



# Standard Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids<sup>1</sup>

This standard is issued under the fixed designation D 1218; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

## 1. Scope \*

1.1 This test method covers the measurement of refractive index, accurate to four decimal places or better, of transparent and light-colored hydrocarbons in the range of 1.3300 to 1.5000 at temperatures from 20 to 30°C by manual (optical-mechanical) or automatic (digital) procedure.

1.2 The manual (optical-mechanical) procedure also covers the measurement of refractive dispersion accurate to the fourth decimal place or better.

NOTE 1—The test method may be suitable for measuring the refractive indices of liquids above 1.5000 and at temperatures both below 20°C and above 30°C; however, the test method precision may not apply. Verification of the accuracy of such measurements will depend upon the availability of reliable, certified reference standards that demonstrate the performance of the instrument used under the particular measuring conditions.

1.3 The test method may not be applicable to liquids that are strongly colored, or that have bubble points so near the test temperature that a reading cannot be obtained before substantial weathering takes place. Liquid color should be limited to No. 4 ASTM Color or lighter, as determined by Test Method D 1500.

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 1193 Specification for Reagent Water<sup>2</sup>

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)<sup>3</sup>

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance<sup>4</sup>

D 6300 Practice for the Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricant<sup>4</sup>

E 1 Specification for ASTM Thermometers<sup>5</sup>

## 3. Terminology

### 3.1 Definitions:

3.1.1 *refractive dispersion*—the difference between the refractive indexes of a substance for light of two different wavelengths, both indexes being measured at the same temperature. For convenience in calculations, the value of the difference thus obtained is usually multiplied by 10 000.

3.1.2 *refractive index*—the ratio of the velocity of light (of specified wavelength) in air, to its velocity in the substance under examination. It may also be 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. This is the relative index of refraction. 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.

## 4. Summary of Test Method

4.1 The refractive index is measured using a high-resolution refractometer of an optical-mechanical or automatic digital type with the prism temperature accurately controlled. The instrument principle is based on the critical angle concept.

## 5. Significance and Use

5.1 Refractive index and refractive dispersion are fundamental physical properties, which can be used in conjunction with other properties to characterize pure hydrocarbons and their mixtures.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04 on Hydrocarbon Analysis.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 05.04.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 14.03.

\*A Summary of Changes section appears at the end of this standard.

## PROCEDURE A—MANUAL (OPTICAL-MECHANICAL) PROCEDURE

### 6. Apparatus

6.1 *Refractometer*, high-resolution optical-mechanical refractometer of the “Abbe” type with suitable measuring range (1.3300 to 1.5000 or higher) and an accuracy/resolution of 0.0001 or better refractive index.

NOTE 2—Prior to 2001, Test Method D 1218 was based on a Bausch & Lomb Refractometer. However, this instrument is no longer manufactured. Currently available manual refractometers are of the “Abbe” type (critical angle refractometers, see X1.2.3). There is a variety of refractometers that have been found to be suitable for this measurement. Some instruments offer a wider measuring range but it is important to verify the uniformity of accuracy across the entire measuring range and to ensure suitable calibration materials are available for this verification.

6.2 *Temperature Control Unit*, either an external liquid bath with both heating and cooling capability and pump for maintaining the indicated prism temperature within 0.1°C, or an internal electronic temperature control system (such as Peltier system). If an external bath is used, the thermostating liquid shall pass the temperature measuring device on leaving, not on entering the prism. The temperature control unit (external or internal) shall have the following control specification:

Stability	± 0.1°C
Uniformity	± 0.1°C
Display Resolution	0.1°C or better

6.3 *Temperature Measuring Device*, for those apparatus employing mercury in glass thermometer, ASTM Thermometer 17C having a range from 19 to 27°C, and conforming to the requirements of Specification E 1. For apparatus using non-mercury in glass thermometer, a platinum resistance probe, thermocouple, or equivalent temperature sensors are acceptable.

6.4 *Light Sources*—The following type of light source can be used in conjunction with an optical-mechanical (Abbe type) refractometer:

- 6.4.1 *Sodium Arc Lamp*, Na<sub>D</sub> line at 589 nm.
- 6.4.2 *Mercury Arc Lamp*, Hg<sub>c</sub> line at 546 nm or Hg<sub>g</sub> line at 436 nm.
- 6.4.3 *Cadmium Arc Lamp*, Cd<sub>C</sub> line at 644 nm.
- 6.4.4 *Mercury-Cadmium Arc Lamp*.
- 6.4.5 *Helium Discharge Lamp*.

NOTE 3—Measurement of refractive dispersion requires more than one type of light source.

6.5 *Light Filters*—Arc lamps can emit a number of spectral lines that result in multiple-borderlines observed in the refractometer. Filters can be used to eliminate unwanted lines (borderlines). Depending upon instrument design, the manufacturer will recommend and supply a suitable filter or adapt a commercially-available filter (for example, interference filter) to suit the application.

### 7. Reagents and Materials

7.1 *n-Pentane, 95 mol % Minimum Purity*, for cleaning the prism faces. (**Warning**—Flammable. Harmful if inhaled. Vapors may cause flash fire.)

NOTE 4—Low boiling hydrocarbon fractions with boiling range 50 to 100°C have also been found to be acceptable.

7.2 *Toluene, HPLC Grade*, for cleaning the prism faces. (**Warning**—Flammable. Vapor harmful.)

7.3 *1-Bromonaphthalene, 98 mol % Minimum Purity*, contact liquid when calibrating with solid reference standard. (**Warning**—Toxic when ingested.)

7.4 *Primary Reference Materials*, for calibrating the instrument.

7.4.1 *Solid Reference Standard*, with the value of refractive index engraved on its upper face.

7.4.2 *Distilled or Deionized Water*, conforming to Type II or III of Specification D 1193. At 20°C,  $n_D = 1.3330$ ; at 25°C,  $n_D = 1.3325$ ; and at 30°C,  $n_D = 1.3319$

7.4.3 *2,2,4-trimethylpentane, 99 mol % Minimum Purity*, at 20°C,  $n_D = 1.3915$ ; at 25°C,  $n_D = 1.3890$ .

7.4.4 *Methylcyclohexane, 99 mol % Minimum Purity*, at 20°C,  $n_D = 1.4231$ ; at 25°C,  $n_D = 1.4206$ .

7.4.5 *Toluene, 99 mol % Minimum Purity*, at 20°C,  $n_D = 1.4969$ ; at 25°C,  $n_D = 1.4941$ . (**Warning**—2,2,4-trimethylpentane, methylcyclohexane, and toluene are all flammable. Their vapor can be harmful.)

NOTE 5—Other pure materials may be used to calibrate the instrument as primary reference material, as long as they can be obtained in 99 mol % minimum purity and accurate values of their refractive indices at specific temperatures are available. The precision of the test method (see 15.1 and 24.1) were obtained using distilled water as the calibrant.

7.5 *Secondary Reference Materials*, for calibrating the instrument.

7.5.1 *Mineral Oil Calibration Standards*, measured and certified by suppliers for specific refractive index ranges and temperatures.

### 8. Sampling

8.1 A sample of at least 0.5 mL is required. The sample shall be free of suspended solids, water, or other materials that may settle onto the prism surface and affect the measured reading. 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 the sample by action of the drying agent, by selective adsorption on the filter, or by fractional evaporation, shall be considered. (**Warning**—Volatile hydrocarbon samples are flammable.)

### 9. Preparation of Apparatus

9.1 The refractometer shall be kept scrupulously clean at all times. Dust and oil can impair the optical component of the instrument. Thoroughly clean the prism faces with toluene, followed by *n*-pentane (see Note 4) (**Warning**—These materials are extremely flammable. Harmful if inhaled. Vapors may cause flash fire.) using cotton swabs, fresh clean lens tissue, or similar material, in accordance with manufacturer’s instructions. Do not dry the prism faces by rubbing with dry cotton.

9.2 Adjust the thermostat bath/circulator settings or the electronic temperature control system so that the temperature indicated by the refractometer temperature measuring device is within 0.1°C of the desired value. Turn on the light source and allow the refractometer to equilibrate for 30 min.

NOTE 6—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.

## 10. Calibration of Refractometer Using Solid Reference Standard

10.1 Thoroughly clean the prism faces and surfaces of the solid reference standard (see 7.4.1). Open the prism assembly. Apply a drop of 1-bromonaphthalene contact liquid, about 1.5 mm in diameter, to the center of the polished surface of the solid reference material. Press the reference standard against the surface of the prism face with the polished end towards the light source.

10.2 Follow the manufacturer's instructions on how to calibrate the instrument using the solid reference standard.

10.3 If the observed refractive index differs from the value engraved on the solid reference standard by more than 0.0001, adjust the refractometer's scale reading to match the certified value, following manufacturer's instructions.

## 11. Calibration of the Refractometer Using Liquid Reference Standards

11.1 Using the procedure described in Section 12, determine the refractive index of any of the Reference Materials specified in 7.4 or 7.5 for a specific test temperature. If the observed refractive index for the chosen reference material at a specified test temperature differs by more than 0.0001 of the listed value, make adjustment to the instrument following manufacturer's instructions so that the observed refractive index corresponds to the listed value.

11.2 For optimum accuracy, use a reference material whose refractive index is close to the desired refractive index range and temperature to calibrate the instrument.

## 12. Procedure

12.1 Ensure that the prism faces are clean and dry. Check that the prism temperature is within 0.1°C of the desired temperature.

12.2 Unlock (if necessary) and open the prism assembly.

12.3 Place one or two drops of the sample on the lower prism face. Close the prism assembly and lock (if necessary). Turn on the light source. Allow 3 min temperature equilibration time.

12.4 Look through the eyepiece and observe the field consisting of a light and dark portion. Follow manufacturer's instructions to adjust the instrument so that the boundary between the light and dark portions of the field is as sharp as possible.

12.5 Following manufacturer's instructions, make any additional adjustment until the sharp boundary line intersects the midpoint of the crosshairs superimposed on the field.

12.6 Read the refractive index on the scale. Repeat 12.5 at least four times, approaching from either side of the sharp boundary line, and average the scale readings.

12.7 Record and report the average refractive index value.

12.8 If instrument is capable of determining refractive dispersion, change the light source to a light source with different wavelength. Determine the refractive index at the secondary wavelength following 12.4-12.7.

NOTE 7—When determining refractive dispersion, it is expected that the instrument would have been calibrated at both wavelengths used.

## 13. Quality Control

13.1 Confirm the performance of the test procedure by analyzing a quality control (QC) sample, which is stable and representative of the sample of interest.

13.1.1 When quality control/quality assurance (QC/QA) protocols are established in the testing facility, these may be used to confirm the reliability of the test result.

13.1.2 When there are no QC/QA protocols established in the testing facility, Appendix X4 can be used to provide guidelines in performing this function.

## 14. Calculation and Report

14.1 Report the average refractive index to four decimal places and the test temperature at which the test was made, for example:

$$n_D = x.xxxx \text{ at } 20^\circ\text{C} \text{ or } n_{589} = x.xxxx \text{ at } 20^\circ\text{C} \quad (1)$$

where  $D$  or the 589 signifies that the sodium  $D$  spectral line was used.

14.2 If a refractive dispersion measurement was made, calculate the absolute value of the difference between the refractive indices at the two wavelengths and multiply the difference by 10 000. Report the calculated value and the test temperature, for example:

$$|(n_g - n_D)| \times 10^4 \text{ at } t = 20^\circ\text{C} \quad (2)$$

where  $g$  and  $D$  signifies that the mercury  $g$  and sodium  $D$  spectral lines were used in the measurement.

## 15. Precision and Bias

15.1 *Precision—Refractive Index:*

15.1.1 The precision for the manual (optical-mechanical) refractive index procedure, as determined by the statistical examination of interlaboratory test results conducted in 1996<sup>6</sup> on eight different samples from ten laboratories, is as follows:

15.1.2 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus, under constant operating conditions, and 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 (95 % confidence level):

$$r_{\text{manual}} = 0.0002 \quad (3)$$

<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1493.