EXHIBIT 150
PART 6

CERTIFICATE

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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.

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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.

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[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.

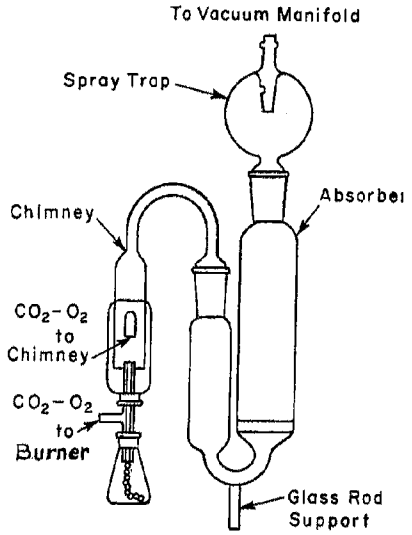


FIG. 1 Illustrative Sketch of the Assembled Lamp Unit

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.

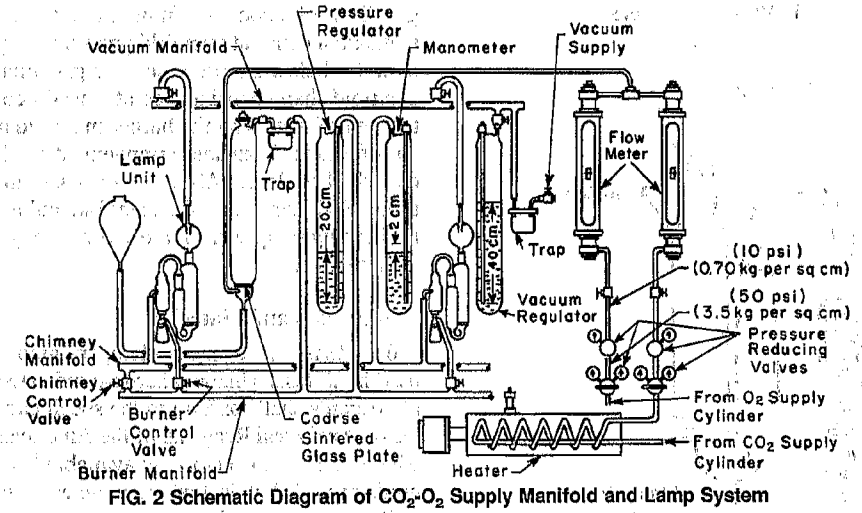


FIG. 2 Schematic Diagram of CO_2-O_2 Supply Manifold and Lamp System

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	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:

$$\text{Sulfur content, mass percent} = 16.03\ M \times (A/10\ W) \quad (1)$$

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:

$$\text{Corrected sulfur content, mass percent} = S - LF \quad (2)$$

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 + 0.025*S*

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

[13] *Annual Book of ASTM Standards*, Vol 05.03.

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.

[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:

$$\text{Sulfur content, mg/kg} = (A/WF) \times 1000 \quad \text{(A1.1)}$$

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

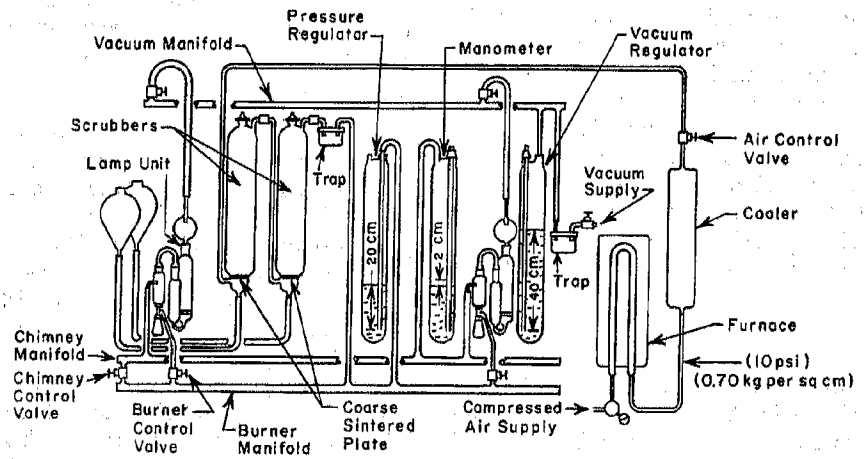


FIG. A2.1 Schematic Diagram of Purified Air Supply Manifold and Lamp System

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

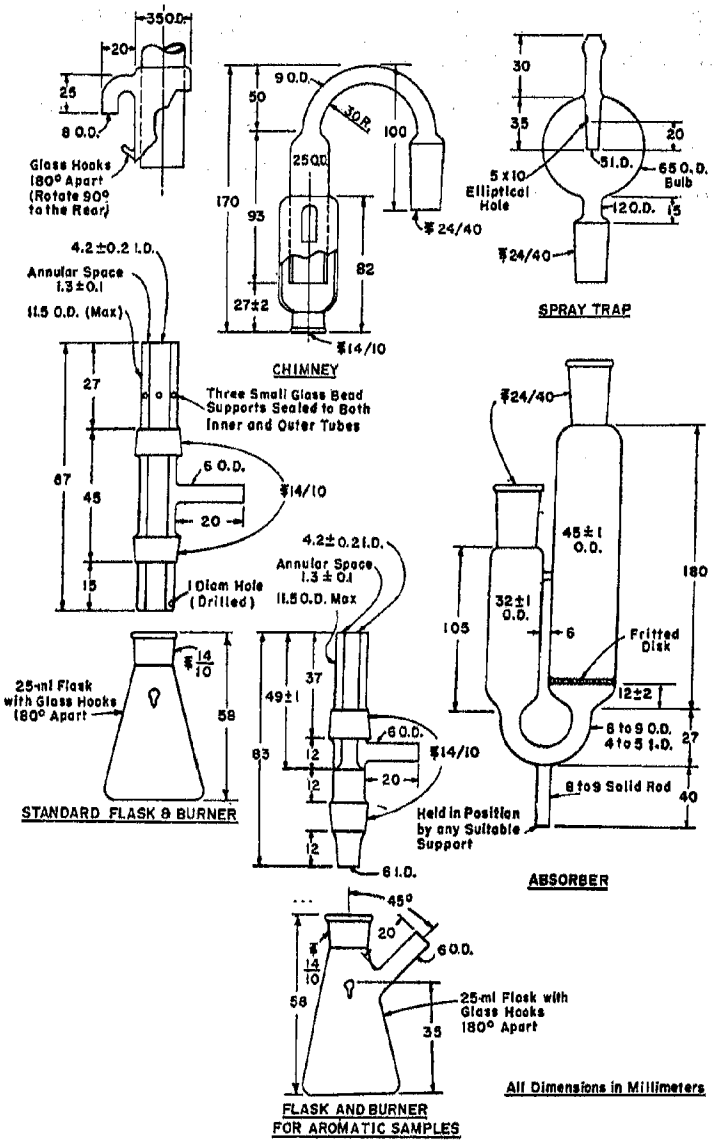
A3.4.1 An absorber of chemically resistant glass conforming to the dimensions shown in Fig. A3.1 and provided with standard-taper glass joints for connection with the chimney and spray trap, shall be used. A fritted disk with average pore diameter from 150 to 200 μm shall be sealed in the larger of two 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.

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.

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

APPROVED

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. ⅔, 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. ⅔can 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:

$$\text{Sulfur, weight percent} = (P - B)13.73/W \quad (1)$$

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.

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.

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WASHINGTON, D.C.

Designation: D 1298 – 99

An American National Standard

MPMS Chapter 9.1

IP 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.

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[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:

$$° \text{API} = 141.5/(\text{sp gr } 60/60°\text{F}) - 131.5 \quad (1)$$

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.

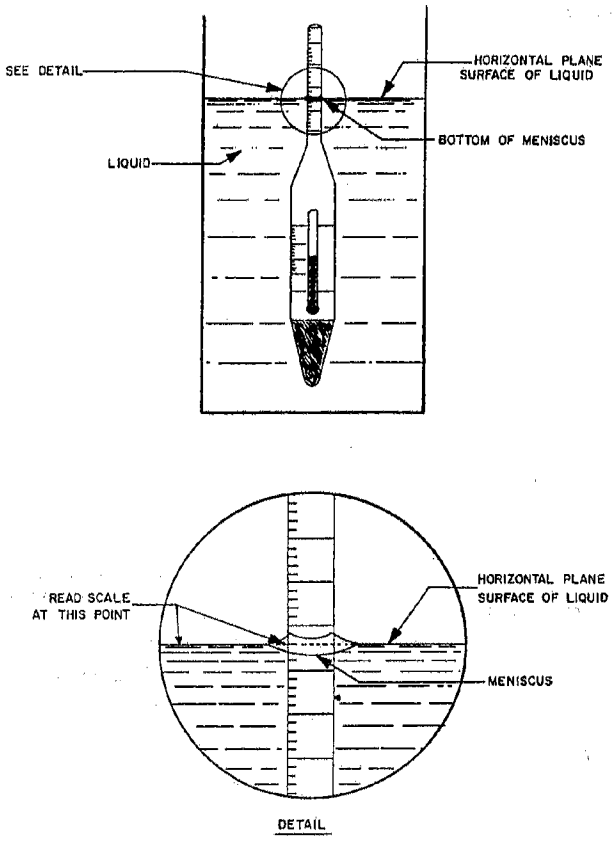

FIG. 1 Hydrometer Scale Reading for Transparent Liquids

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:

$$\rho r = \frac{\rho t}{1-[23\times10^{-6}\ (t-r)-2\times10^{-8}\ (t-r)^2]} \qquad (2)$$

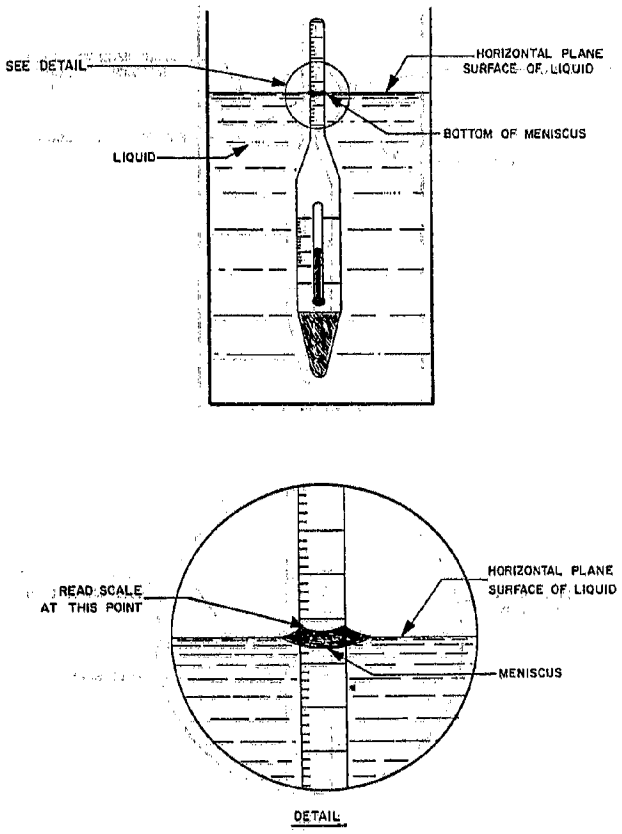

FIG. 2 Hydrometer Scale Reading for Opaque Fluids

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.

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.

TABLE 3 Example PMT Table Numbers

Material	Density at 15°C kg/m^3	Density at 20°C kg/m^3	Relative Density at 60/60°F	°API
Crude petroleum	53A	59A	23A	5A
Petroleum products	53B	59B	23B	5B
Lubricating oils	53D	59D	–	5D

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/m^3 kg/L or g/mL	0.5 0.0005	1.2 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/m^3 kg/L or g/mL	0.6 0.0006	1.5 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.

Document Name: ASTM D1335: Standard Test Method for Tuft Bind of Pile Floor Coverings

CFR Section(s): 24 CFR 200.945(a)(1)(ii)

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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]

This Standard is issued under the fixed designation D 1335; 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.

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

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[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 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.

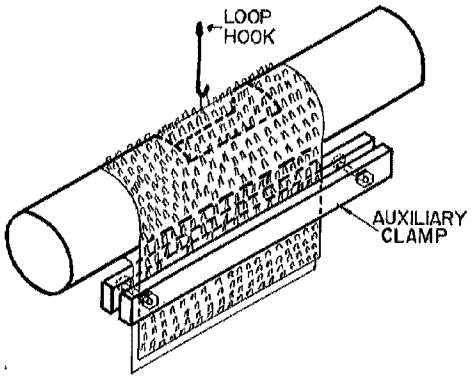

FIG. 1 Specimen Holder, Cut-Away Type.

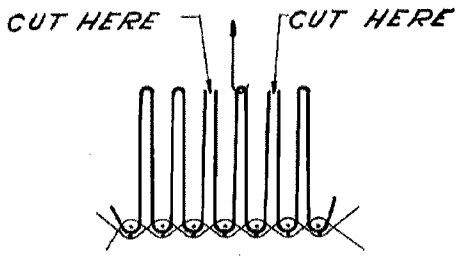

FIG. 2 Arrangement of Hook and Loops for Testing.

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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.

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[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.

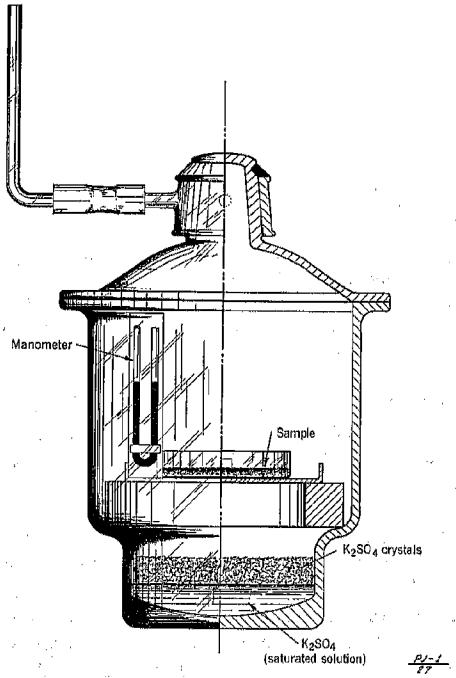

FIG. 1 Vacuum-Type Desiccator

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.

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WASHINGTON, D.C.

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.

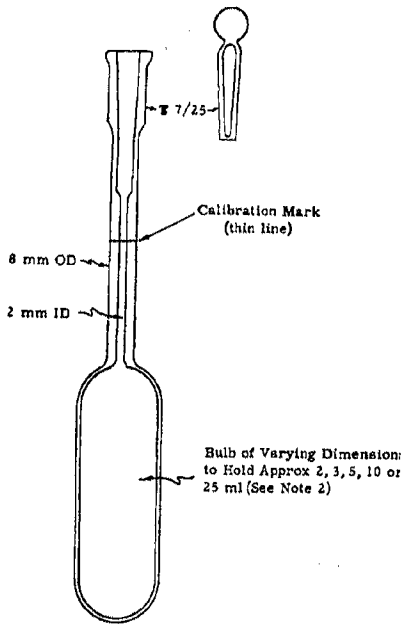


FIG. 1 Bingham-Type Pycnometer

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°.

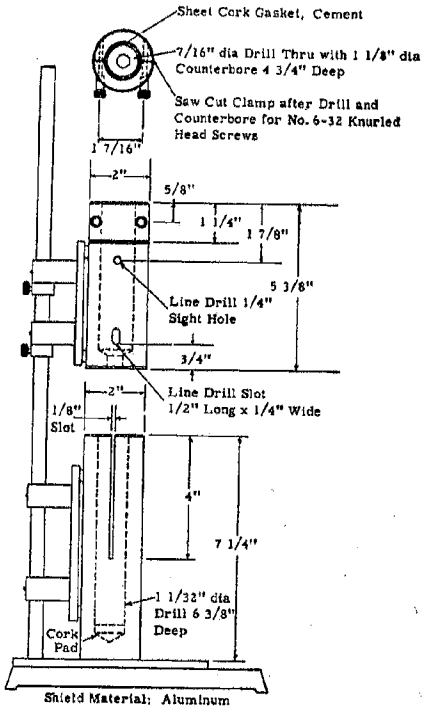
6.5 *Hypodermic Syringes*, 2 to 30-mL capacity, of chemically resistant glass, equipped with a 170-mm, 16-gage (0.065 in.) filling needle made from stainless-steel tubing, as shown in Fig. 3 .

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



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

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).

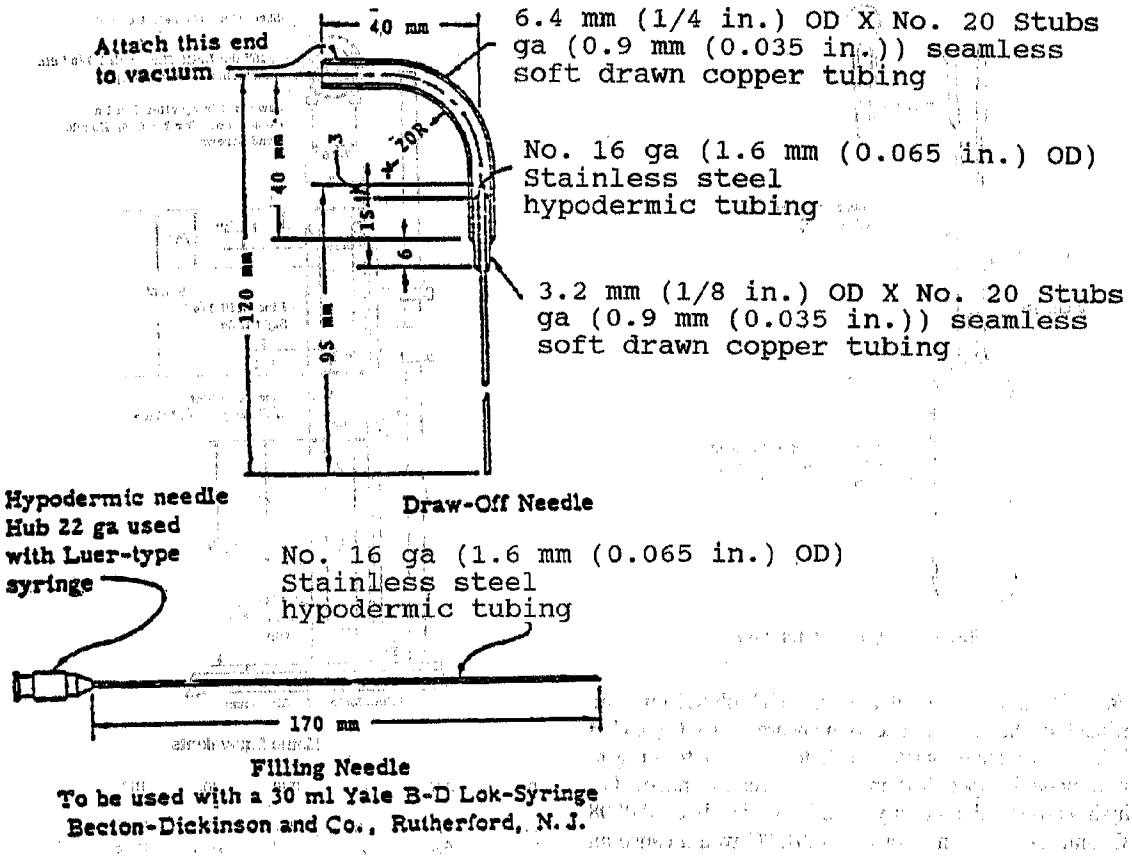


FIG. 3 Accessories for Bingham-Type Pycnometer

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

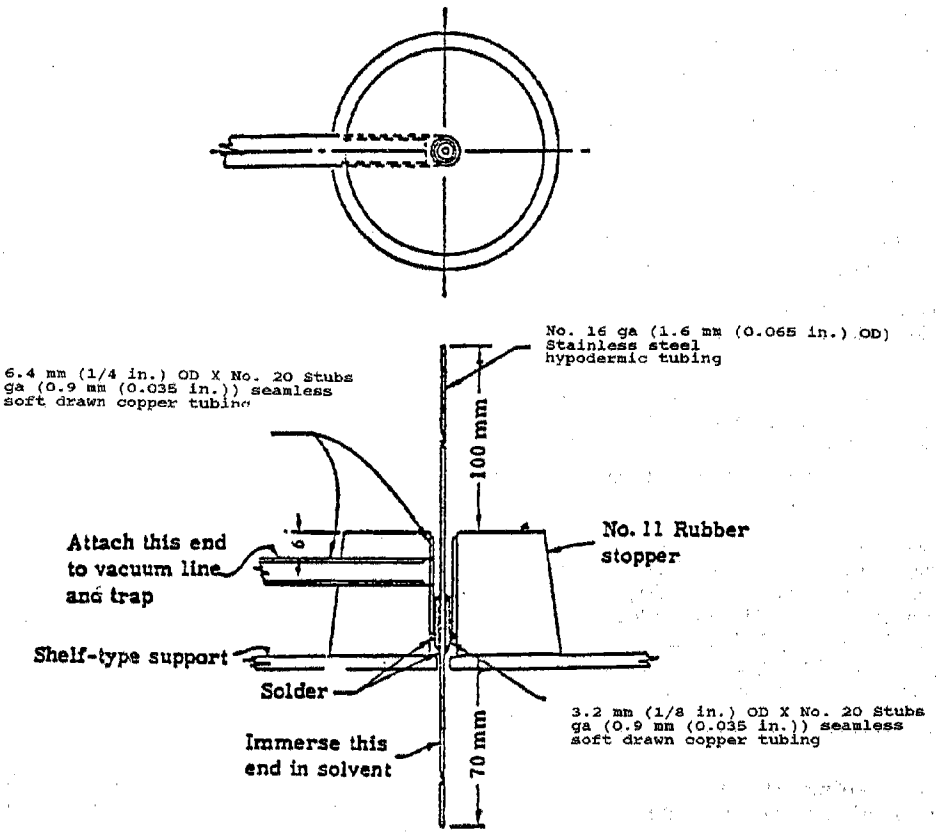


FIG. 4 Cleaner Assembly for Bingham-Type Pycnometer

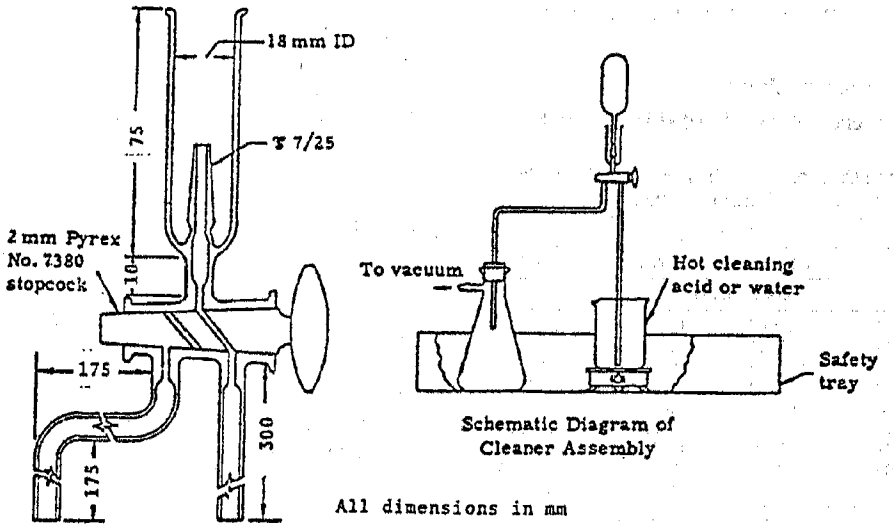


FIG. 5 All-Glass Pycnometer Cleaner Assembly for Use with Hot Chromic Acid Cleaning Solution

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:

$$F_t = (\text{density of water at } t°C)/(\text{weight of water in pycnometer at } t°C) \quad (1)$$

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:

$$\text{Density in vacuum } (d_t),\ \text{g/mL} = (F_t)(W_t) + C \quad (2)$$

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

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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.

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[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.

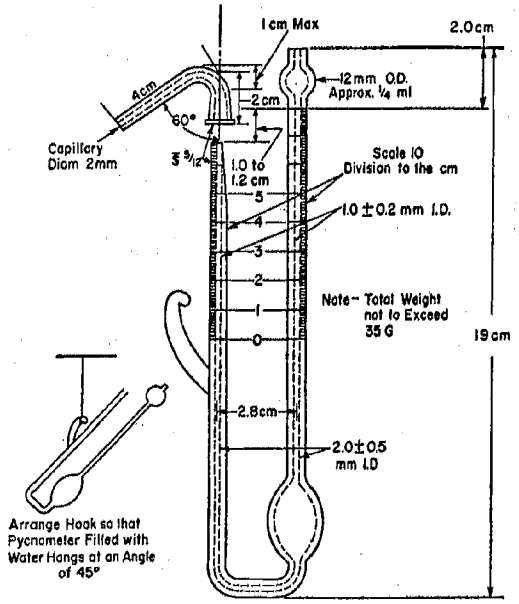


FIG. 1 Pycnometer

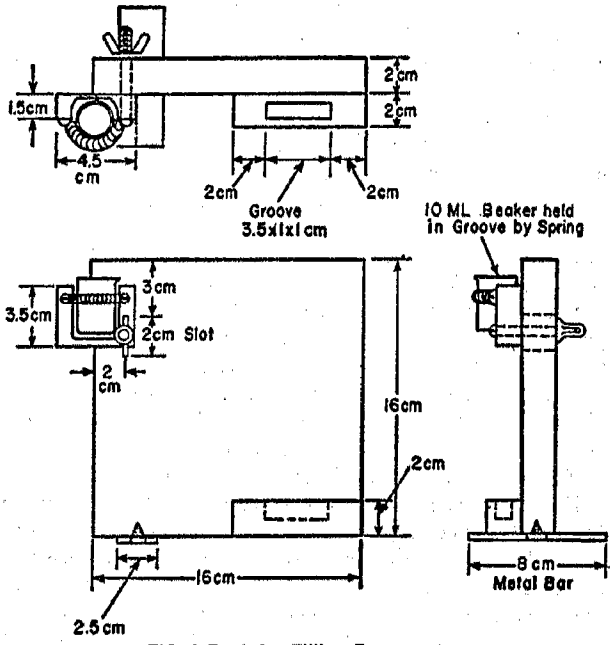


FIG. 2 Rack for Filling Pycnometer

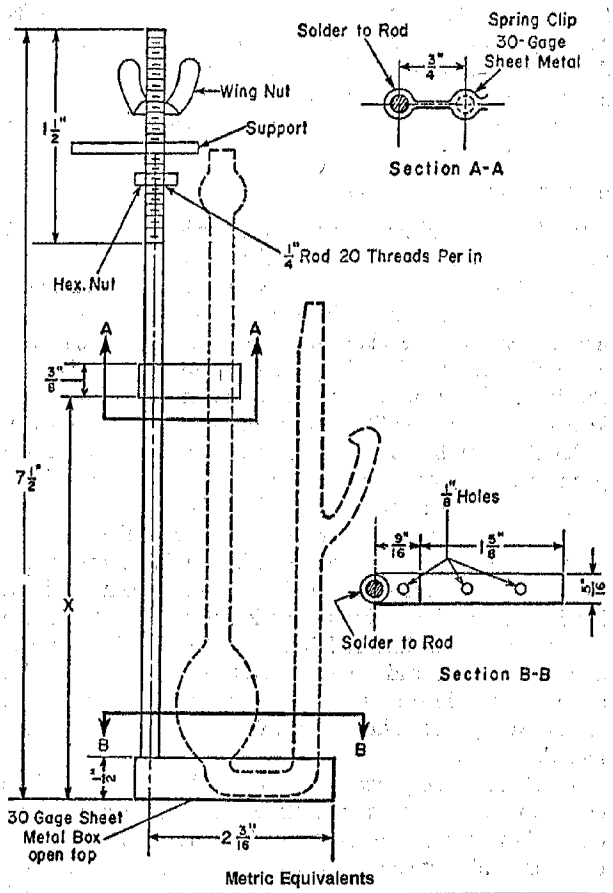
glycerin, or oil bath having a depth of at least 305 mm (12 in.) and provided with heating, stirring, and thermostating devices adequate to maintain desired temperatures in the range from 20 to 100°C with an accuracy of ±0.01°C.

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	⅝	14.3	2³⁄₁₆	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:

$$V_2 = V_1(1 + ct) \quad (1)$$

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

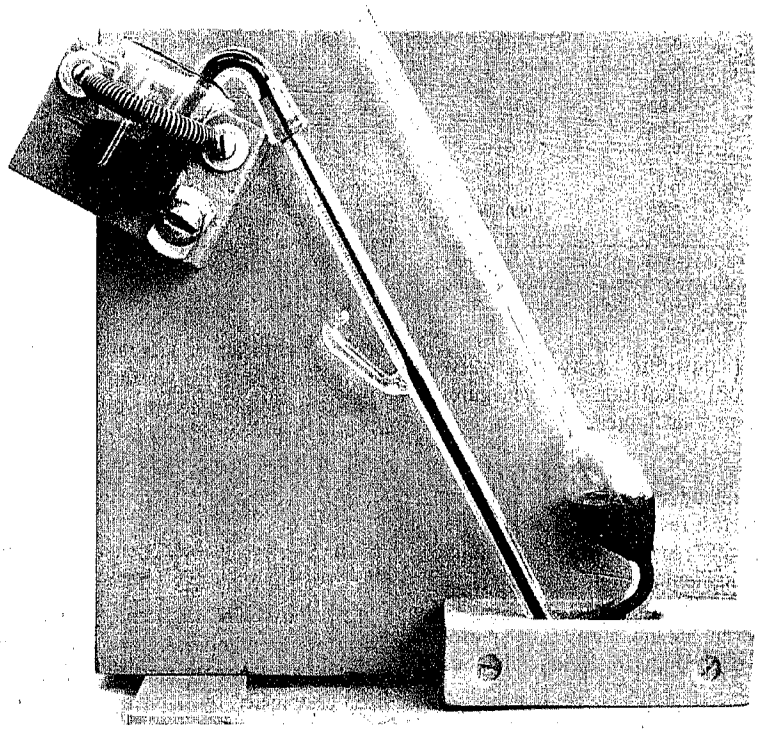


FIG. 4 Pycnometer Filling Assembly

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:

$$\text{Density in vacuum, } d_t, \text{ g/mL} = (W/V) + C \quad (2)$$

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

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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.

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[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/m²·K	W/cm·K	cal/s·cm·K	kg-cal/h·m·K	kg-cal·cm/h·m²·K	Btu/h·ft·°F	Btu·in/h·ft²·°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/m²·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·m²·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.	$1.2 \times 10^{+1}$	1.055×10^{-1}	$2.679 \times 10^{+2}$
Btu·in/h·ft²·°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}	$1.411 \times 10^{+1}$	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·m²/W.
[B] Recommended (SI) units.

TABLE 2 Conversion Factors for Thermal Transmittance[A]

To Convert Thermal Transmittance	Multiply by					
From to	W/m²·K[B]	W/cm²·K	cal/s·cm²·K	kg-cal/h·m²·K	Btu/h·ft²·°F	clo⁻¹
W/m²·K	1.	$1. \times 10^{-4}$	2.388×10^{-5}	8.598×10^{-1}	1.761×10^{-1}	1.548×10^{-1}
W/cm²·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·cm²·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·m²·K	1.163	1.163×10^{-4}	2.778×10^{-5}	1.	2.048×10^{-1}	1.8×10^{-1}
Btu/h·ft²·°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·m²/W.
[B] Recommended (SI) units.

between the hot plate and the cool atmosphere (W/m²·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/m²·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]	Multiply by									
From to	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}	8.598×10^{-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	Multiply by					
From to	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

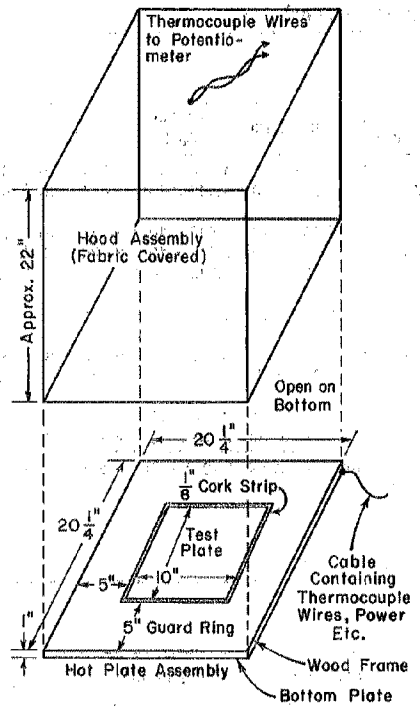

FIG. 1 Guard Ring Hot Plate For Thermal Transmittance Test

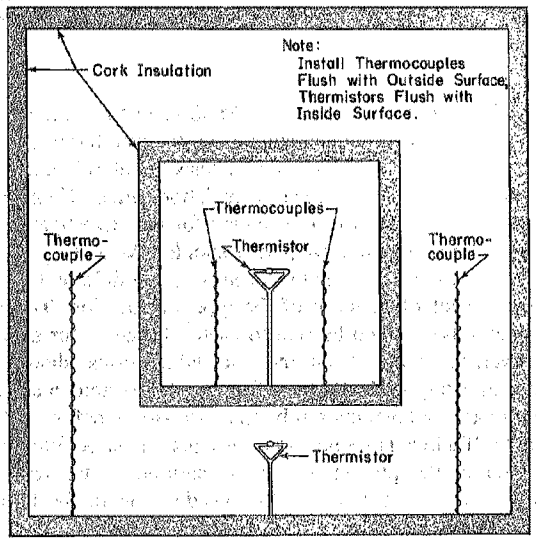

FIG. 2 Hot Plate, Top View, Showing Location of Thermistors and Thermocouples on Test Section and Guard Ring

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.

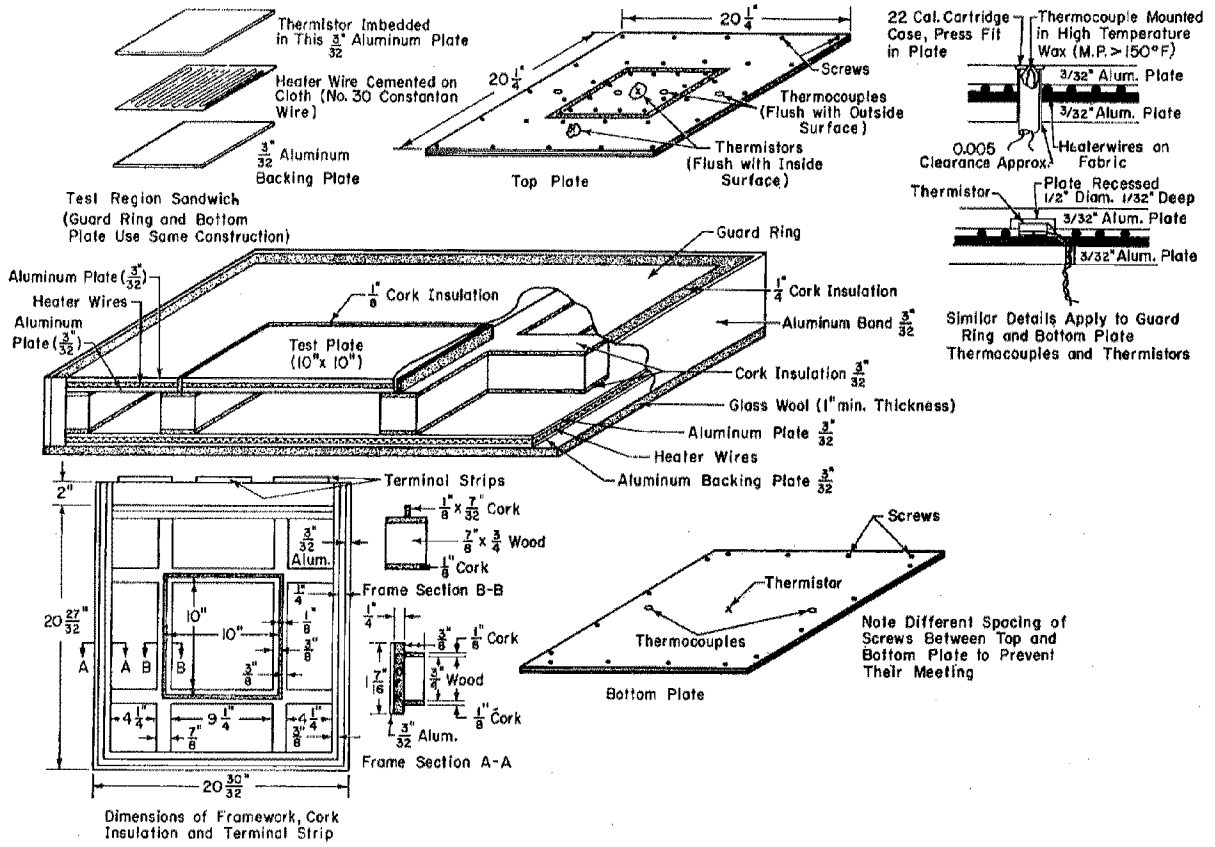

FIG. 3 Apparatus for Measurement of Thermal Transmittance, Showing Dimensions

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/m^2·K, using Eq 1:

$$U_1 = P/[A \times (T_p - T_a)] \quad (1)$$

where:

P = power loss from test plate, W,
A = area of test plate, m^2,
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:

$$1/U_2 = (1/U_1) - (1/U_{bp}) \quad (2)$$

or

$$U_2 = (U_{bp} \times U_1)/(U_{bp} - U_1) \quad (3)$$

10.4 Calculate the intrinsic thermal conductivity of the fabric alone, k, using Eq 4:

$$k = U_2 \times t_i/1000 \quad (4)$$

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:

$$R = 1/U_2 \quad (5)$$

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:

$$R' = 1/k \quad (6)$$

10.7 Calculate the intrinsic thermal resistance in Clo units using equation (7):

$$\text{Intrinsic Clo} = 1.137/U \quad (7)$$

10.8 Calculate the specific thermal resistance in Clo units using equation (8):

$$\text{Specific Clo} = 1.137/k \quad (8)$$

10.9 Calculate the bulk density, B, of the fabric, using Eq 9:

$$B = M/t \quad (9)$$

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:

$$U_{20} = U_{2t} \times (t_t/t_0) \quad (10)$$

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:

$$T_m = (T_a + T_p)/2 \quad (11)$$

where:
T_a = atmosphere temperature, and
T_p = plate surface temperature.

10.12 Calculate the effective insulation ratio, I_r, using Eq 12:

$$I_r = U_{bp}/U_1 \quad (12)$$

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

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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.

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APPROVED

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

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.

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[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: $1.5 \times 0.28 = 0.42$, 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]

$$\text{For } Y \le 0.9\text{: } V = UY$$

$$\text{For } Y \ge 0.9\text{: } V = \{AY^{1/3}\} - B - \{C/[(DY - E)^2 + F]\} + \{G/(Y^H)\} + \{J\sin(KY^{1/3} + L)\} + \{(M/Y)\sin[N(Y - 2)]\} - \{[P/(QY)]\sin[S(Y - T)]\}$$

where:

$A = 2.49268$	$G = 0.0133$	$P = 0.0037$
$B = 1.5614$	$H = 2.3$	$Q = 0.44$
$C = 0.985$	$J = 0.0084$	$S = 1.28$
$D = 0.1073$	$K = 4.1$	$T = 0.53$
$E = 3.084$	$M = 0.0221$	$U = 0.87445$
$F = 7.54$	$N = 0.39$	$W = 0.9967$

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	9.59	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

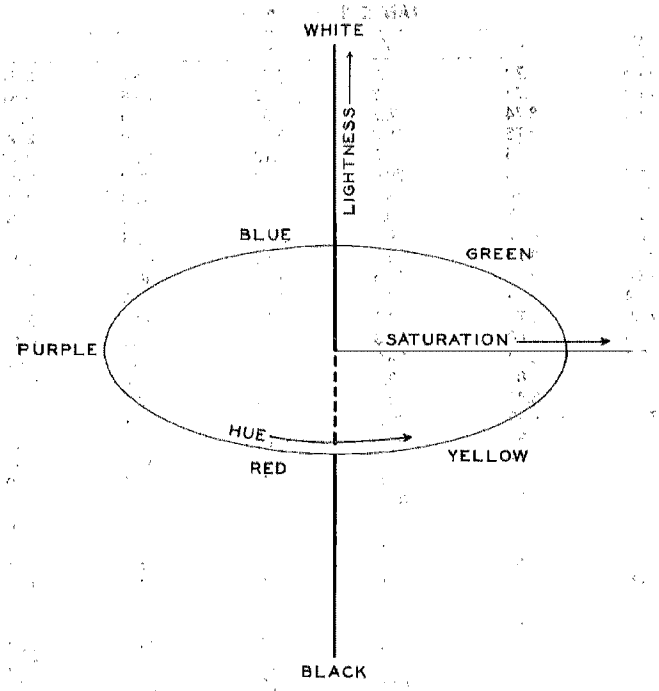

FIG. 1 Dimensions of the Surface-Color-Perception Solid

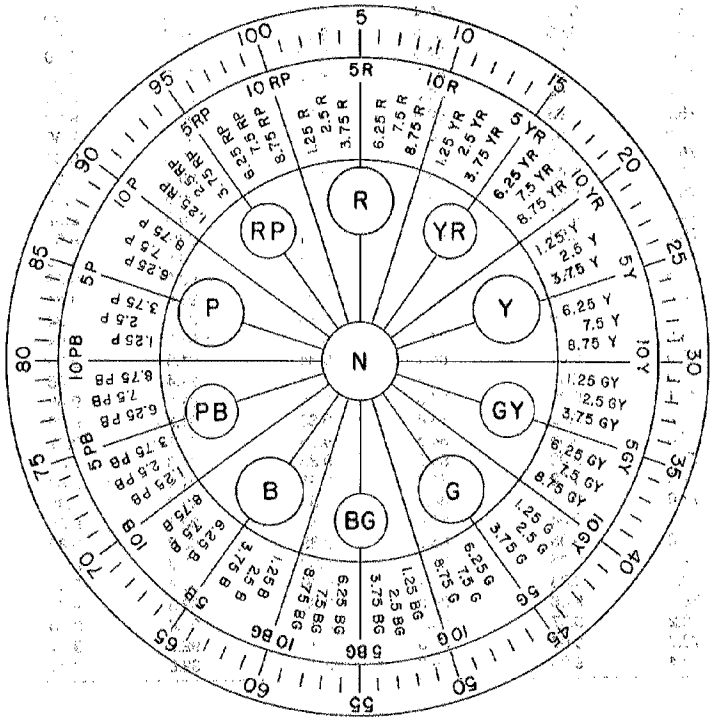

FIG. 2 Designation Systems for Munsell Hue

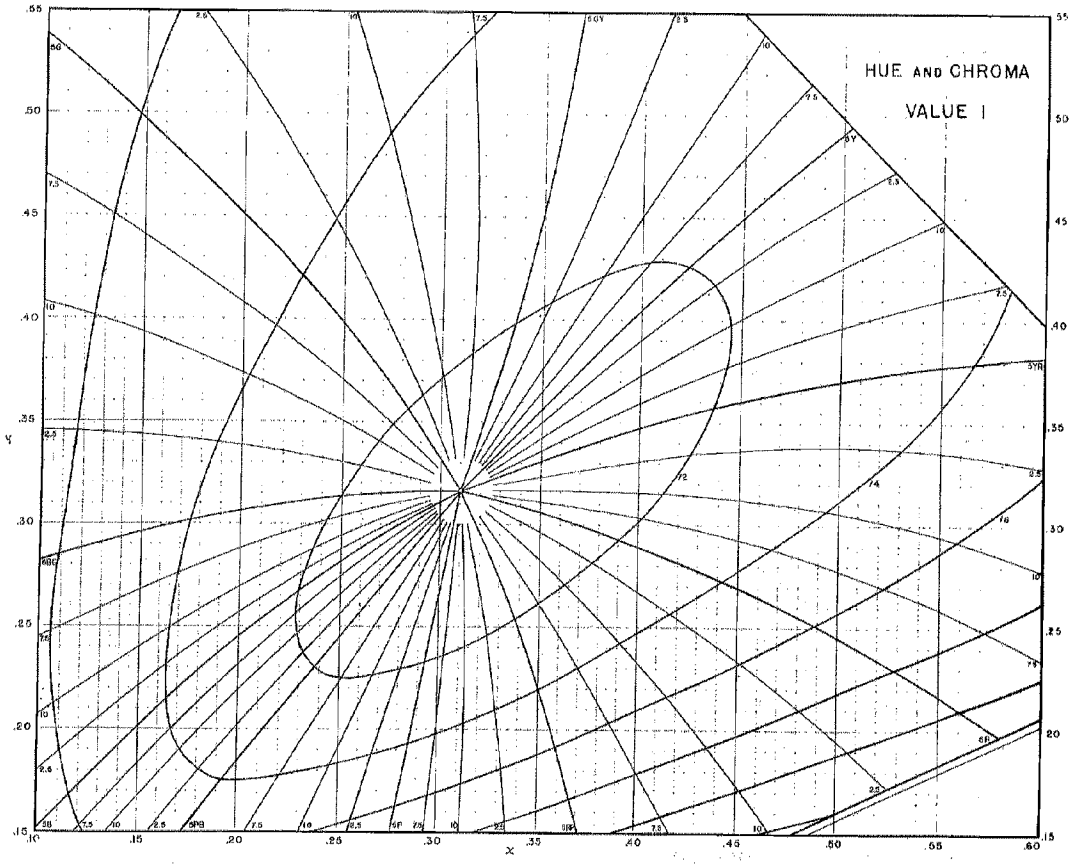

FIG. 3 Munsell Value 1—Loci of Constant Hue and Constant Chroma in CIE (x,y) Coordinates

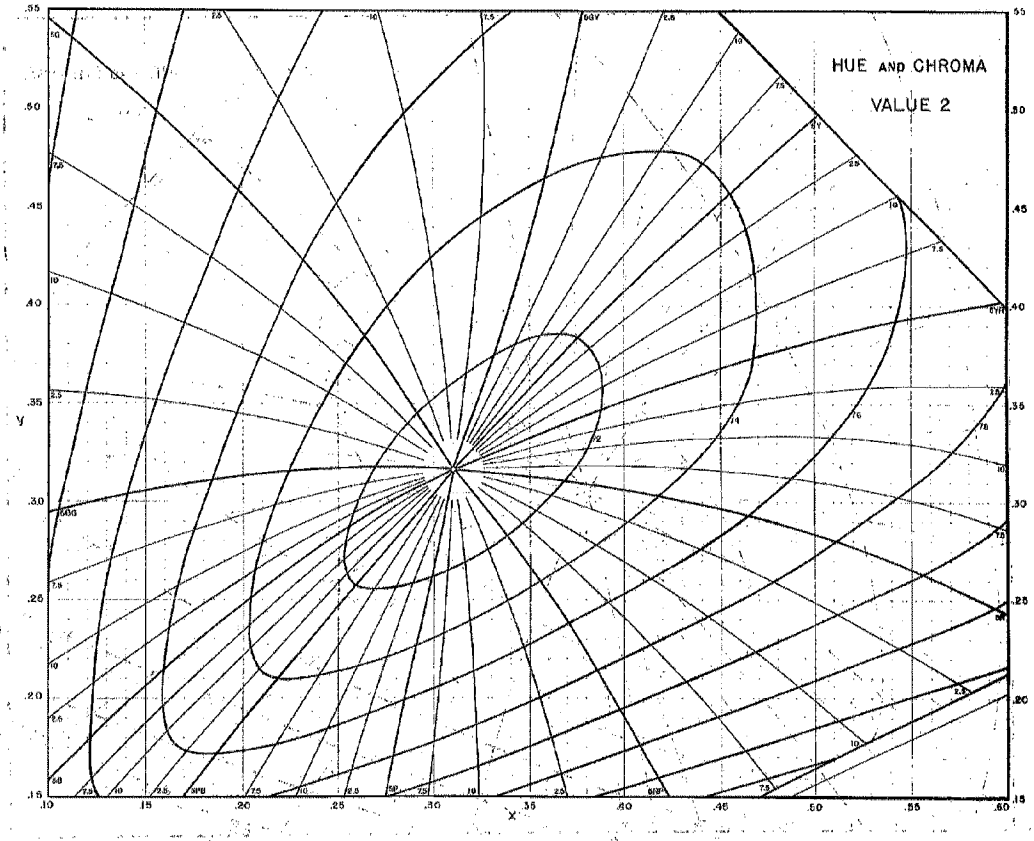

FIG. 4 Munsell Value 2—Loci of Constant Hue and Constant Chroma in CIE (x,y) Coordinates

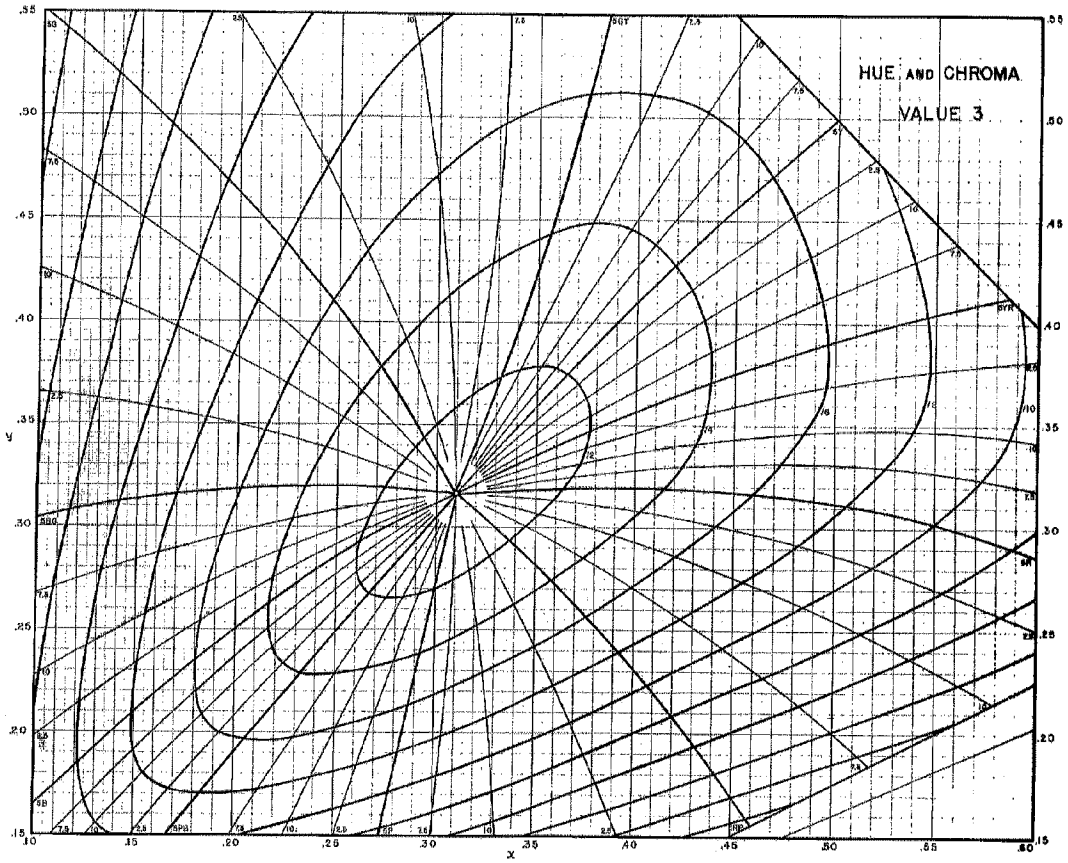

FIG. 5 Munsell Value 3—Loci of Constant Hue and Constant Chroma in CIE (x,y) Coordinates

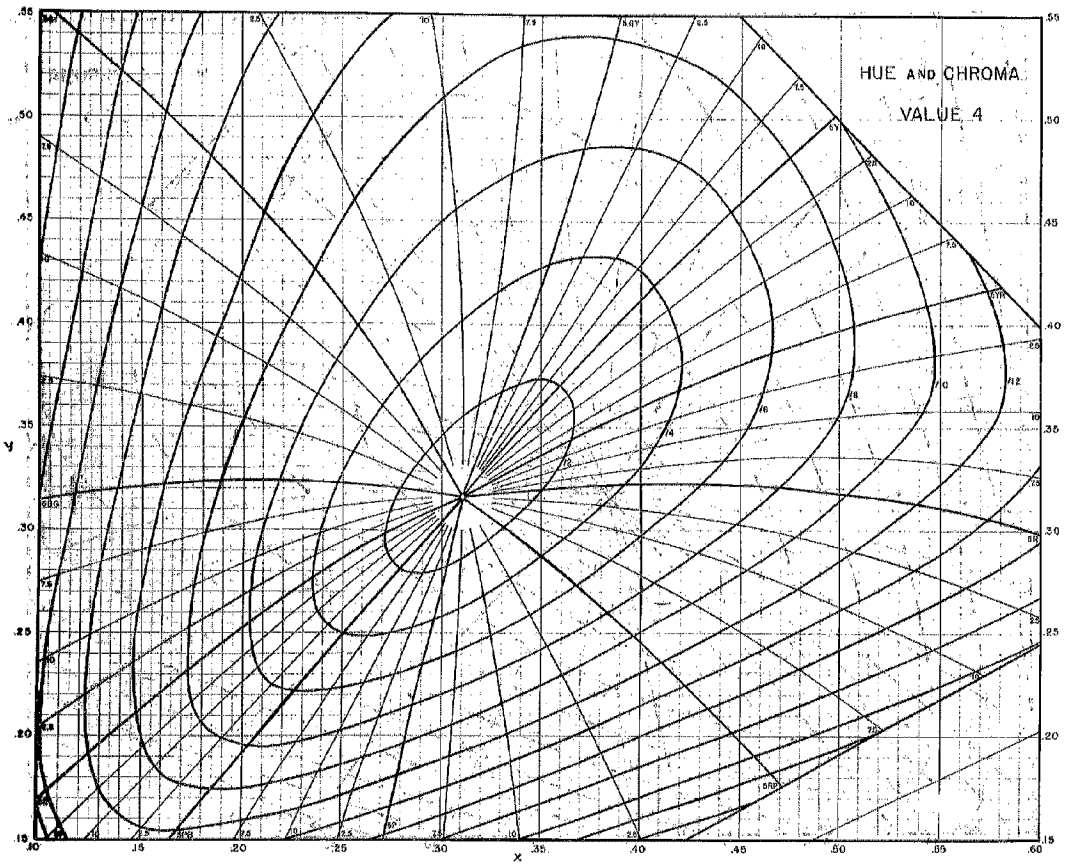

FIG. 6 Munsell Value 4—Loci of Constant Hue and Constant Chroma in CIE (x,y) Coordinates

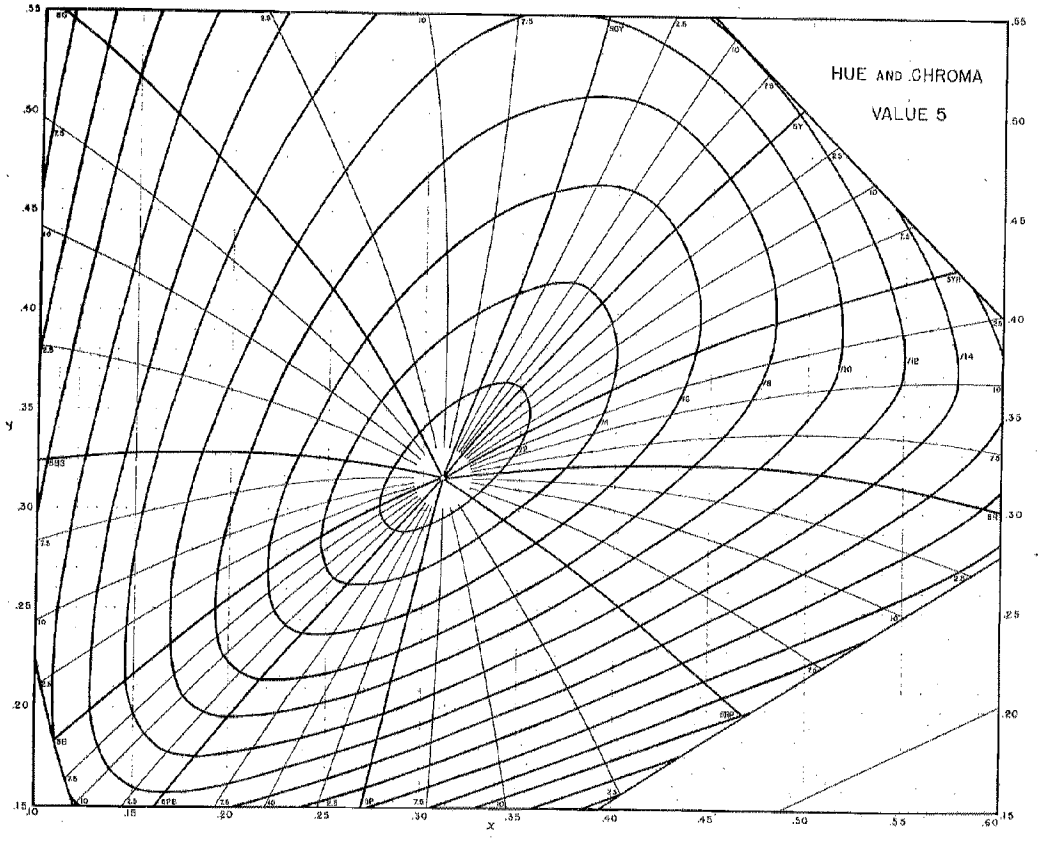

FIG. 7 Munsell Value 5—Loci of Constant Chroma in CIE (*x*,*y*) Coordinates

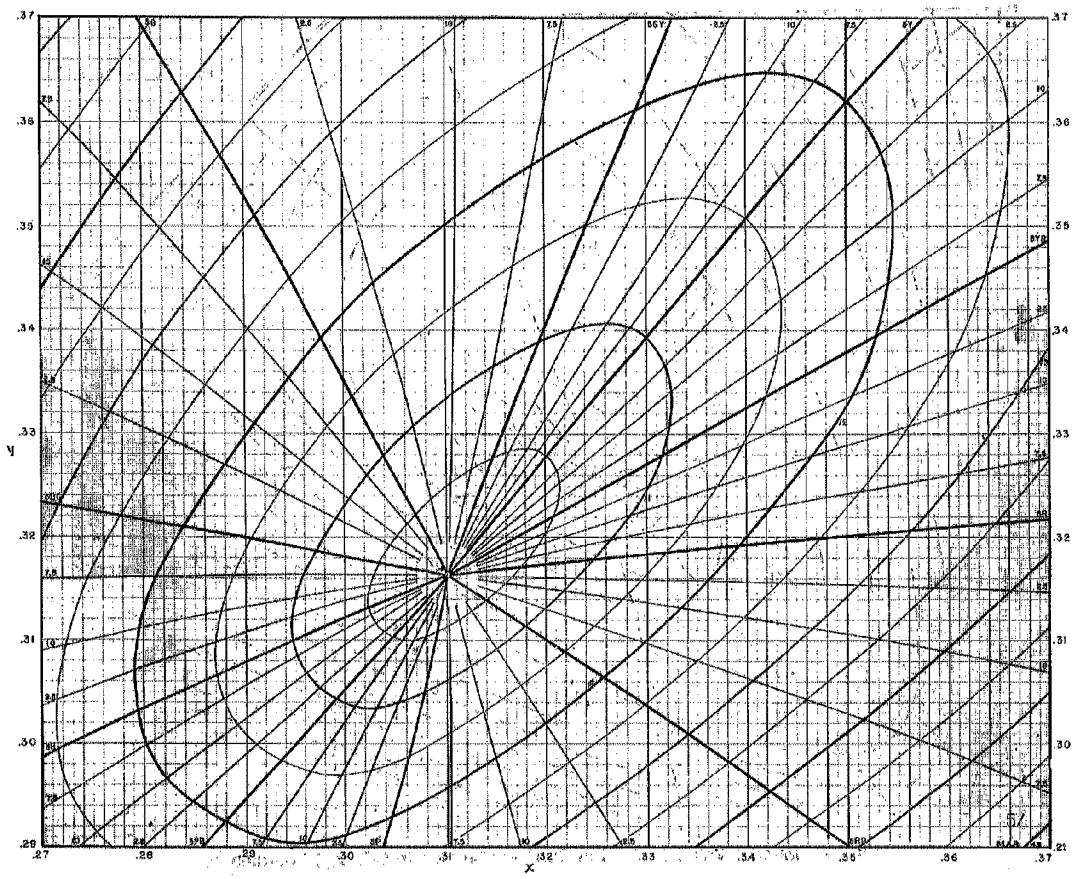

FIG. 8 Munsell Value 5—Loci of Constant Hue and Constant Chroma, Near Gray, in CIE (*x*,*y*) Coordinates

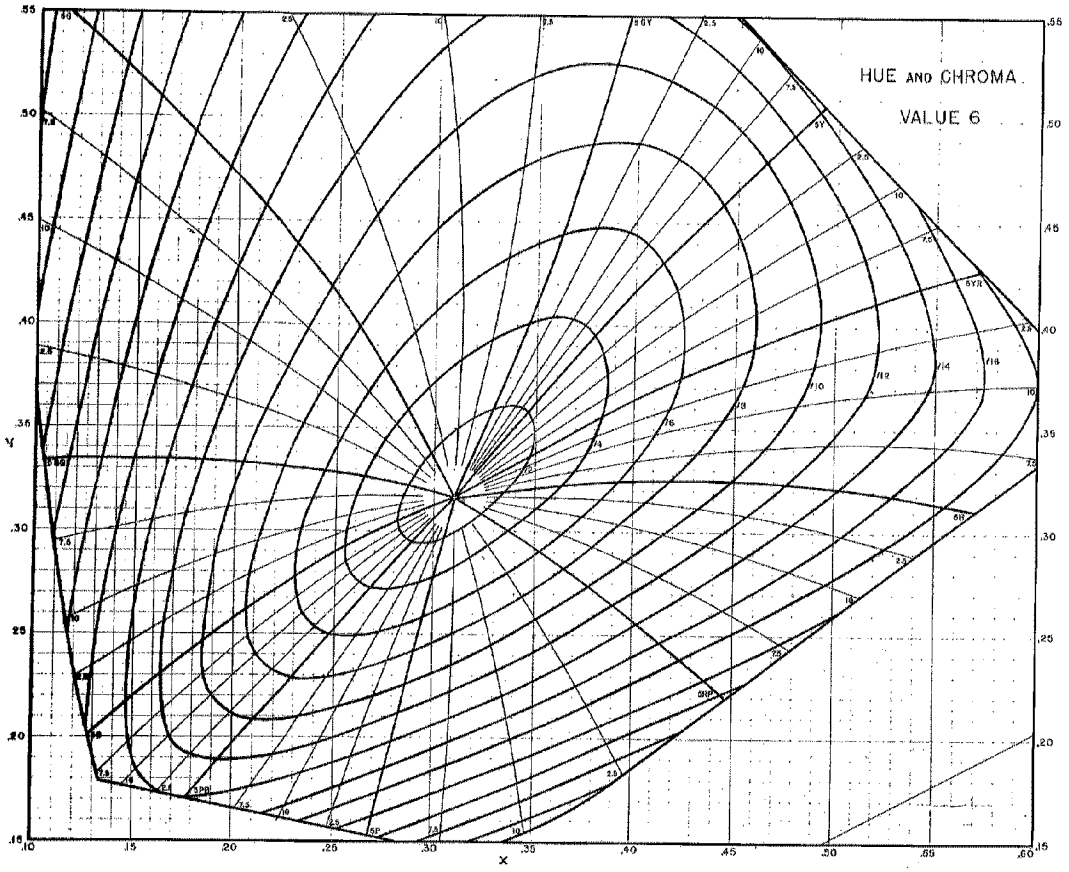

FIG. 9 Munsell Value 6—Loci of Constant Hue and Constant Chroma in CIE (*x*,*y*) Coordinates

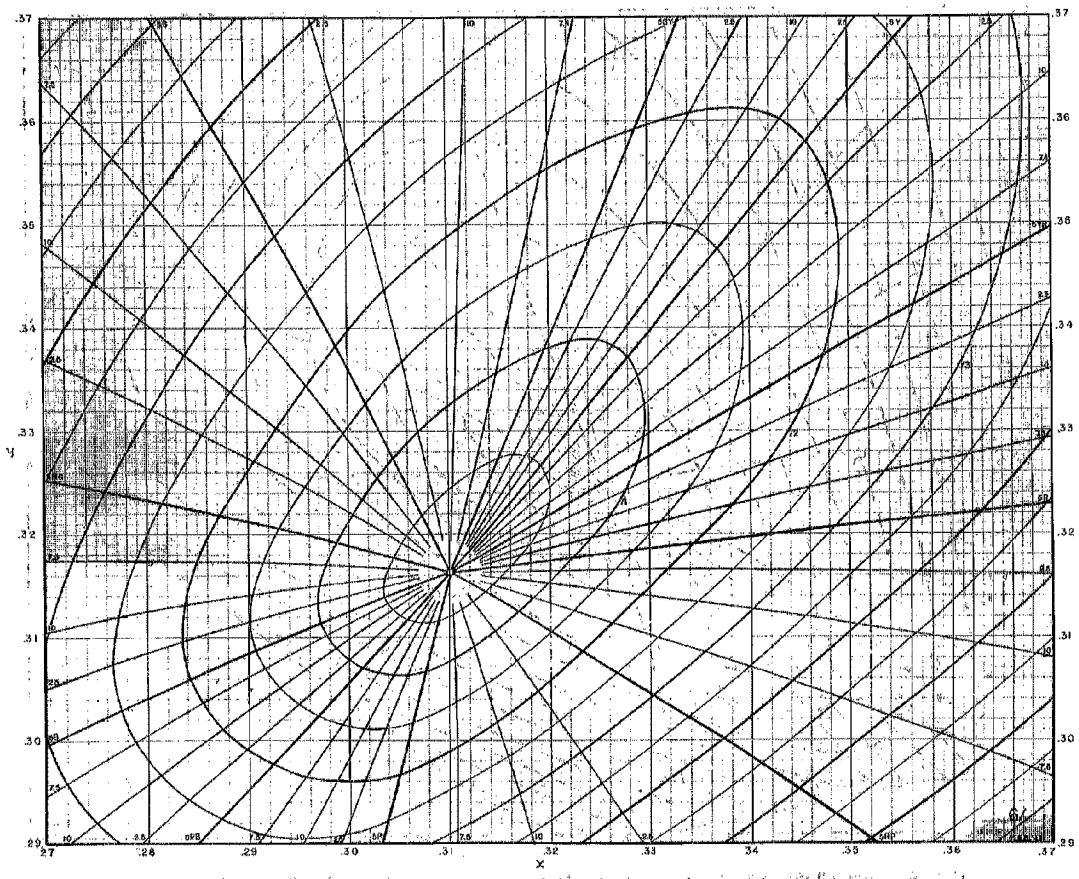

FIG. 10 Munsell Value 6—Loci of Constant Hue and Constant Chroma, Near Gray, in CIE (*x*,*y*) Coordinates

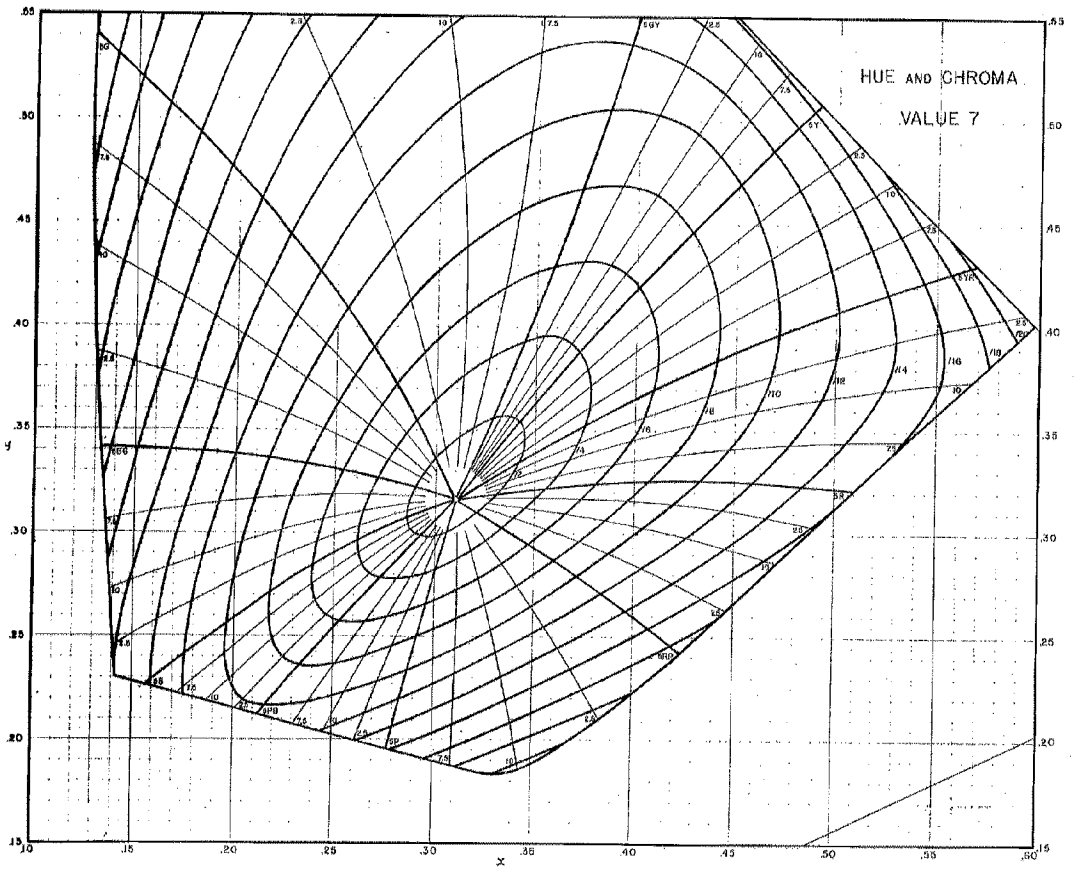

FIG. 11 Munsell Value 7—Loci of Constant Hue and Constant Chroma in CIE (x,y) Coordinates

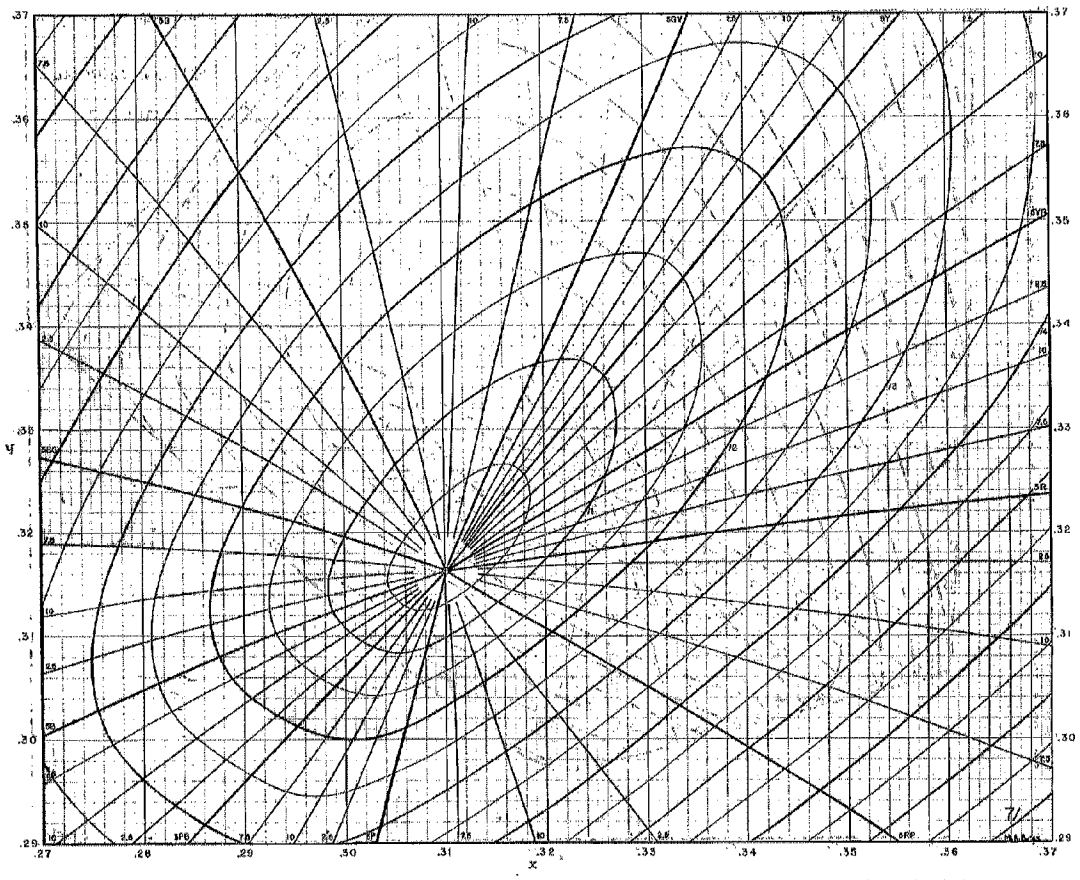

FIG. 12 Munsell Value 7—Loci of Constant Hue and Constant Chroma, Near Gray, in CIE (x,y) Coordinates

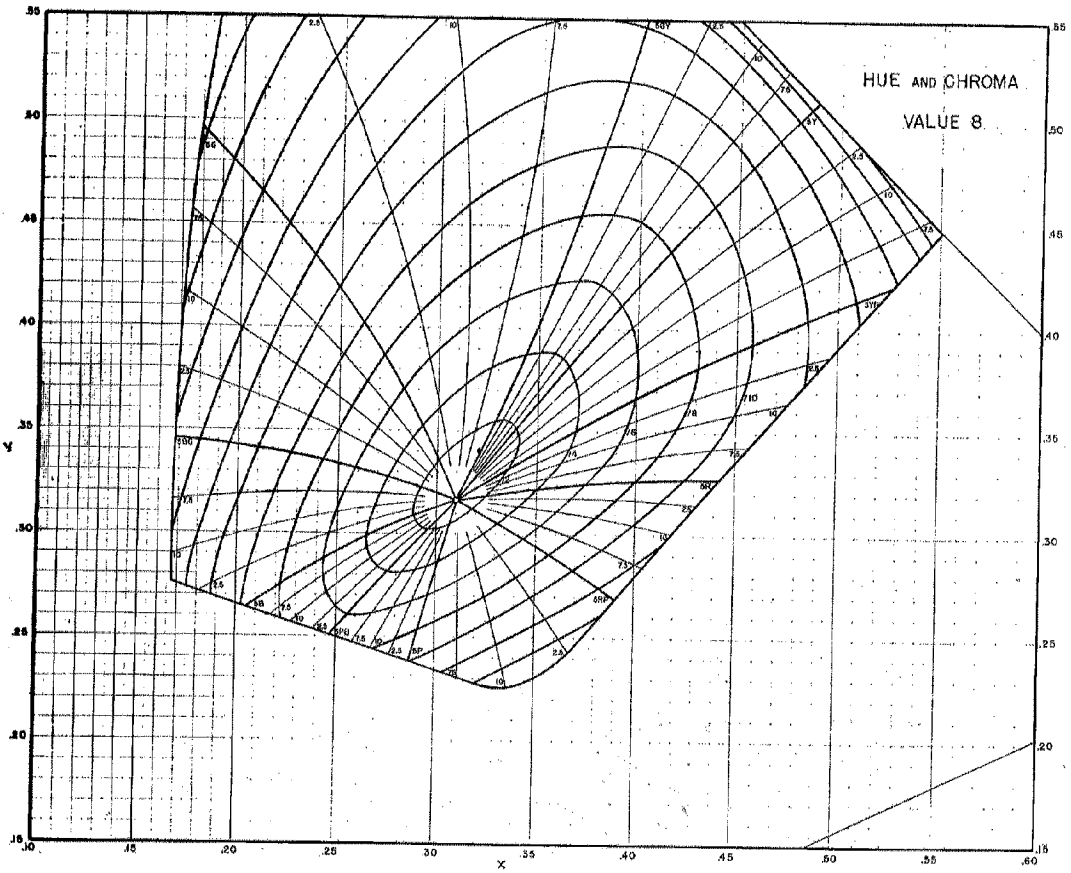

FIG. 13 Munsell Value 8—Loci of Constant Hue and Constant Chroma in CIE (x,y) Coordinates

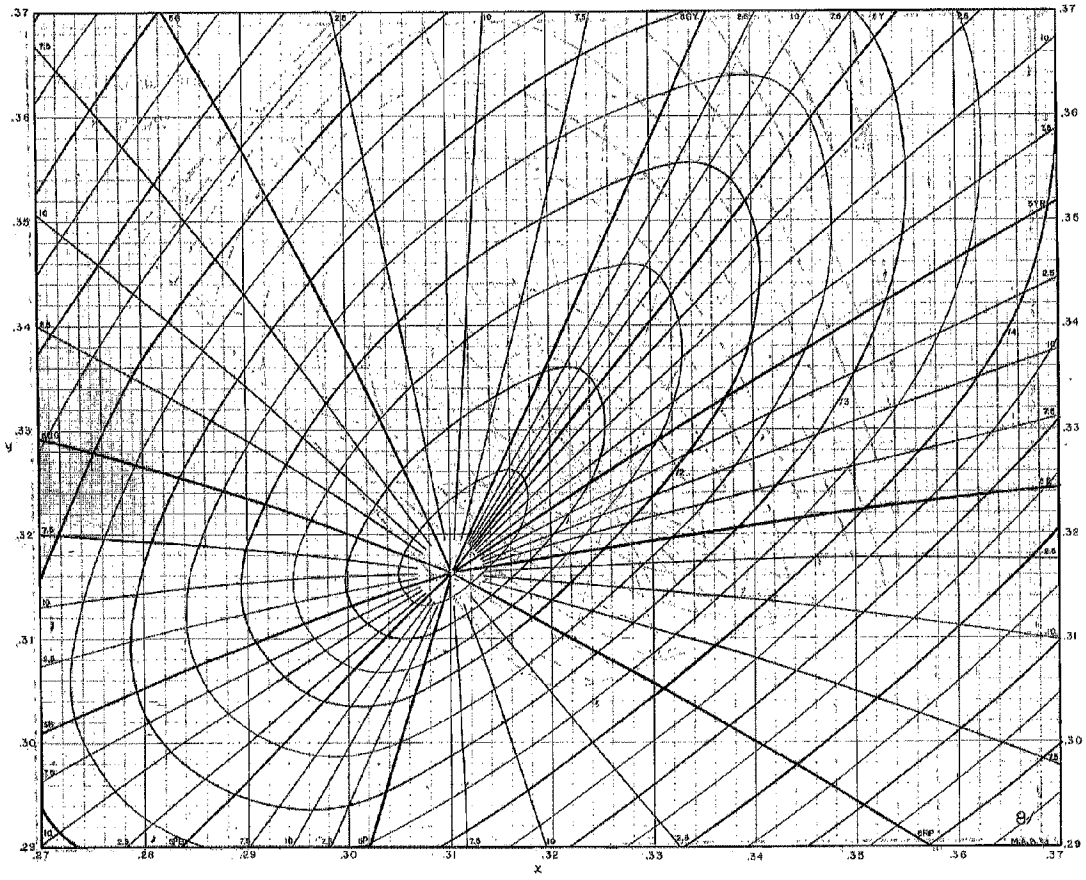

FIG. 14 Munsell Value 8—Loci of Constant Hue and Constant Chroma, Near White, in CIE (x,y) Coordinates

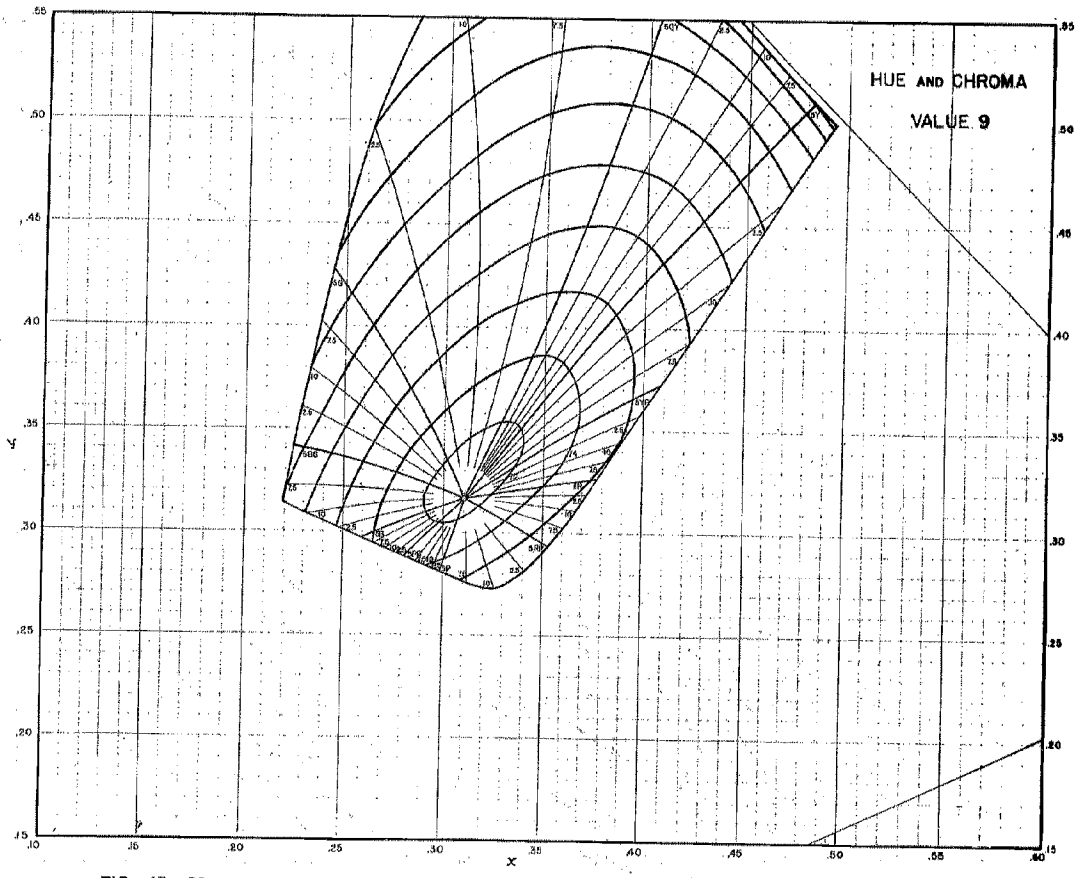

FIG. 15 Munsell Value 9—Loci of Constant Hue and Constant Chroma in CIE (*x*,*y*) Coordinates

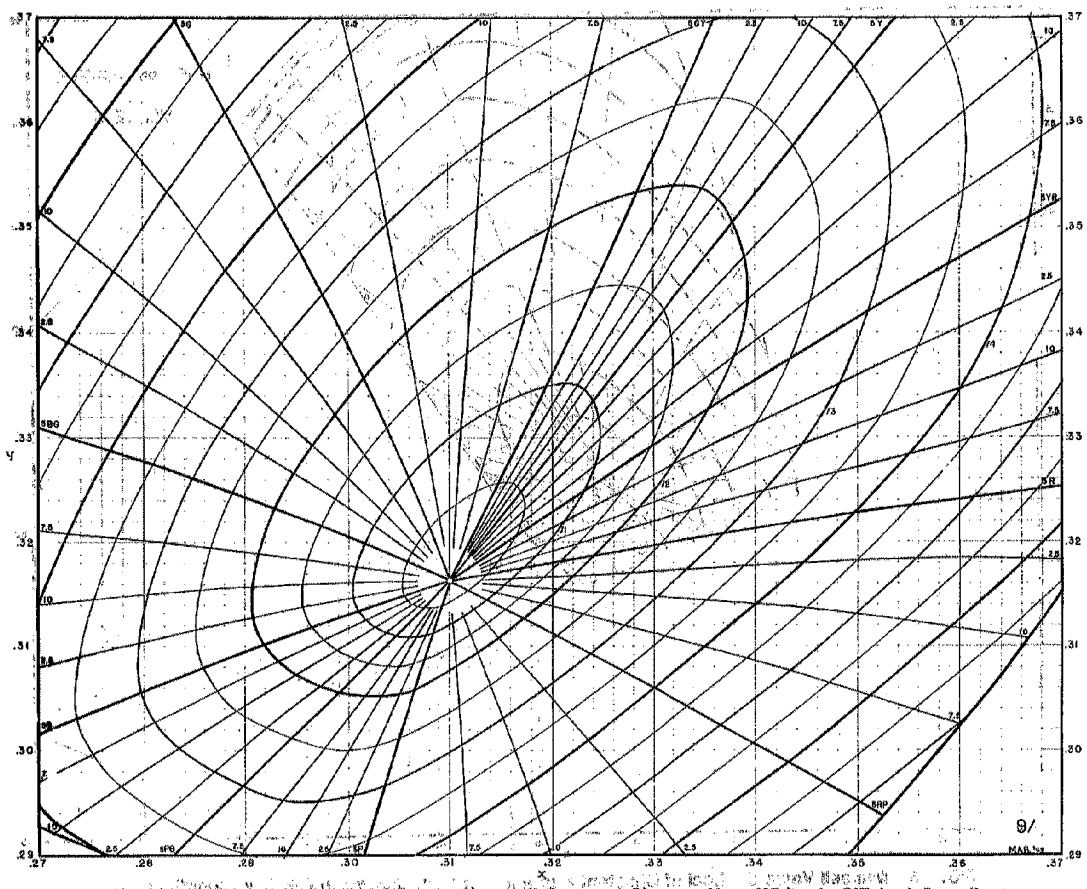

FIG. 16 Munsell Value 9—Loci of Constant Hue and Constant Chroma, Near White, in CIE (x,y) Coordinates

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.

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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.

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[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.

7.5 *Hydrochloric Acid* (3 + 197)—Dilute 30 mL of concentrated hydrochloric acid (HCl, relative density 1.19) to 2 L with water.

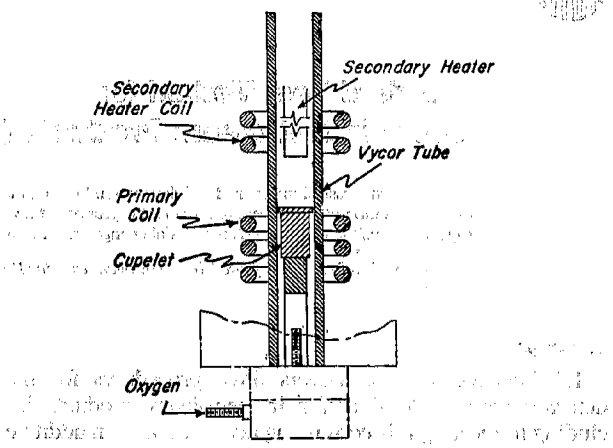

FIG. 1 Combustion Tube

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).

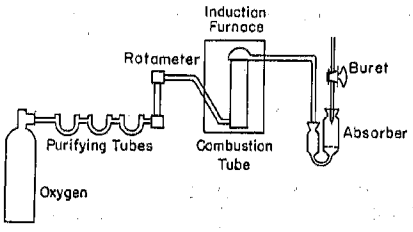

FIG. 2 Schematic Illustration of Induction-Type Furnace

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.

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.

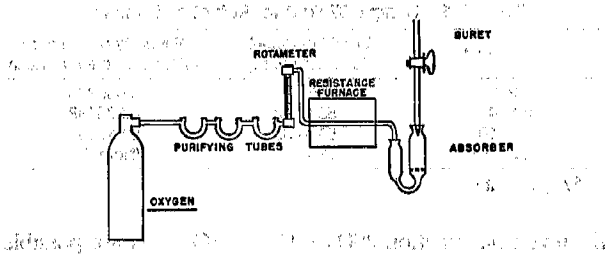

FIG. 3 Schematic Illustration of Resistance-Type Furnace

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 (⅛ 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:

$$\text{Sulfur, mass \%} = (100\,(V - V_b) \times F_s \times C)/W \qquad (3)$$

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.

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

APPROVED

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

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.

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[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:

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

$$S_O = 0.016X + 0.033$$
$$S_T = 0.060X + 0.039$$

where:

S_O = single-operator precision,
S_T = overall precision, and
X = determined concentration of copper, mg/L.

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:

$$\text{Copper, } \mu g/L = \frac{1000 \times B}{A} \quad (1)$$

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, Neucuproine (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

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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.

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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.

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[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

[7] ISO R161-1960, Pipes of Plastics Materials for the Transport of Fluids (Outside Diameters and Nominal Pressures) Part 1, Metric Series.

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)	±0.060 (±1.27)
12	12.750 (323.85)	±0.015 (±0.38)	±0.075 (±1.90)	±0.060 (±1.52)

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

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 1 (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 X1.1 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.

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	
	Unthreaded	Threaded	Unthreaded	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)

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
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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.

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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.

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[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.