

REFRACTIVE INDEX OF HYDROCARBON LIQUIDS

UOP Method 816-80

SCOPE

This method is for determining the refractive index, accurate to one unit in the fourth decimal place, of transparent and light-colored hydrocarbon liquids in the range of 1.33 to 1.60 at a temperature of 20 °C. This method is not applicable to liquids having colors darker than ASTM Color No. 4 (see ASTM Method D 1500) or to liquids having bubble points so near the test temperature that a reading cannot be obtained before substantial weathering takes place.

OUTLINE OF METHOD

The refractive index is measured with a precision refractometer using a monochromatic (sodium) light source. The calibration of the instrument is verified by means of a solid reference standard and/or certified liquid standards.

DEFINITION

Refractive Index: the ratio of the velocity of light (of specific 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 (relative index).

APPARATUS

Light source, Raymaster, Omnirange lamp unit, includes 9-prong adapter for Na/10 Lamp, available from Valentine Instrument Co., or equivalent

Reading Lens, thermometer, focusing, Cat. No. S-81565, Sargent-Welch Scientific, or equivalent

Refractometer, Abbe precision type, range 1.3 to 1.7 for the sodium D line, capable of reading the fourth decimal place with estimations of tenths in the fifth place, Improved Precision Model 450A available from Valentine Instrument Co., or equivalent

Sodium lamp, for Omnirange lamp unit, Na/10 available from Valentine Instrument Co., or equivalent

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Thermometer, ASTM Saybolt Viscosity 17 C, range from 19-27 C, Cat. No. S-80755A, Sargent-Welch Scientific Co., or equivalent

Water bath, external circulating, capable of maintaining the indicated prism temperature constant within 0.02 C, Haake Model FKP, Cat. No. S-84883-67, Sargent-Welch Scientific, or equivalent

REAGENTS AND MATERIALS

Lens paper, optical, book form, 10 x 14 cm, Cat. No. S-44325, Sargent-Welch Scientific Co., or equivalent

Methylcyclohexane, primary standard, available from API Standard Reference Office

Monobromonaphthalene, 97% minimum purity, available from Valentine Instrument Co., or equivalent

Pipet, disposable, capillary, Pasteur, 145 mm, Cat. No. S-69647A, Sargent-Welch Scientific Co., or equivalent

Toluene, 99.9% minimum purity

Toluene, primary standard, available from API Standard Reference Office

2,2,4-Trimethylpentane, primary standard, available from API Standard Reference Office

Tubing, rubber, 7.9 mm ID, 1.6 mm wall thickness, Cat. No. S-73659, Sargent-Welch Scientific Co., or equivalent (3-m required)

PROCEDURE

The following general directions apply to all Abbé type refractometers and are intended as a guide to the analyst. For specific instructions with a particular refractometer consult the manufacturer's instruction manual.

Preparation of Apparatus

Connect the inlet and outlet ports on the refractometer to the circulating water bath with rubber tubing so that the thermometer is located in the outlet stream from the measuring prism. Fill the bath with distilled water and adjust the bath thermostat so that the temperature indicated by the thermometer at the refractometer is within 0.02 C of 20 C. Turn on the sodium vapor lamp and allow it to warm up for 30 minutes. Control the ambient room temperature so that it is within 2 degrees of the test temperature.

Open the prism case and thoroughly clean the faces of both prisms by gently wiping them with clean lens tissue saturated with toluene. Pass the tissue over the faces very lightly until they show no tendency to streak. Avoid rubbing the prisms. Loss of polish of the refracting prism will result in hazy border lines, while extensive rubbing of the illuminating prism will result in a reduction of contrast between the light and dark portions of the field.

Handle the refractometer with care and keep the exterior of it scrupulously clean at all times.

Standardization of Apparatus

Solid Standard

The refractometer can be checked with either liquids or solids of known refractive index to determine if it needs adjustment. A solid standard is usually provided with the refractometer and is convenient to use since it is not appreciably affected by temperature changes. If a solid standard is available, position the refractometer as described in the manufacturer's manual so as to accommodate it. Attach one of the polished surfaces of the solid standard to the face of the refracting prism with a small drop of monobromonaphthalene (see *NOTE*). Read the refractive index of the solid as described under *Technique*. If the reading thus obtained differs from the known value of the standard, adjust the refractometer in accordance with the manufacturer's instructions.

Liquid Standard

Liquid standards can be used separately or in conjunction with a solid standard for standardization of the instrument. It is advisable to develop a calibration curve and periodically check the accuracy of the refractometer with several liquid standards even though a solid standard is available. The liquid standards should span the range of refractive indices normally encountered. The accompanying table lists appropriate standards with their approximate indices.

Recommended Liquid Standards

Compound	Approximate Refractive Index
2,2,4-Trimethylpentane	1.39
Methylcyclohexane	1.42
Toluene	1.49

Check the refractometer for adjustment by introducing a sample of API Standard methylcyclohexane. Read the refractive index as described under *Technique* and if the value thus obtained differs from the known value of the standard, make appropriate adjustments to the refractometer in accordance with the manufacturer's instructions. Verify the accuracy of the setting by analyzing a fresh sample of methylcyclohexane and measuring its refractive index as described under *Technique*. Measure the refractive index of API Standard 2,2,4-trimethylpentane and toluene as described under *Technique*.

Calibration Curve

Construct a calibration curve as follows: Subtract the observed refractive index from the certified value for 2,2,4-trimethylpentane. Plot this value along the ordinate against the observed refractive index along the abscissa. When plotting the observed refractive index the smallest scale division should be equivalent to 0.00002 which will allow corrections to be made in the fifth decimal place. A large sheet of graph paper will be required or, preferably, expand that portion of the curve which is being used.

Subtract the observed refractive index from the certified value for toluene and plot this value against the observed refractive index in the same manner. Draw a straight line from the point representing the deviation

found for 2,2,4-trimethylpentane to zero at the refractive index of methylcyclohexane. Also draw a straight line from the same zero point to the point representing the deviation found for toluene.

Technique

Set up the instrument and clean the prisms as described in *Preparation of Apparatus*. Liquids to be tested must be free from solid particles and lint. Introduce several drops of the sample to be tested onto the horizontal face of the refracting prism using a dropping pipet or a glass rod. Take care so that the rod or pipet does not touch the face of the prism. Close the prism case securely. Allow the sample to equilibrate for several minutes. Adjust the instrument so that the field border line (n_D Line) is visible in the telescope. Adjust the focus, according to the manufacturer's manual, so that a very sharp border line between the light and dark portions of the field is obtained. Set the cross hairs exactly on the border line and read the refractive index using the scale on the instrument. If a scale illuminator switch is used, take care not to inadvertently touch other parts of the refractometer when operating it, since doing so may change the setting previously made. Make five separate settings of the cross hairs on the border line, alternating in approaching this setting from both dark and light sides of the field and read the refractive index for each setting. Record the average of these five readings.

CALCULATIONS

Correct the observed refractive index by either adding or subtracting the amount indicated by the calibration curve.

NOTE

Solid standards when placed upon the prism should not be moved. When removing them from the prism, they should be lifted off, not slid, since sliding will cause wear to the prism and possible scratching.

PRECISION

Based on 8 replicate determinations, the estimated standard deviation (esd) for the refractive index of 2,2,4-trimethylpentane was calculated to be 0.00003.

Duplicate results by the same operator should not differ by more than 0.00012 (95% probability).

TIME FOR ANALYSIS

The elapsed time for one analysis is 0.2 hour. The labor requirement is 0.15 man-hour.

REFERENCE

ASTM Methods D 1218, D 747, www.astm.org

SUGGESTED SUPPLIERS

API Standard Reference Office, Carnegie-Mellon University, Pittsburgh, PA 15213
Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60076
Valentine Instrument Co., 2184 Primrose Ave., Vista, CA 92083