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Standard Test Method for Refractive Index of Viscous Materials¹

This standard is issued under the fixed designation D1747; 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 U.S. Department of Defense.

1. Scope

1.1 This test method covers the measurement of refractive indexes, accurate to two units in the fourth decimal place, of transparent and light-colored viscous hydrocarbon liquids and melted solids that have refractive indexes in the range between 1.33 and 1.60, and at temperatures from 80 °C to 100 °C. Temperatures lower than 80 °C can be used provided that the melting point of the sample is at least 10 °C below the test temperature.

1.2 This test method is not applicable, within the accuracy stated, to liquids having colors darker than ASTM Color No. 4, ASTM color as determined by Test Method D1500, to liquids which smoke or vaporize readily at the test temperature, or to solids melting within 10 °C of the test temperature.

NOTE 1—The instrument can be successfully used for refractive indices above 1.60; but since certified liquid standards for ranges above 1.60 are not yet available, the accuracy of measurement under these conditions has not been evaluated.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (<http://www.epa.gov/mercury/faq.htm>) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

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 appro-*

priate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D362 Specification for Industrial Grade Toluene (Withdrawn 1989)³

D841 Specification for Nitration Grade Toluene

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

E1 Specification for ASTM Liquid-in-Glass Thermometers

E77 Test Method for Inspection and Verification of Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *refractive index*—the ratio of the velocity of light (of specified wavelength) in air, to its velocity in the substance under examination. The relative index of refraction is defined as the sine of the angle of incidence divided by the sine of the angle of refraction, as light passes from air into the substance. If absolute refractive index (that is, referred to vacuum) is desired, this value should be multiplied by the factor 1.00027, the absolute refractive index of air. The numerical value of refractive index of liquids varies inversely with both wavelength and temperature.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0D on Physical and Chemical Methods.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

TABLE 1 Primary Liquid Standards

Certified Standard	Approximate Refractive Index,
	n_D
<i>n</i> -Hexadecane	1.41
<i>trans</i> -Decahydronaphthalene	1.44
1-Methylnaphthalene	1.59

4. Summary of Test Method

4.1 The refractive index normally is measured by the critical angle method using monochromatic light from a sodium lamp. The instrument is previously adjusted by means of calibration obtained with certified liquid standards.

5. Significance and Use

5.1 Refractive index is a fundamental physical property that can be used in conjunction with other properties to characterize pure hydrocarbons and their mixtures.

5.2 The use of refractive index in correlative methods for the determination of the gross composition of viscous oils and waxes often requires its measurement at elevated temperatures.

6. Apparatus

6.1 *Refractometer*, precision Abbé-type,⁴ having a range in refractive index from 1.30 to 1.63. Other instruments reading to at least four decimal places may be used.

NOTE 2—When other instruments are used, follow the manufacturer's instructions for operation, maintenance, calibration, and analysis. For accepting the instrumentation for use, analysis of an NIST-traceable certified material to ensure accuracy should be performed.

6.2 *Thermostat and Circulating Pump*, capable of maintaining the indicated prism temperature constant within 0.02 °C. The circulating fluid consists of ethylene glycol or a mixture of 30 % to 40 % by volume of glycerin in water flowing through the prisms at a fixed rate of at least 2.5 L/min. For work at 100 °C, properly controlled wet steam is also suitable.

NOTE 3—The constancy of the prism temperature can be seriously affected by variations in ambient conditions, such as air drafts or changes in room temperature. Reasonable precautions should be taken to minimize these factors. Insulation placed on the thermostat, circulating fluid lines, and refractometer also may prove to be helpful.

6.3 *Thermometers, or Equivalent Temperature-Measuring Devices*, conforming to Thermometer 21C for determinations at 80 °C or Thermometer 22C for determinations at 100 °C as given in Specification E1 are recommended. See Test Method E77 for guidance on inspection and verification of mercury-in-glass thermometers. Equivalent temperature-measuring devices should have the same accuracy and resolution as Thermometers 21C and 22C.

6.3.1 In case of dispute, the test method shall be carried out using the specified mercury-in-glass thermometer.

6.3.2 The temperature-measuring device, suitably calibrated, shall be positioned to measure the temperature of the prism (see Note 4) within an appropriate holder. The holder

⁴ The Abbé-type precision refractometer is no longer available but may be obtainable from instrument exchanges or used equipment suppliers. Other precision refractometers may be suitable, but they have not as yet been tested cooperatively.

shall provide for adequate immersion of the temperature-measuring device and for free flow of the circulating fluid. The temperature-measuring device holder assembly shall be insulated with a suitable material, such as cork.

NOTE 4—In the precision Abbé type refractometer, the thermostating liquid should pass the thermometer on leaving, not on entering, the prism assembly.

6.4 *Thermocouple*,⁵ copper-constantan foil type, 0.013 mm thickness, and precision potentiometer. The thermocouple is calibrated by immersing to a depth of 25 mm in a circulating liquid thermostat and comparing with a thermometer of known accuracy.

6.5 *Light Source, Sodium Arc Lamp*—The light source shall be a sodium arc lamp, which shall be used only after the removal of Amici compensating prisms, if there are any present in the instrument.

NOTE 5—If the field division as observed in 12.2 shifts when the Amici prism is rotated, the prism should be removed to avoid incorrect readings.

7. Solvents

7.1 *Cleaning Solvent*, any suitable solvent capable of cleaning the apparatus as described in Section 10. *1,1,1*, Trichloroethane has been found suitable to use. (**Warning**—Harmful if inhaled. High concentration can cause unconsciousness or death. Contact can cause skin irritation and dermatitis.)

7.2 *Toluene*, conforming to Specification D362 or Specification D841. (**Warning**—Flammable. Vapor harmful.)

8. Reference Standards

8.1 *Primary Liquid Standards*—Organic liquids listed in Table 1, with the values of their refractive indexes for the sodium *D* line certified at 20 °C, 25 °C, 30 °C, 80 °C, and 100 °C.⁶ (**Warning**—Primary standards are combustible.)

8.2 *Working Standards*—For working standard hydrocarbons, reasonably well-purified samples of *n*-hexadecane, *trans*-decahydronaphthalene, and 1-methylnaphthalene may be used. Their exact values are determined by comparison with standard samples of the same hydrocarbons having certified values of refractive index. (**Warning**—Working standards are combustible.)

9. Sample

9.1 A sample of at least 0.5 mL is required. The sample shall be free of suspended solids, water, or other materials that tend to scatter light. Water can be removed from hydrocarbons by treatment with calcium chloride followed by filtering or centrifuging to remove the desiccant. The possibility of changing the composition of a sample by action of the drying agent, by selective adsorption on the filter, or by fractional evaporation, shall be considered.

⁵ The sole source of supply of the apparatus known to the committee at this time is RdF Corp., 23 Elm Avenue, Hudson, NH 03051. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁶ Available from API Standard Reference Office, Carnegie-Mellon University, Pittsburgh, PA 15213.