837
or					
butane and heavier, max, vol %	2.5	...	...	2.5	D 2163
pentane and heavier, max, vol %	...	2.0	2.0	...	D 2163
Propylene content, max, vol %	...	...	...	5.0	D 2163
Residual matter:					
residue on evaporation 100 mL, max, mL	0.05	0.05	0.05	0.05	D 2158
oil stain observation	pass[D]	pass[D]	pass[D]	pass[D]	D 2158
Relative density at 60/60°F (15.6/15.6°C)	[E]	[E]	[E]	...	D 1657 or D 2598
Corrosion, copper, strip	No. 1	No. 1	No. 1	No. 1	D 1838[F]
Sulfur, ppmw	185[G]	140[G]	140[G]	123[G]	D 2784
Hydrogen sulfide	pass	pass	pass	pass	D 2420
Moisture content	pass	...	...	pass	D 2713
Free water content	...	none[H]	none[H]	...	

[A] Equivalent to Propane HD-5 of GPA Standard 2140.

[B] The permissible vapor pressures of products classified as PB mixtures must not exceed 208 psig (1430 kPa) and additionally must not exceed that calculated from the following relationship between the observed vapor pressure and the observed relative density:

Vapor pressure, max = 1167 − 1880 (relative density 60/60°F) or 1167 − 1880 (density at 15°C)

A specific mixture shall be designated by the vapor pressure at 100°F in pounds per square inch gage. To comply with the designation, the vapor pressure of the mixture shall be within + 0 to − 10 psi of the vapor pressure specified.

[C] In case of dispute about the vapor pressure of a product, the value actually determined by Test Method D 1267 shall prevail over the value calculated by Practice D 2598.

[D] An acceptable product shall not yield a persistent oil ring when 0.3 mL of solvent residue mixture is added to a filter paper, in 0.1-mL increments and examined in daylight after 2 min as described in Test Method D 2158.

[E] Although not a specific requirement, the relative density must be determined for other purposes and should be reported. Additionally, the relative density of PB mixture is needed to establish the permissible maximum vapor pressure (see Footnote *B*).

[F] This method may not accurately determine the presence of reactive materials (for example, H_2S, S°) in liquefied petroleum gas if the product contains corrosion inhibitors or other chemicals which diminish the reaction with the copper strip.

[G] The total sulfur limits in these specifications *do include* sulfur compounds used for stenching purposes.

[H] The presence or absence of water shall be determined by visual inspection of the samples on which the relative density is determined.

6. Keywords

6.1 butane; liquefied petroleum (LP) gases specifications; propane

APPENDIX

(Nonmandatory Information)

X1. SIGNIFICANCE OF ASTM SPECIFICATIONS FOR LIQUEFIED PETROLEUM (LP) GASES

X1.1 General

X1.1.1 Liquefied petroleum gas products are composed of those readily liquefiable hydrocarbon compounds which are produced in the course of processing natural gas and also in the course of the conventional refining of crude oil. The composition of liquefied gases can vary widely depending upon the source and the nature of the treatment to which the products have been subjected.

X1.1.2 There are many uses for liquefied petroleum gases. Important uses are, (*1*) as domestic, commercial, and industrial fuels, (*2*) as a carbon source material in metal treating operations, (*3*) as refinery raw materials for synthetic gasoline production, and (*4*) as petrochemical raw materials. The nature of the needs dictates the required composition characteristics in these various applications. Since the last three uses of those listed are in the category of specialty applications which involve special requirements, they are excluded from consideration in the specifications.

X1.1.3 In substance, the ASTM Specifications for Liquefied Petroleum Gases are designed to properly define acceptable products for domestic, commercial, and industrial uses. In many cases it will be found that products meeting the specifications will also be usable in applications other than the ones for which they were designed. The following can be accepted as a general guide in the more common use applications of the three types of fuels:

X1.1.3.1 *Commercial Propane*—This fuel type is adequate for domestic, commercial, and industrial use, particularly in geographical areas and in seasons where low ambient temperatures are common, and where uniformity of fuel is an important consideration. Commercial propane is suitable for certain low severity internal combustion engine applications.

X1.1.3.2 *Commercial PB Mixtures*—This fuel type, since it covers a broad range of mixtures, permits the tailoring of fuels to specific needs. The various mixtures find application as domestic, commercial, and industrial fuel in areas and at times when low ambient temperature conditions are less frequently encountered.

X1.1.3.3 *Commercial Butane*—This fuel type finds limited application as a domestic fuel in areas of warmer climates. It is similarly used in industrial applications where problems of fuel vaporization are not present.

X1.1.3.4 *Special-Duty Propane*—This fuel type is a special liquefied petroleum gas product tailored to meet the restrictive needs of internal combustion engines operating under moderate to high engine severity. Fuel products of this type will be less variable in composition and combustion characteristics than the other products covered by this specification.

X1.2 Significance and Use

X1.2.1 This specification addreses commercial liquefied petroleum gases, consisting of either propane or butane or mixtures thereof. Consequently, the important characteristics of these products can be defined and controlled by a relatively few simple measurements. The specification test methods provided achieve the desired results. The significance of the various tests as they can apply to consumer problems is summarized here.

X1.2.1.1 *Vapor Pressure, Volatility, and Relative Density*:

(*a*) *Vapor Pressure* is an indirect measure of the most extreme low-temperature conditions under which initial vaporization can be expected to take place. It can be considered as a semiquantitative measure of the amount of the most volatile material present in the product. It can also be used as a means for predicting the maximum pressures which may be experienced at fuel tank temperatures. Vapor pressure becomes more significant when it is related to volatility.

(*b*) *Volatility*, expressed in terms of the 95 % evaporated temperature of the product, is a measure of the amount of least volatile fuel component present in the product. Coupled with a vapor pressure limit, it serves to assure essentially single-component products in the cases of commercial propane and commercial butane fuel types. When volatility is coupled with a vapor pressure limit which has been related to gravity, as in the case of the commercial PB-mixture type of fuels, the combination serves to assure essentially two component mixtures for such fuels. When coupled with a proper vapor pressure limit, this measurement serves to assure that special-duty propane products will be composed chiefly of propane and propylene and that propane will be the major constituent.

(*c*) *Relative Density*, by itself, has little significance. It becomes of value only when related to vapor pressure and volatility. Since relative density is of importance in meeting transportation and storage requirements it is always determined for all liquefied petroleum gas products.

X1.2.1.2 *Other Product Characteristics*—While the vaporization and combustion characteristics of commercial liquefied gas products are completely defined for the normal use applications by vapor pressure, volatility, and relative density, as given in X1.2.1.1, there are other items which either affect or might affect the results obtained in some specific use applications. For that reason, limits are specified for residue content, copper corrosion, sulfur content, moisture content, and free water content to provide assurance of product dependability under the more extreme conditions of use.

(*a*) *Residue* is a measure of the concentration of soluble hydrocarbon materials present in the product which are substantially less volatile than the liquefied petroleum gas product being sampled. Control of residue content is of importance in applications where the fuel is used in liquid or vapor feed systems (where fuel vapors are withdrawn from the top of the LPG storage container). In either case, failure to limit the permissible concentration of residue materials may result in troublesome deposits or regulating equipment may become fouled, or both.

(*b*) *Copper Corrosion* limits are for the purpose of providing assurance that difficulties will not be experienced in the deterioration of the copper and copper-alloy fittings and connections which are commonly used in many types of utilization, storage, and transportation equipment. The copper corrosion test will detect the presence of hydrogen sulfide, which is highly toxic. The copper corrosion limits also provide assurance that the LP-Gas will not contain H_2S in such quantities as to present a health and safety hazard if it is known that the product does not contain corrosion inhibitors or other chemicals which diminish the reaction with the copper strip. In addition, Test Method D 2420 is recommended as a field test and added safeguard to ensure that LP-Gas does not contain detectable amounts of hydrogen sulfide.

(*c*) *Sulfur Content* limits are provided to more completely define liquefied petroleum gas products because these products are generally lower in sulfur content than most other petroleum-derived fuels. The limit on sulfur content minimizes sulfur oxide emissions and limits potential corrosion by exhaust gases from combustion of LPG.

(*d*) *Moisture Content* is a measure of the approximate percentage saturation of the product with water. This measurement is a requirement only on the commercial and special duty propane types of liquefied petroleum gas. The purpose of moisture content control is to provide assurance that pressure reducing regulators and similar equipment will operate consistently without troublesome freeze-ups caused by the separation of dissolved water from the product.

(*e*) *Free Water Content* is of importance only on the commercial PB-mixtures and commercial butane type products. These two types of products are normally used under ambient conditions which are mild and, as a consequence, the only requirement is vigilance to assure that no free water is present.

ASTM Logo Removed **D 1835**

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. ***HEED THIS NOTICE***: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM D1890: Standard Test Method for Beta Particle Radioactivity of Water

CFR Section(s): 40 CFR 136.3(a)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: D 1890 – 96

Standard Test Method for
Beta Particle Radioactivity of Water[1]

This standard is issued under the fixed designation D 1890; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the measurement of beta particle activity of water, as referenced to the beta energy of ^{137}Cs, not corrected for conversion electrons. It is applicable to beta emitters having maximum energies above 0.1 MeV and at activity levels above 0.02 Bq/mL of radioactive homogeneous water for most counting systems. This test method is not applicable to samples containing radionuclides that are volatile under conditions of the analysis.

1.2 This test method can be used for either absolute or relative determinations. In tracer work, the results may be expressed by comparison with a standard which is defined to be 100 %. For radioassay, data may be expressed in terms of a known radionuclide standard if the radionuclides of concern are known and no fractionation occurred during processing, or may be expressed arbitrarily in terms of some other standard such as cesium-137. General information on radioactivity and measurement of radiation may be found in the literature [2] and Practice D 3648.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[3]
D 1193 Specification for Reagent Water[3]
D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[3]
D 3370 Practices for Sampling Water from Closed Conduits[3]
D 3648 Practice for the Measurement of Radioactivity[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Becquerel*—a unit of radioactivity equivalent to 1 nuclear transformation per second.

3.1.2 *beta energy, maximum*—the maximum energy of the beta-particle energy spectrum produced during beta decay of a given radioactive species.

NOTE 1—Since a given beta-particle emitter may decay to several different quantum states of the product nucleus, more than one maximum energy may be listed for a given radioactive species.

3.1.3 *counter background—in the measurement of radioactivity*, the counting rate resulting from factors other than the radioactivity of the sample and reagents used.

NOTE 2—Counter background varies with the location, shielding of the detector, and the electronics; it includes cosmic rays, contaminating radioactivity and electrical noise.

3.1.4 *counter beta-particle efficiency—in the measurement of radioactivity*, that fraction of beta particles emitted by a source which is detected by the counter.

3.1.5 *counter efficiency—in the measurement of radioactivity*, that fraction of the disintegrations occurring in a source which is detected by the counter.

3.1.6 *radioactive homogeneous water*—water in which the radioactive material is uniformly dispersed throughout the volume of water sample and remains so until the measurement is completed or until the sample is evaporated or precipitating reagents are added to the sample.

3.1.7 *reagent background—in the measurement of radioactivity of water samples*, the counting rate observed when a sample is replaced by mock sample salts or by reagent chemicals used for chemical separations that contain no analyte.

NOTE 3—Reagent background varies with the reagent chemicals and analytical methods used and may vary with reagents from different

---

[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

Current edition approved Feb. 10, 1996. Published April 1996. Originally published as D 1890 – 61 T. Last previous edition D 1890 – 90.

[2] Friedlander, G., et al., *Nuclear and Radiochemistry*, 3rd Ed., John Wiley and Sons, Inc., New York, NY, 1981.

Price, W. J., *Nuclear Radiation Detection*, 2nd Ed., McGraw-Hill Book Co., Inc., New York, NY, 1964.

Lapp, R. E., and Andrews, H. L., *Nuclear Radiation Physics*, 4th Ed., Prentice-Hall Inc., New York, NY, 1972.

Overman, R. T., and Clark, H. M., *Radioisotope Techniques*, McGraw-Hill Book Co., Inc., New York, NY, 1960.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 11.02.

manufacturers and from different processing lots.

3.2 *Definitions*—For terms not defined in this test method or in Terminology D 1129, reference may be made to other published glossaries.[5]

4. Summary of Test Method

4.1 Beta radioactivity may be measured by one of several types of instruments composed of a detecting device and combined amplifier, power supply, and scaler—the most widely used being proportional or Geiger-Müller counters. Where a wide range of counting rates is encountered (0.1 to 1300 counts per seconds), the proportional-type counter is preferable due to a shorter resolving time and greater stability of the instrument. The test sample is reduced to the minimum weight of solid material having measurable beta activity by precipitation, ion exchange resin, or evaporation techniques. Beta particles entering the sensitive region of the detector produce ionization of the counting gas. The negative ion of the original ion pair is accelerated towards the anode, producing additional ionization of the counting gas and developing a voltage pulse at the anode. By use of suitable electronic apparatus, the pulse is amplified to a voltage sufficient for operation of the counter scaler. The number of pulses per unit of time is related to the disintegration rate of the test sample. The beta-particle efficiency of the system can be determined by use of prepared standards having the same radionuclide composition as the test specimen and equivalent residual plated solids. An arbitrary efficiency factor can be defined in terms of some other standard such as cesium-137.

5. Significance and Use

5.1 This test method was developed for the purpose of measuring the gross beta radioactivity in water. It is used for the analysis of both process and environmental water to determine gross beta activity.

6. Measurement Variables

6.1 The relatively high absorption of beta particles in the sample media and any material interposed between source and sensitive volume of the counter results in an interplay of many variables which affect the counting rate of the measurement. Thus, for reliable relative measurements, hold all variables constant while counting all test samples and standards. For absolute measurements, appropriate correction factors are applied. The effects of geometry, backscatter radiation, source diameter, self-scatter and self-absorption, absorption in air and detector window for external counters, and counting coincidence losses have been discussed[2] and may be described by the following relation:

$$cps = Bq_b\,(G_p)(f_{bs})(f_{aw})(f_d)(f_{ssa})(f_c) \tag{1}$$

where:

cps = recorded counts per second corrected for background,

Bq_b = disintegrations per second yielding beta particles,

G_p = point source geometry (defined by the solid angle subtended by the sensitive area of the detector),

f_{bs} = backscatter factor or ratio of cps with backing to cps without backing,

f_{aw} = factor to correct for losses due to absorption in the air and window of external detectors. It is equal to the ratio of the actual counting rate to that which would be obtained if there were no absorption by the air and window between the source and sensitive volume of the detector. Expressed in terms of absorption coefficient and density of absorber, $f_{aw} = e^{-\mu x}$, where μ = absorption coefficient, in square centimetres per milligram, and x = absorber density in milligrams per square centimetre.

f_d = factor to correct a spread source counting rate to the counting rate of the same activity as a point source on the same axis of the system,

f_{ssa} = factor to correct for the absorption and scatter of beta particles within the material accompanying the radioactive element, and

f_c = factor for coincident events to correct the counting rate for instrument resolving time losses and defined by the simplified equation, $f_c = 1 - nr$, where, n = the observed counts per second, and r = instrument resolving time in seconds. Generally, the sample size or source to detector distance is varied to obtain a counting rate that precludes coincident losses. Information on the effect of random disintegration and instrument resolving time on the sample count rate as well as methods for determining the resolving time of the counting system may be found in the literature.

For most applications, a detector system is calibrated using a single beta emitting radionuclide and an efficiency of detection, f_o, response curve generated for various sample residue weights. The efficiency of detection for each sample residual weight incorporates all the factors mentioned above so that:

$$f_o = cps/Bq = (G_p)(f_{bs})(f_{aw})(f_d)(f_{ssa})(f_c) \tag{2}$$

6.1.1 In tracer studies or tests requiring only relative measurements in which the data are expressed as being equivalent to a defined standard, the above correction factors can be simply combined into a counting efficiency factor. The use of a counting efficiency factor requires that sample mounting, density of mounting dish, weight of residue in milligrams per square centimetre, and radionuclide composition, in addition to conditions affecting the above described factors, remain constant throughout the duration of the test and that the comparative standard be prepared for counting in the same manner as the test samples. The data from comparative studies between independent laboratories, when not expressed in absolute units, are more meaningful when expressed as percentage relationships or as the equivalent of a defined standard. Expressing the data in either of these two ways minimizes the differences in counters and other equipment and in techniques used by the laboratories conducting the tests.

6.2 The limit of sensitivity for both Geiger-Muller and proportional counters is a function of the background counting rate. Massive shielding or anti-coincidence detectors and

---

[5] American National Standard Glossary of Terms in Nuclear Science and Technology (ANSI N1.1).

circuitry, or both, are generally used to reduce the background counting rate to increase the sensitivity.

7. Interferences

7.1 Material interposed between the test sample and the instrument detector, as well as increasing density in the sample containing the beta emitter, produces significant losses in sample counting rates. Liquid samples are evaporated to dryness in dishes that allow the sample to be counted directly by the detector. Since the absorption of beta particles in the sample solids increases with increasing density and varies inversely with the maximum beta energy, plated solids shall remain constant between related test samples and should duplicate the density of the solids of the plated standard.

7.2 Most beta radiation counters are sensitive to alpha, gamma, and X-ray radiations, with the degree of efficiency dependent upon the type of detector.[2] The effect of interfering radiations on the beta counting rate is more easily evaluated with external-type counters where appropriate absorbers can be used to evaluate the effects of interfering radiation.

8. Apparatus

8.1 *Beta Particle Counter*, consisting of the following components:

8.1.1 *Detector*—The end-window Geiger-Muller tube and the internal or external sample gas-flow proportional chambers are the two most prevalent commercially available detector types. The material used in the construction of the detector should be free from detectable radioactivity. When detectors contain windows, the manufacturer shall supply the window density expressed in milligrams per square centimetre. To establish freedom from undesirable characteristics, the manufacturer shall supply voltage plateau and background counting rate data. Voltage plateau data shall show the threshold voltage, slope, and length of plateau. Detectors requiring external positioning of the test sample are mounted on a tube support of low-density material (aluminum or plastic) and positioned so the center of the window is directly above the center of the test sample. The distance between the detector window and test sample plays an important part in determining the geometry of the system and can be varied for external counters to correspond more favorably with such factors as activity level, source size, sensitivity requirements, energy of beta particles, etc. A convenient arrangement is to combine the tube mount with a sample holder containing slots for positioning the sample at three or four distances from the detector window, varying from approximately 5 to 100 mm from tube flange.

8.1.2 *Detector Shield*—The detector assembly is surrounded by an external radiation shield of massive metal equivalent to approximately 51 mm of lead and lined with 3.2-mm thick aluminum. The material of construction should be free from detectable radioactivity. The shield has a door or port for inserting or removing specimens. Detectors having other than completely opaque windows are light sensitive. The design of the shield and its openings shall eliminate direct light paths to the detector window; beveling of door and opening is generally satisfactory. The percentage of the beta particles scattered from the walls of the shield into the detector can be reduced by increasing the internal diameter of the shield. The use of a detector without a shield will significantly increase the background and the detection capability.

8.1.3 *Scaler*—Normally the scaler, mechanical register, power supply, and amplifier are contained in a single chassis, generally termed the scaler. The power supply and amplifier sections are matched by the manufacturer with the type of detector to produce satisfactory operating characteristics and to provide sufficient range in adjustments to maintain controlled conditions. The manufacturer shall provide resolving time information for the counting system. The scaler shall have capacity for storing and visually displaying at least 10^6 counts and with a resolving time no greater than 250µ s for use with Geiger Muller detectors or 5 µs for use with proportional detectors. The instrument shall have an adjustable input sensitivity matched and set by the manufacturer to that of the detector, and a variable high-voltage power supply with indicating meter.

8.2 *Sample Mounting*—Sample mounting shall utilize dishes having a flat bottom of a diameter no greater than that of the detector window preferably having 3.2-mm high side walls with the angle between dish bottom and side equal to or greater than 120° to reduce side-wall scattering (Note 4). Dishes shall be of a material that will not corrode under the plating conditions and should be of uniform surface density preferably great enough to reach backscatter saturation.[2]

NOTE 4—Sample dishes with vertical side walls may be used but the exact positioning of these dishes relative to the detector is very important. This factor becomes critical for dishes having the same diameter as the detector. Dishes having side walls more than 3.2 mm in height are not recommended. Stainless steel has been found to be satisfactory for this purpose.

8.3 *Alpha Particle Absorber*—Aluminum or plastic, having a uniform density such that total absorbing medium (air plus window plus absorber) between sample and sensitive volume of detector is approximately equal to 7 mg/cm^2 of aluminum. The absorber diameter shall be equal to or greater than the detector window and should be placed against the window to minimize scattering of the beta particles by the absorber. This absorber is not used when counting beta particles with maximum energies below 0.35 MeV due to the high-count rate loss by absorption (about 48 % at 0.35 MeV in 7 mg/cm^2 of aluminum). The alpha particle absorber is not recommended for use with internal beta particle detectors, especially when either the composition or activity ratios of the radionuclides or radioactivity level might vary significantly between samples. Chemical separation of the alpha and beta particle emitters produces a higher degree of accuracy for internal detector measurements. Use published information[2] on beta particle absorption as a guide.

9. Reagents

9.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without increasing the background of the measurement.

NOTE 5—Some chemicals, even of high purity, contain naturally occurring radioactive elements, for example, rare earths and potassium compounds. Also, some chemical reagents, including organic compounds, have been found to be contaminated with artificially produced radionuclides. Consequently, when carrier chemicals are used in the analysis of low-radioactivity samples, the radioactivity of the carriers shall be determined under identical analytical conditions as used for the sample, including amounts of residual solids in the dish. The radioactivity of the reagents may be considered as background and subtracted from the test sample counting rate. This increased background reduces the sensitivity of the measurement.

9.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193, Type III.

9.3 *Cesium-137 Solution*—A ^{137}Cs solution containing approximately 200 Bq/mL with a calibration that is traceable to National Institute of Standards and Technology (NIST).

9.4 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

10. Sampling

10.1 Collect the sample in accordance with Practices D 3370.

10.2 Preserve the sample in a radioactively homogeneous state.

NOTE 6—A sample may be made homogeneous by addition of a reagent in which the radionuclides or compounds of the radionuclides present in the sample would be soluble in large concentrations. Addition of acids, complexing agents, or chemically similar stable carriers may be used to obtain homogeneity. Consideration of the chemical nature of the radionuclides and compounds present and the subsequent chemistry of the method will indicate the action to be taken. The addition of chemicals (HCl) corrosive to the mounting dish shall be avoided to prevent increased absorption of beta particles by the increased residual solids.

11. Establishing Gas Proportional Counter Operating Plateau

11.1 Put the instrument into operation according to the manufacturer's instructions. Place the counter's quality control standard (Note 7) having an approximate disintegration rate of 170 Bq in the counting position closest to the detector and begin counting. Slowly increase the high voltage until the first counts are observed and record the“ threshold” voltage. Raise the voltage 20 to 25 V (or some other convenient unit) above threshold, stop counting, reset the scaler to zero, and determine the count rate. Advance the voltage in small equal increments of 20 or 25 V, determining the count rate at each voltage. The count rate should rise initially, reach an approximately constant value (plateau), and then increase rapidly at the end of the plateau. The operating time at voltages above the plateau shall be minimized to avoid extensive arcing of the detector. If the plateau is 150 V in length, additional measurements are not necessary. Some newer computer controlled gas proportional counters (commercially available) have software that automatically measures the detector plateau through the use of an algorithm that controls the high voltage and scaler units of the system.

NOTE 7—The counter's quality control standard may be any available radionuclide having a high percentage of beta particle emission, a half-life sufficiently long to minimize decay corrections, and a maximum beta particle energy above 0.5 MeV. Knowledge of its true beta disintegration rate is not essential. The radionuclide shall be fixed permanently to the dish and distributed uniformly over an area preferably smaller than the dish bottom; electrodeposition and flaming of a salt-free solution are the two methods most generally used. The standard may be covered by thin aluminum or plastic of sufficient thickness to exclude any alpha particles originating from the source and to protect against damage. The dish shall be securely mounted for reproducible positioning. Any loss of activity in the control standard, other than by natural decay, requires establishment of a new control chart (see Section 12). For external counters, the ratio of control standard source diameter to detector window diameter should not exceed 0.33 to avoid the effect of a spread source on the counting geometry.

11.2 Plot the counting rate of the control standard against the indicated voltage. The voltage setting that corresponds to a value approximately 75 V above the "knee" of the curve shall be used as the operating voltage, provided this voltage is 50 V below the highest voltage on the plateau; otherwise the operating voltage shall be that at approximately the mid-point of the plateau (Note 8). A plateau slope of less than 3 %/100 V is desirable, but slopes between 3 and 6 %/100 V can be tolerated if a stable power supply is used. Refer to Practice D 3648 for additional details relative to operating voltage plateaus for gas proportional counters. Check the voltage plateau and operating voltage of the instrument on a regular schedule determined by experience and after any repair or major adjustment of the instrument. Shortening of the plateau length or an increase in slope are indications of a deteriorating detector.

NOTE 8—The counting life of the detector may be shortened by operation at a voltage higher than required for reliable performance. Consequently, the lowest voltage that meets the above conditions and will provide reproducible data should be chosen as the operating voltage.

12. Control of Instrument Operation

12.1 Tolerance or statistical control charts are used to assure that the instrument is operating to within pre-specified limits of the initial calibration. Repetitive measurements of a quality control source are taken to develop the tolerance or statistical control chart. The QC source is then used on a daily or prior to use basis to ensure proper operation. Refer to Practice D 3648 for the preparation of a tolerance or statistical control chart.

13. Calibration and Standardization for General Measurements

13.1 Place a known amount of cesium-137 standard (approximately 200 Bq) into a volume of water having a dissolved salts content equivalent to those of the test samples and prepare for counting as directed in Section 15. Throughout the experiment, the evaporation, mounting, counting, and density of plate

---

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

solids of this reference standard shall be identical with those of the test samples. Count for a length of time required to produce the desired statistical precision (typically 1 %; refer to Practice D 3648). The combined efficiency factor, f_o, for each dissolved salt weight is then expressed as a fraction of the disintegration rate, Bq, of the reference standard according to Eq. 2.

14. Calibration and Standardization for Tracer Experiments

14.1 Add a known quantity of activity of a reference solution of the tracer (approximately 200 Bq) to a radioactivity-free standard test sample and process in accordance with Section 15.

15. Procedure

15.1 Place an appropriate volume of the test specimen in a glass beaker, make 0.5 *M* with HNO_3, and evaporate to 1 to 2 mL. Quantitatively transfer to the mounting dish and evaporate to dryness. Adjust the heat carefully to prevent spattering or boiling. A ring heater having a continuously variable voltage control, or adjustable infrared heat lamps, is the preferable heat source for the final evaporation and drying. Uniform spreading of the residual salts is necessary for reliable comparative data. The salts shall be thoroughly mixed to assure uniform and homogeneous distribution of the radioactive nuclides in the deposit. Inhomogeneity may result in poor reproducibility. Cool hygroscopic solids in a dry atmosphere and store in a desiccator until the start of counting. Place the sample in the counter and count for a time interval sufficient to obtain the desired statistical uncertainty. Record the scaler reading. Transfer of large volume samples to smaller beakers as evaporation nears completion makes for easier transfer of the test specimen to the mounting dish. All transfers shall be made with reagent water. The sample size shall be chosen with consideration for the absorption of beta particles in the residual solids. Information[2] on the range energy relationship of beta particles in aluminum should be used as a guide to obtain the desired results.

15.2 Precipitation methods may be used to expediently concentrate the radioactive material into small amounts of precipitate. The precipitate is separated and washed free of precipitant by centrifugation or filtration. Choose the method of separation that will produce a uniform deposit of precipitate after quantitatively transferring to the mounting dish or filter paper for counting. Calibrate the instrument under counting conditions identical to those used for the samples. More detailed information on the techniques and equipment for separation and mounting of the precipitate may be found in the literature. [2]

16. Calculation

16.1 Results may be expressed in observed counts per second per millilitre or Bq/mL. This test method is useful for comparing activities of a group of samples, as in tracer experiments. Results may also be reported in terms of equivalent cesium-137 activity or other standard radionuclide activity, using the empirical efficiency determined by use of a reference standard. If it is known that only one nuclide is present, its disintegration rate may be determined by use of the efficiency factor determined from a reference standard of that nuclide obtained from the National Institute of Standards and Technology (NIST) or from a NIST-traceable standard. Calculate the results as follows:

$$beta\ concentration\ (Bq / mL) = C_{net} / (f_o \times V) \tag{3}$$

where:

C_{net} = net count rate (s^{-1}),
V = test specimen, mL, and
f_o = detector efficiency factor.

The total propagated uncertainty of the beta concentration is calculated as:

$$\sigma_{Bq/mL} = Bq / mL \times [(\sigma_{C_{net}} / _{C_{net}})^2 + (\sigma_{fo} / f_o)^2 + (\sigma_V / V)^2]^{1/2} \tag{4}$$

where:

$\sigma_{C_{net}} / C_{net}$ = relative counting uncertainty,
σ_{fo}/f_o = relative detector efficiency uncertainty, and
σ_V/V = relative uncertainty in the sample volume measurement.

The net count rate and counting uncertainty, $\sigma_{C_{net}}$, are defined as:

$$C_{net} = CR_S - CR_B = C_s / t_s - C_B / t_B \tag{5}$$

$$^{\sigma}{}_{C_{net}} = (C_s / t_s^2 + C_B / t_B^2)2½ \tag{6}$$

where:

CR_s = sample count rate (s^{-1}),
CR_B = background count rate (s^{-1}),
C_s = sample counts,
C_B = background counts,
t_s = counting time of sample(s), and
t_B = counting time of background(s).

The *a priori* minimum detectable concentration (*MDC*) is calculated using the equation:

$$MDC = 2.71/(t_s \times k) + 4.65 \times \sigma_B / k \tag{7}$$

where:

σ_B = $(CR_B/t_s)^{½}$, and
k = $f_o \times V$.

A more detailed discussion on the minimum detectable concentration concept can be found in Practice D 3648.

17. Precision and Bias

17.1 The overall precision and bias of this test method within its designated range varies with the quantity being tested according to Table 1.

17.2 This collaborative test for the determination of gross beta activity in water was conducted by six laboratories at three concentration levels ranging from 4.6 to 46.5 Bq/mL and containing 8 mg, 19.5 mg, and 40 mg of solids, respectively. Each laboratory processed three replicates per level.

TABLE 1 Determination of Precision and Bias

Amount Added, Bq/mL	Average Calculated Amount, Bq/mL	± Bias	± % Bias	Statistically Significant (5 % C.I.)	Precision	
					S_t	S_o
4.60 ± 0.12	4.6 ± 1.0	s0.0	0.0	No	0.997	0.288
18.55 ± 0.50	20.0 ± 1.0	+ 1.5	+ 7.8	Yes	1.04	0.617
46.5 ± 1.2	50.6 ± 2.6	+ 4.1	+ 8.8	Yes	2.63	1.50

17.3 The precision and bias statements for this test method were obtained using Practice D 2777 – 86.

17.4 These test data were obtained using select water matrices. For other matrices these data may not apply.

18. Quality Control

18.1 Before this test method is utilized for the analysis of samples, a counter quality control or tolerance chart shall be established to ensure that the counting system is operating within prescribed limits. The quality control or tolerance chart shall be established at the time the counting system is calibrated.

18.2 Prepare a quality control or tolerance chart as recommended in Practice D 3648. The counting system shall be checked by analyzing a QC source daily or prior to use. The result of the QC analysis shall be tabulated or plotted on the control or tolerance chart and evaluated according to Practice D 3648.

18.3 Evaluate the counting system's background periodically. The background data shall be maintained in a logbook or plotted on a trend chart.

18.4 Precision and bias can be assessed in the following manner: the precision of an individual measurement can be approximated by the total propagated uncertainty and bias can be assessed by the analysis of NIST traceable spiked samples with known quantities of radioactivity.

19. Keywords

19.1 gross beta radioactivity; gross radioactivity measurement; proportional counter; water

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

This Document Posted By
Public.Resource.Org, Inc.,
a California Nonprofit
Organization.

Not Affiliated Or
Authorized by ASTM
or by the United States
Government

Document Name: ASTM D1943: Standard Test Method for Alpha Particle Radioactivity of Water

CFR Section(s): 40 CFR 136.3(a)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

[redacted] ASTM Logo Removed

Designation: D 1943 – 96

Standard Test Method for Alpha Particle Radioactivity of Water[1]

This standard is issued under the fixed designation D 1943; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the measurement of alpha particle activity of water. It is applicable to alpha emitters having maximum energies above 3.9 MeV and at activity levels above 0.02 Bq/mL of radioactive homogeneous water. This test method is not applicable to samples containing alpha-emitting radionuclides that are volatile under conditions of the analysis.

1.2 This test method can be used for either absolute or relative determinations. In tracer work, the results may be expressed by comparison with a standard that is defined to be 100 %. For radioassay, data may be expressed in terms of alpha disintegration rates after calibration with a suitable standard. General information on radioactivity and measurement of radiation has been published[2] and summarized in Practice D 3648.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[3]

D 1193 Specification for Reagent Water[3]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[3]

D 3370 Practices for Sampling Water[3]

D 3648 Practice for the Measurement of Radioactivity[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129. For terms not defined in this test method or in Terminology D 1129, reference may be made to other published glossaries.[5]

4. Summary of Test Method

4.1 The test sample is reduced by evaporation or a suitable chemical method to the minimum weight of material having measurable alpha activity. Alpha radioactivity is measured by an instrument composed of a detecting device, amplifier, power supply, and scaler—the most widely used being proportional and scintillation counters. In the proportional counter, which may be of the windowless or thin window type, alpha particles entering the sensitive region of the detector produce ionization of the counting gas. The negative ion of the original ion pair is accelerated towards the anode, producing additional ionization of the counting gas and developing a voltage pulse at the anode. In the scintillation detector, alpha particles interact with the material of the phosphor, transferring some of their energy to electrons. These electrons subsequently lose part of their energy by excitation rather than ionization of atoms, and the excited atoms revert to the ground state by re-emitting energy in the form of light quanta. A suitable light-sensitive device, usually a multiplier phototube, transforms the resulting flashes of light into voltage impulses. By use of suitable electronic apparatus, the pulse is amplified to a voltage sufficient for operation of the counting scaler. The number of pulses per unit time is related to the disintegration rate of the test sample. The efficiency of the system can be determined by use of a suitable alpha standard having equivalent residual plated solids.

5. Significance and Use

5.1 This test method was developed for the purpose of measuring gross alpha radioactivity in water. It is used for the analysis of both process and environmental water to determine

---

[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

Current edition approved Feb. 10, 1996. Published April 1996. Last previous edition D 1943 – 90.

[2] Friedlander, G., et al., *Nuclear and Radiochemistry*, 3rd Ed., John Wiley and Sons, Inc., New York, NY, 1981.

Price, W. J., *Nuclear Radiation Detection*, 2nd Ed., McGraw-Hill Book Co., Inc., New York, NY, 1964.

Lapp, R. E., and Andrews, H. L., *Nuclear Radiation Physics*, 4th Ed., Prentice-Hall Inc., New York, NY, 1972.

Overman, R. T., and Clark, H. M., *Radioisotope Techniques*, McGraw-Hill Book Co., Inc., New York, NY, 1960.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 11.02.

[5] American National Standard Glossary of Terms in Nuclear Science and Technology (ANSI N1.1) available from the American National Standards Institute, 1430 Broadway, New York, NY 10018.

gross alpha activity which is often a result of natural radioactivity present in minerals.

6. Measurement Variables

6.1 The relatively high absorption of alpha particles in the sample media affects the counting rate of the measurement. Effects of geometry, back-scatter, source diameter, as well as the purity, pressure variation, and type of counting gas used shall also be considered. Thus, for reliable relative measurements, the variables shall be held constant while counting all test samples and standards. For absolute measurements, appropriate efficiency factors shall be applied. If a windowless proportional counter is employed, the sample mount shall be electrically conducting.

6.1.1 In tracer studies or tests requiring only relative measurements, in which the data are expressed as being equivalent to a defined standard, the above correction factors can be simply combined into a counting efficiency factor. The use of a counting efficiency factor requires that sample mounting, material of mounting dish, and weight of residue (milligrams per square centimetre), in addition to conditions affecting the above described factors, remain constant throughout the duration of the test and that the comparative standard be prepared for counting in the same manner as the test samples. The data from comparative studies between independent laboratories when not expressed in absolute units are more meaningful when expressed as percentage relationships or as equivalent of a defined standard.

6.2 The limit of sensitivity for both scintillation and proportional counters is a function of the background counting rate which should be as low as is feasible. Massive shielding is not used for alpha counters. The maximum activity for this test method is 1600 Bq.

7. Interferences

7.1 Solids content in the sample containing the alpha emitter produces significant losses in sample counting rates of about 10 to 15 % loss at 1 mg/cm^2. Liquid samples shall be evaporated to dryness onto dishes that allow the sample to be counted directly by the detector. Solids on the dish shall remain constant in amount between related test samples, and should duplicate the density of the solids of the plated standard.

7.2 Most alpha counters are insensitive to beta, gamma, and X radiations.[2]

8. Apparatus

8.1 *Alpha Particle Counter*, consisting of either a proportional detector or a scintillation detector, and a scaler conforming to the following requirements:

8.1.1 *Proportional Detector*—This may be one of several types commercially available. The material used in the construction of the detector should contain a minimal amount of detectable radioactivity. To establish freedom from undesirable characteristics, the manufacturer shall supply voltage plateau and background counting rate data. Voltage plateau data shall show the threshold voltage, slope, and length of plateau for a particular input sensitivity.

8.1.2 *Scintillation Detector*—This may be one of several types commercially available. It shall consist of an "activated" zinc sulfide phosphor having a minimum effective diameter of 36.5 mm and a superficial density of 10 to 15 mg/cm^2. The phosphor shall be mounted so that it can be attached and optically coupled to a multiplier phototube. Extraneous light shall be excluded from the phosphor either by its being covered with a thin (less than 1 mg/cm^2) opaque window or by enclosing the assembly in a lightproof sample changer. The material used in the construction of the detector shall be free from detectable radioactivity. To establish freedom from undesirable characteristics, the manufacturer shall supply voltage plateau and background counting rate data. Voltage plateau data shall show the threshold voltage, slope, and length of a plateau for a specified scaler sensitivity.

8.1.3 *Scaler*—Often the scaler, mechanical register, power supply, and amplifier are contained in a single chassis, generally termed the scaler. The power supply and amplifier sections shall be matched with the type of detector to produce satisfactory operating characteristics and to provide sufficient range in adjustments to maintain controlled conditions. The manufacturer shall provide resolving time information for the counting system. The scaler shall have capacity for storing and visually displaying at least 10^6 counts with a resolving time no greater than 5 μs. The instrument shall have an adjustable input sensitivity that can be matched to the detector and a variable high voltage power supply with indicating meter.

8.2 *Sample Mounting Dish*—Dishes having a flat bottom of a diameter slightly less than the inside diameter of the detector. Flat dishes are preferred, but dishes may be used that have 3.2-mm high side walls with the angle between dish bottom and side equal to or greater than 120°. Dishes shall be of a material that will not corrode under the plating conditions and shall be of uniform surface density; platinum and stainless steel have been used for this purpose.

9. Reagents

9.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity and free from radioactivity to preclude detrimental effects. Some chemicals, even of high purity, contain naturally occurring radioactive elements, for example, uranium, actinium, and thorium. Consequently, when carrier chemicals are used in the analysis of low-radioactivity samples, the radioactivity of the carriers shall be determined under identical analytical conditions of the sample including residual dish solids. The radioactivity of the reagents shall be considered as background and subtracted from the test sample counting rate.

9.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming

---

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

to Specification D 1193, Type HI.

9.3 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

9.4 *Nitric Acid* (1 + 30)—Mix 1 volume of concentrated HNO_3(sp gr 1.42) with 30 volumes of water.

9.5 *Alpha-Emitting Radioactive Standard Solution* (~200 Bq/mL), traceable to the National Institute of Standards and Technology (NIST).

10. Sampling

10.1 Collect the sample in accordance with Practices D 3370.

10.2 Preserve the sample in a radioactive homogeneous state. A sample shall be made radioactive homogeneous by addition of a reagent in which the radionuclides or compounds of the radionuclides present would be soluble in large concentrations. Addition of acids, complexing agents, or stable chemically similar carriers may be used to obtain homogeneity. Consideration of the chemical nature of the radionuclides and compounds present and the subsequent chemistry of the method shall indicate the action to be taken.

11. Establishing Gas Proportional Counter Operating Plateau

11.1 Put the instrument into operation according to the manufacturer's instructions. Place a quality control (QC) source in the detector, set the sensitivity control near its maximum and turn the "count" switch to "count" position. Slowly increase the high voltage until the first counts are observed and record the "threshold" voltage. Advance the voltage in increments of convenient magnitude (approximately 25 V) and determine the counting rate at four or more settings of the sensitivity control at each voltage setting. Measure the background counting rate at each of the settings using an empty sample mounting dish in place of the standard.

11.1.1 The QC source may be any available alpha-emitting radionuclide having a half life sufficiently long to eliminate decay corrections. Knowledge of its true disintegration rate is not essential. The radionuclide shall be permanently fixed to the dish and uniformly distributed over an area preferably smaller than the dish bottom; electro-deposition and flaming of a salt-flee solution are the two methods most generally used. Quality control sources are commercially available.

11.2 Plot the gross counting rate of the standard against the voltage. The counting rate should rise initially as the voltage is increased, then, for at least some of the settings of the sensitivity control, reach an approximate constant value, and finally rise again. The" plateau" of the curve should be at least 100 V in length and have a slope less than 2 %/100 V; however, shorter plateaus or one with greater slope shall be acceptable if a well regulated high voltage power supply is available.

11.3 Plot the ratio of the square of the net counting rate of the standard to the background counting rate against the voltage for each of the settings of the sensitivity control.

11.4 Determine the optimum conditions for operation of the instrument by selecting values for the high-voltage and sensitivity adjustments that correspond to some point lying on the plateau of the counting-rate-versus-voltage plot and near the maximum value of the ratio of the sample-squared-to-background counting rates.

12. Control of Instrument Operation

12.1 Tolerance or statistical control charts are used to assure that the instrument is operating to within pre-specified limits of the initial calibration. Repetitive measurements of a quality control source are taken to develop the tolerance or statistical control chart. The QC source is then used on a daily or prior-to-use basis to ensure proper operation. Refer to Practice D 3648 for the preparation of a tolerance or statistical control chart.

13. Calibration and Standardization for General Measurements

13.1 Place a known amount of a NIST-traceable alpha standard (approximately 200 Bq) into a volume of water sufficient to dissolve salts (or into a volume of water containing dissolved salts) equivalent to those of the test samples and prepare for counting as directed in Section 15. Throughout the experiment, the evaporation, mounting, counting, and density of plate solids of this reference standard shall be identical with those of the test samples. Count for a length of time required to produce the desired statistical reliability (typically 1 %). The efficiency factor for each dissolved salt weight, f_o, is then expressed as a fraction of the disintegration rate (Bq) of the reference standard and is calculated according to the following equation:

$$f_o = cps / Bq \quad (1)$$

where:

cps = the measured counts per second.

The alpha emitting standard should have approximately the same alpha particle energy as the nuclides of interest so that mass attenuation effects can be estimated appropriately.

13.1.1 Purified natural uranium, of which the specific activity is 0.25 Bq per microgram, has been found satisfactory for this purpose. Other alpha-emitter preparations of known disintegration rate, for example, ^{241}Am or ^{237}Np, may also be used. When available, all calibration solutions shall be NIST traceable.

14. Calibration and Standardization for Tracer Experiments

14.1 Add a known quantity of activity from a reference solution of the tracer (approximately 180 Bq) to a radioactivity-free standard test sample and process as directed in Section 15.

15. Procedure

15.1 Place an appropriate volume of the test solution in a glass beaker, add 3 mL of concentrated HNO_3(sp gr 1.42) for each 100 mL of solution, and evaporate to 1 to 2 mL. Quantitatively transfer to the mounting dish and evaporate to dryness. Adjust the heat carefully to prevent spattering or boiling. A ring heater having a continuously variable voltage control or adjustable infrared heat lamps are the preferable heat sources for the final evaporation and drying. Uniform spreading of the residual salts is necessary for reliable comparative data. After drying, heat the dish to dull redness for a few seconds using a burner. Cool hygroscopic solids in a dry atmosphere and store in a desiccator until the start of counting. Place the sample in the counter and count for a time interval sufficient to attain the desired statistical reliability. Record the reading of the register. Transfer of large volume samples to smaller beakers as evaporation nears completion makes for easier transfer of the test specimen to the mounting dish. Make all transfers with $HNO_3(1 + 30)$. Choose the sample size with consideration for the absorption of alpha particles in the residual solids. The size should be such that the density of the deposit on the plate shall not exceed 5 mg/cm^2.

15.2 Precipitation methods may be used expediently to concentrate the radioactive material into small amounts of precipitate. The precipitate is separated and washed free of precipitant by centrifugation or filtration. Choose the method of separation that will produce a uniform deposit of precipitate after quantitatively transferring to the mounting dish for counting. Calibrate the instrument under counting conditions identical to those used for the samples. More detailed information is published[2] on the techniques and equipment for separation and mounting of the precipitate.

16. Calculation

16.1 Results may be expressed in observed counts per second per millilitre or Bq/mL. This test method is useful for comparing activities of a group of samples, as in tracer experiments. Results may also be reported in terms of equivalent americium-241 activity or other standard radionuclide activity using the empirical efficiency determined by use of a reference standard. If it is known that only one nuclide is present, its disintegration rate may be determined by use of the efficiency factor determined from a reference standard of that nuclide obtained from the National Institute of Standards and Technology (NIST) or from a NIST-traceable standard. Calculate the results as follows:

$$alpha\ concentration\ (Bq/mL) = C_{net} / (f_o \times V) \tag{2}$$

where:

C_{net} = net count rate (s^{-1}),
V = test specimen, mL, and
f_o = detector efficiency factor.

The total propagated uncertainty of the alpha concentration is calculated as:

$$\sigma_{Bq/mL} = Bq/mL \times [(\sigma_{Cnet} / C_{net})^2 + (\sigma_{fo}/f_o)^2 + (\sigma_V / V)^2]^{1/2} \tag{3}$$

where:

$\sigma_{Cnet/Cnet}$ = relative counting uncertainty,
σ_{fo}/f_o = relative detector efficiency uncertainty, and
σ_V/V = relative uncertainty in the sample volume measurement.

The net count rate and counting uncertainty, σ_{Cnet}, are defined as:

$$C_{net} = CR_S - CR_B = C_S / t_S - C_B / t_B \tag{4}$$

$$\sigma_{C_{net}} = (C_S / t_S^2 + C_B / t_B^2)^{1/2} \tag{5}$$

where:

CR_s = sample count rate (s^{-1}),
CR_B = background count rate (s^{-1}),
C_s = sample counts,
C_B = background counts,
t_s = counting time of sample(s), and
t_B = counting time of background(s).

The *a priori* minimum detectable concentration (*MDC*) is calculated using the equation:

$$MDC = 2.71 / (t_s \times k) + 4.65 \times \sigma_B / k \tag{6}$$

where:

σ_B = $(CR_B / t_s)^{1/2}$, and
k = $f_o \times V$.

A more detailed discussion on the minimum detectable concentration concept can be found in Practice D 3648.

17. Precision and Bias

17.1 The overall precision and bias of this test method within its designated range varies with the quantity being tested according to Table 1.

17.2 This collaborative test for the determination of gross alpha activity in water was conducted by six laboratories at three concentration levels ranging from 1.03 to 4.17 Bq/mL and containing 8 mg, 19.5 mg, and 40 mg of solids, respectively. Each laboratory processed three replicates per level.

17.3 The precision and bias statements for this test method were obtained using Practice D 2777 – 86.

18. Quality Control

18.1 Before this test method is utilized for the analysis of samples, a counter quality control or tolerance chart shall be established to ensure that the counting system is operating within prescribed limits. The quality control or tolerance chart shall be established at the time the counting system is calibrated.

18.2 Prepare a quality control or tolerance chart as recommended in Practice D 3648. The counting system shall be checked by analyzing a QC source daily or prior to use. The result of the QC analysis shall be tabulated or plotted on the control or tolerance chart and evaluated according to Practice D 3648.

18.3 Evaluate the counting system background periodically. The background data shall be maintained in a logbook or plotted on a trend chart.

18.4 Precision and bias can be assessed in the following manner: the precision of an individual measurement can be approximated by the total propagated uncertainty and bias can be assessed by the analysis of NIST traceable spiked samples with known quantities of radioactivity.

19. Keywords

19.1 gross alpha radioactivity; gross radioactivity measurement; proportional counter; water

TABLE 1 Determination of Precision and Bias

Amount Added, Bq/mL	Average Calculated Amount, Bq/mL	± Bias	± % Bias	Statistically Significant (5 % C.I.)	Precision	
					S_t	S_o
1.03 ± 0.01	0.98 ± 0.20	−0.05	−4.9	No	0.195	0.058
2.08 ± 0.02	2.08 ± 0.46	+ 0.00	+ 0.0	No	0.458	0.137
4.17 ± 0.05	3.97 ± 0.83	−0.20	−4.8	No	0.828	0.347

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.