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# Standard Test Method for Refractive Index of Viscous Materials[1]

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 appropriate 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 Lubricantsand 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, $n_D$ |
|---|---|
| *n*-Hexadecane | 1.41 |
| *trans*-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 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.

[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.

[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.

## 10. Preparation of Apparatus

10.1 The refractometer shall be kept scrupulously clean at all times. Dust and oil, if allowed to accumulate on any part of the instrument, will find its way into the moving parts, causing wear and eventual misalignment. If permitted to collect on the prism, dust will dull the polish, resulting in hazy lines.

10.2 Thoroughly clean the prism faces with fresh, clean lens tissue or surgical-grade absorbent cotton saturated with a suitable solvent. Pass the swab very lightly over the surface until it shows no tendency to streak. Repeat the procedure with a fresh swab and solvent until both the glass and adjacent polished metal surfaces are clean. Do not dry the prism faces by rubbing with dry cotton.

10.3 Adjust the thermostat so that the temperature as indicated by the thermocouple inserted between the prism faces and wet with oil is within 0.2 °C of the desired test temperature. This temperature is to be held constant to within 0.02 °C during the test. Observe and record the thermometer reading corresponding to the test temperature. Turn on the sodium arc lamp and allow it to warm up for 30 min.

## 11. Standardization with Reference Liquids

11.1 Introduce a sample of the API Standard *trans*-decahydronaphthalene to the prism which is adjusted to the chosen test temperature of 80 °C or 100 °C, turn the telescope adjustment screw until a refractive index scale reading corresponding to the certified refractive index for *trans*-decahydronaphthalene is observed, and adjust the instrument according to the instructions given by the manufacturer until the sharp boundary between the light and dark portions of the field passes through the intersection of the crosshairs of the telescope.

11.2 Check the accuracy of this setting by loading a fresh sample of *trans*-decahydronaphthalene and measure its refractive index at the test temperature following the procedure described in Section 12. If the value for the refractive index differs from the certified value by 0.0001 or more units, then repeat the procedure given in 11.1 until a satisfactory check is obtained.

11.3 Measure the refractive index of API Standard *n*-hexadecane and 1-methylnaphthalene at the test temperature following the procedure described in Section 12.

11.4 Construct a calibration curve for use at the chosen test temperature. Plot the difference between the observed refractive index for *n*-hexadecane and its certified value along the ordinate against the refractive index level along the abscissa. Also plot the difference between the observed and certified refractive indices for 1-methylnaphthalene in the same manner. Draw a straight line from the point representing the deviation found for *n*-hexadecane to zero at the certified refractive index of *trans*-decahydronaphthalene. Likewise, draw a straight line from this same zero point to the deviation found for 1-methylnaphthalene.

11.5 If it is desired to measure the refractive index of samples at a temperature other than 80 °C or 100 °C, obtain calibration data by repeating 11.1 – 11.4 at this desired temperature. Determine the refractive indices for the API Standard compounds, *n*-hexadecane, *trans*-decahydronaphthalene, and 1-methylnaphthalene at the desired temperature by plotting the certified refractive indices at 20 °C, 25 °C, 30 °C, 80 °C, and 100 °C against temperature and drawing a smooth curve between the points.

11.6 *Precautions*—In using pure liquids for calibration or checking of calibration of an Abbé-type refractometer, the following precautions should be observed:

11.6.1 Before inserting the hydrocarbon calibrating liquids, the prisms should be flushed with solvents and cleaned as described in 8.2. It is advisable to preheat the solvent before use to minimize thermal shock to the prism. This should be followed by several such flushings with the test liquid and wiping with lens paper. After such cleaning, a reading with the test liquid should be taken as described in Section 11. This should be followed by another flushing with the test liquid before taking another reading of the test liquid in the prescribed manner. The prisms cannot be considered free from contaminating substances until two such determinations on the test liquid agree within the limits given in 11.6.2.

11.6.2 In setting the edge of the field on the crosshairs, readings should be taken in pairs, approaching the alidade setting from one direction only as recommended by the manufacturer. Several such sets will probably be necessary before satisfactory agreement is obtained. Satisfactory agreement is 0.00005 to 0.0001.

11.6.3 For results of highest accuracy, the calibration with hydrocarbons of known properties should be made immediately before the determination on the sample.

11.6.4 Fluctuations in ambient temperatures should be minimized as much as possible during the test.

## 12. Procedure

12.1 Thoroughly clean the prism faces as described in 10.2. Adjust the thermostat so that the temperature indicated by the thermocouple placed between the faces of the closed prism (loaded with oil) is within 0.2 °C of the desired value. The thermocouple is used for establishing the correct temperature level and may be removed during measurements of refractive index. The observed reading of the thermometer at this temperature must be held constant to 0.02 °C in the measurements to follow.

12.2 Close the prism box and let it stand for 3 min to 5 min to ensure temperature equilibrium between the prisms and the circulating bath liquid. Melt samples which are normally solid in a small container and charge as a liquid to the prism. Charge the sample from a small pipet or medicine dropper through the refractometer opening or onto the prisms open just enough to admit the sample. About 0.2 mL to 0.5 mL of the sample should be allowed to flush through before completely closing the prisms. Samples of low volatility or high viscosity may be placed directly onto the prism surface by means of a stirring rod, *being careful not to touch the prism surface with the rod.* If not enough sample has been used to fill the space between the prisms completely, or evaporation causes the field division in the telescope to become uneven, clean the prisms thoroughly before employing a new portion of the sample.