Standard Test Method for
Refractive Index and Refractive Dispersion of Hydrocarbon Liquids

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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of refractive indexes, accurate to six units in the fifth decimal place, and refractive dispersions, accurate to twelve units in the fifth decimal place, of transparent and light-colored hydrocarbon liquids that have refractive indexes in the range from 1.33 to 1.50, and at temperatures from 20 to 30°C. The test method is not applicable within the accuracy stated to liquids having colors darker than No. 4 ASTM Color as determined by Test Method D 1500, to liquids having bubble points so near the test temperature that a reading cannot be obtained before substantial weathering takes place, to liquids having a refractive index above 1.50, or to measurements made at temperatures above 30°C.

NOTE 1—The instrument can be successfully used for refractive indexes above 1.50, and at temperatures both below 20°C and above 30°C, but as yet certified liquid standards for the ranges above a refractive index of 1.50 are not available, so the precision and accuracy of the instrument under these conditions have not been evaluated. Similarly, certified refractive indexes of liquids at temperatures other than the 20 to 30°C range are not available, although the instrument can be used up to 50°C.

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

2. Referenced Documents

2.1 ASTM Standards:
D 841 Specification for Nitration Grade Toluene
D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
E 1 Specification for ASTM 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. 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.

3.1.2 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.

4. Summary of Test Method

4.1 The refractive index is measured by the critical angle method with a Bausch & Lomb Precision Refractometer using monochromatic light. The instrument is previously adjusted by means of a solid reference standard and the observed values are corrected, when necessary, by a calibration obtained with certified liquid standards.

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.

6. Apparatus

6.1 Refractometer, Bausch & Lomb, “Precision” type, range 1.33 to 1.64 for the sodium D line.

6.2 Thermostat and Circulating Pump, capable of maintaining the indicated prism temperature constant within 0.02°C of the desired test temperature. The thermostating liquid should pass the thermometer on leaving, not on entering, the prism assembly.

6.3 Thermometer—ASTM Saybolt Viscosity Thermometer 17°C having a range from 19 to 27°C, and conforming to the requirements of Specification E 1. The thermometer shall be used in an approved holder, as shown in Fig. 1, such that almost total immersion (not more than emergent stem) is obtained, and reading to 0.01°C is possible.

6.4 Light Sources—The following light sources have been found satisfactory:

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 D 02.04 on Hydrocarbon Analysis.

2 Designation: D 1500 - 92

3 Designation: D 841 - 92.

4 Annual Book of ASTM Standards, Vol 06.04.

5 Manufactured by Bausch & Lomb Optical Co., Rochester, NY, Catalog No. 33-45-03. All instrument terminology used in this method corresponds with that used in the “Reference Manual” supplied with the instrument. Production of this refractometer was discontinued in 1976. However it may be obtainable from instrument exchanges or used equipment suppliers. If other available instrumentation is used, the precision statements of Section 13 will not apply.
6.4.1 Sodium Arc Lamp—The Unitized “Sodium Lab Arc” is furnished with the instrument.

6.4.2 Mercury Arc Lamp—The H-4 type capillary mercury arc is furnished as an accessory to the refractometer.

6.4.3 Hydrogen Discharge Lamp—Any type of lamp capable of producing light having an intensity of at least 32 lx (3 footcandles) on an area of 1 cm² on the entrance face of the illuminating prism. The luminous intensity may be conveniently measured by means of a photographic light meter held 254 mm (10 in.) from the lamp and perpendicular to the light beam. For convenience, the lamp should be mounted on an extension of the sodium lamp support.

6.4.4 Other Sources—Helium may be used in place of hydrogen in the lamp discussed in 6.4.3.

6.4.5 Light Filters—For isolating the various spectral lines from the above sources, special light filters are required. The following are tentatively recommended:

<table>
<thead>
<tr>
<th>Wave-length, Å</th>
<th>Spectral Line</th>
<th>Filter</th>
</tr>
</thead>
<tbody>
<tr>
<td>6678</td>
<td>Helium</td>
<td>Corning No. 2404</td>
</tr>
<tr>
<td>6356</td>
<td>H₆</td>
<td>None required. May use Corning No. 2404.</td>
</tr>
<tr>
<td>5893</td>
<td>NaD</td>
<td>None required.</td>
</tr>
<tr>
<td>5461</td>
<td>Hg₆</td>
<td>Watten No. 62, or No. 77A, Corning Nos. 3486 + 4303 + 5120.</td>
</tr>
<tr>
<td>5016</td>
<td>Helium</td>
<td>Watten No. 45.</td>
</tr>
<tr>
<td>4861</td>
<td>H₂</td>
<td>Corning Nos. 5030 + 3387, 4303, or Watten No. 45.</td>
</tr>
<tr>
<td>4358</td>
<td>Hg₂</td>
<td>Corning Nos. 5113, 3389 + 5850.</td>
</tr>
</tbody>
</table>

Note 2—In determinations of refractive indexes above approximately 1.53 (wherever the short wavelengths show a higher scale reading than the long) this system of filters is rendered worthless and filters must be chosen which remove all spectral lines of shorter wavelength than the one being read. Below this refractive index, the specific filters listed above, which remove spectral lines of longer wavelengths than the one being read, should be used.

7. Solvents

7.1 n-Pentane, 95 mol % minimum purity.

Note 3: Warning—Extremely flammable. Harmful if inhaled. Vapors may cause flash fire.

7.2 Toluene, conforming to Specification D 841.

Note 4: Warning—Flammable. Vapor harmful.

8. Reference Standards

8.1 Solid Reference Standard, accurate to ±0.00002 with the value of the refractive index engraved upon its upper face.

8.2 Primary Liquid Standards—The organic liquids listed below, with the values of their refractive indexes for the D, F, and C lines certified at 20, 25, and 30°C, obtained from the API Standard Reference Office.¹

<table>
<thead>
<tr>
<th>Compound</th>
<th>nD</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,2,4-Trimethylpentane</td>
<td>1.39</td>
</tr>
<tr>
<td>Methycyclohexane</td>
<td>1.42</td>
</tr>
<tr>
<td>Toluene</td>
<td>1.49</td>
</tr>
</tbody>
</table>

Note 5: Warning—Flammable.

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 of the filter, or by fractional evaporation, shall be considered.

Note 6: Warning—Volatile hydrocarbon samples are flammable.

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 their 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 a swab of surgical-grade absorbent cotton saturated with a suitable solvent such as toluene. Pass the swab very lightly over the surface until it shows no tendency to streak. Repeat this procedure with n-pentane until both the glass and the 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 indicated by the refractometer thermometer is within ±0.02°C of the desired value; turn on the sodium vapor lamp and allow it to warm up 30 min.

Note 7—An error of 0.02°C in temperature of the sample will cause an error of $1 \times 10^{-5}$ in the refractive index of methycyclohexane.

10.4 Control the ambient temperature within 1°C of the test temperature. This can be done by regulation of the room temperature or by placing the instrument inside a specially designed constant-temperature box. The instrument shall also be so situated that it will not be subject to drafts.

¹ Carnegie-Mellon University, Pittsburgh, PA.
11. Standardization of Apparatus and Technique

11.1 Thoroughly clean the prism faces and surfaces of the solid reference standard as described in 10.2, finally brushing the surfaces with a clean camel's-hair brush. Fix the hinged part in a wide-open position. Apply a drop of monobromo-naphthalene, about 1.5 mm in diameter, to the center of the polished surface of the reference standard. Press the reference standard against the surface of the stationary prism with the polished end toward the light. If the proper amount of contacting liquid has been used, a continuous film of liquid will form between the prism and the reference standard, and the field will appear evenly illuminated. If not, irregular dark spots will appear in the illuminated field of the telescope when the knurled knob is turned and the light is in line with the longitudinal axis of the telescope. Gently manipulate the reference standard by pressure on one edge or another until the interference bands, as seen with the aid of the auxiliary lens, appear to extend horizontally in the rectangular contact area. It is well to keep the liquid wedge at such an angle that three to five bands can be seen, and the fringe pattern should appear centered in the exit pupil of the telescope.

Note 8—If there is any trace of roughness as the contact is being made, remove the reference standard and clean all surfaces again. More damage can be done to the prism surface in this operation than in weeks of use with liquids, if grit comes between the two surfaces during this contact. The amount of liquid should be just enough to fill the contact area completely, leaving no liquid at the front edge of the reference standard.

11.2 Set the instrument to the scale reading corresponding to the refractive index engraved on the solid reference standard. Rotate the sodium lamp base while viewing the telescope until a sharp vertical line appears in the illuminated field and does not move with the rotation of the lamp. Adjust the eyepiece of the telescope to bring the cross hairs into sharp focus.

11.3 Move the alidade by means of the hand wheel until the critical line on the left side of the band intersects the cross hairs, and read the scale. Repeat the setting at least twice and, between settings, shift the lamp slightly while observing the critical line in order to make sure a false line is not being observed. Average the scale readings for all the settings.

11.4 Convert the average scale reading to refractive index by means of the table for the sodium D line. To give correct readings, without application of corrections, the average value obtained may differ from that engraved on the test specimen by more than 0.00002.

11.5 If adjustment is necessary, set the scale to the reading corresponding to the value engraved on the solid reference standard, by means of the hand wheel on the side of the instrument. If the critical line is to the left of the intersection of the cross hairs, loosen the small screw on the left of the telescope and slowly tighten the one on the right until the lines coincide; if the critical line is to the right of the intersection, use the opposite procedure. At the final adjustment both screws should be snug but not tight. Again check the setting as in 11.3.

12. Standardization with Reference Liquids

12.1 Measure the refractive indexes of each of the primary liquid standards listed in 8.2 for the D, F, and C lines, at the test temperature 20, 25, or 30°C, following the procedure described in Section 13. If the values obtained do not agree with the certified values within 0.00003, determine a correction curve for each wavelength from an average of five independent determinations on each of the three certified liquid standards. A plot of the average error against refractive index provides a correction for all observed indexes between these points.

Note 9—This does not imply that the refractive index engraved on the test specimen is necessarily inaccurate, but tends to correct an error introduced in the determination by the failure to obtain grazing incidence in the case of liquid samples. This fault, and other instrumental errors, if present, are inherent in the refractometer design and their magnitude varies with the refractive index of the liquid and different instruments.

12.2 To observe any changes with time and use in the relative positions of prism and alidade, each operator shall check the instrument with the calibrated solid reference standard prior to his use of the instrument.

13. Procedure

13.1 Thoroughly clean the prism faces as described in 10.2. Adjust the thermostat so that the temperature indicated by the refractometer thermometer is within 0.02°C of the desired value.

13.2 In testing nonviscous liquid samples, close the prism box and let stand for 4 to 5 min to ensure temperature equilibrium between the prisms and the circulating water. By means of a small pipet or medicine dropper, introduce a small quantity of sample into the tubing between the prism faces. Turn the knurled head at the base of the telescope so as to bring the auxiliary lens into the light path, and observe through the face of the working prism. If the space between the prisms is completely filled with liquid, the field will be uniformly illuminated; bubbles or unfilled spaces will appear black. If the space is not completely filled, open the prism box slightly several times and add more liquid. Do not attempt to measure refractive indexes until the space between the prisms is completely filled.

13.3 In testing viscous liquids, open the prism box and apply the sample to the faces of both prisms, spreading evenly with a round wooden applicator stick. Never use metal or glass for this purpose as these may scratch the prism faces. Close the prism box slowly to avoid straining the hinge and locking mechanism.

13.4 Adjust the illuminant to be in line with the telescope and bring the border line approximately to the reticle. While viewing the rear prism face by means of the auxiliary lens, rotate the lamp bracket to the right until only the extreme left side of the prism appears to be illuminated. If this rotation is carried too far, vertical interference lines will appear in the back face. These are generally irregular and rather faint. The best adjustment for contrast and illumination seems to be the point just before these fringes become distinct.

13.5 Adjust the eyepiece of the telescope so as to bring the cross hairs into sharp focus, set the cross hairs on the critical edge and read the scale of the instrument. Readjust the position of the vapor lamp and repeat at least four times, approaching from either side of the critical edge, and record the average scale reading. (In order to avoid the possibility of using a false edge, it is best to adjust the position of the light
source each time a setting is made rather than make four settings on one positioning of the lamp.

13.6 Without changing the position of the prism assembly, place other desired light sources into the angular position (with respect to the rear face of the refracting prism) occupied by the sodium lamp. Take average scale readings for the desired lines in the manner described in 13.4.

13.7 In testing volatile samples, clean the prism faces without changing the position of the prism assembly or the lamp, recharge with sample, and read immediately.

14. Calculation and Report

14.1 Convert the observed scale readings to refractive indexes by use of the tables supplied with the instrument and report these values and the temperature at which the test was made, distinguishing between the various spectral lines used (for example, \( n_D = 1 \cdots \) or \( n_{589.3} = 1 \cdots \)).

14.2 To obtain refractive dispersion, subtract, \( n_\Lambda_2 \) and \( n_\Lambda_1 \). Report the result and the temperature at which the test was made (for example \( (n_{F} - n_C) \times 10^4 \text{ at } t = \cdots \) or \( (n_{K} - n_D) \times 10^4 \text{ at } t = \cdots \)).

15. Precision and Bias

15.1 Results should not differ from the mean by more than the following amounts:

| 
| --- | --- |
| **Reproducibility** | **Repeatability** |
| One Operator Apparatus | Different Operators and Apparatus |
| Refractive index | 0.00006 | 0.00006 |
| Refractive dispersion | 0.00012 | 0.00012 |

15.2 **Bias**—The difference of results from the established value when compared to pure reference materials is not expected to be more than.

- Refractive Index \( \pm 0.00006 \)
- Refractive Dispersion \( \pm 0.00012 \)

15.2.1 Specific bias has not been established by cooperative testing.

15.3 The precision of this test method was not obtained in accordance with Research Report RR: D-2-1007, “Manual on Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants.”

16. Keywords

- hydrocarbons; refractive dispersion; refractive index; refractometer

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