Designation: C 1648 – 06

Standard Guide for Choosing a Method for Determining the Index of Refraction and Dispersion of Glass[1]

This standard is issued under the fixed designation C 1648; 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 guide identifies and describes seven test methods for measuring the index of refraction of glass, with comments relevant to their uses such that an appropriate choice of method can be made. Four additional methods are mentioned by name, and brief descriptive information is given in Annex A1. The choice of a test method will depend upon the accuracy required, the nature of the test specimen that can be provided, the instrumentation available, and (perhaps) the time required for, or the cost of, the analysis. Refractive index is a function of the wavelength of light; therefore, its measurement is made with narrow-bandwidth light. Dispersion is the physical phenomenon of the variation of refractive index with wavelength. The nature of the test-specimen refers to its size, form, and quality of finish, as described in each of the methods herein. The test methods described are mostly for the visible range of wavelengths (approximately 400 to 780μm); however, some methods can be extended to the ultraviolet and near infrared, using radiation detectors other than the human eye.

1.1.1 List of test methods included in this guide:

1.1.1.1 Becke line (method of central illumination),

1.1.1.2 Apparent depth of microscope focus (the method of the Duc de Chaulnes),

1.1.1.3 Critical Angle Refractometers (Abbe type and Pulfrich type),

1.1.1.4 Metricon[2] system,

1.1.1.5 Vee-block refractometers,

1.1.1.6 Prism spectrometer, and

1.1.1.7 Specular reflectance.

1.1.2 Test methods presented by name only (see Annex A1):

1.1.2.1 Immersion refractometers,

1.1.2.2 Interferometry,

1.1.2.3 Ellipsometry, and

1.1.2.4 Method of oblique illumination.

1.2 *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.*

1.3 **Warning**—Refractive index liquids are used in several of the following test methods. Cleaning with organic liquid solvents also is specified. Degrees of hazard associated with the use of these materials vary with the chemical nature, volatility, and quantity used. See manufacturer's literature and general information on hazardous chemicals.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

E 167 Practice for Goniophotometry of Objects and Materials[4]

E 456 Terminology Relating to Quality and Statistics

3. Terminology

3.1 *Definitions:*

3.1.1 *dispersion*, *n*—the physical phenomenon of the variation of refractive index with wavelength.

3.1.1.1 *Discussion*—The term, "dispersion," is commonly used in lieu of the more complete expression, "reciprocal relative partial dispersion." A dispersion-number can be defined to represent the refractive index as a function of wavelength over a selected wavelength-range; that is, it is a combined measure of both the amount that the index changes and the non-linearity of the index versus wavelength relationship.

3.1.2 *resolution*, *n—as expressed in power of 10*, a commonly used term used to express the accuracy of a test method in terms of the decimal place of the last reliably measured digit of the refractive index which is expressed as the negative power of 10. As an example, if the last reliably measured digit is in the fifth decimal place, the method would be designated a 10^{-5} method.

3.2 *Symbols*:

n = index of refraction

[1] This guide is under the jurisdiction of ASTM Committee C14 on Glass and Glass Products and is the direct responsibility of Subcommittee C14.11 on Optical Properties.

Current edition approved Oct. 1, 2006. Published February 2007.

[2] Metricon is a trademark of Metricon Corporation 12 North Main Street, P.O. Box 63, Pennington, New Jersey 08534.

[3] 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.

[4] Withdrawn.

TABLE 1 Spectral Lines for Measurement of Refractive Index[A]

Fraunhofer Line	A'	C	C'	D	d	e	F	F'	g	G'	h
Element	K	H	Cd	Na	He	Hg	H	Cd	Hg	H	Hg
Wavelength Nanometers	786.2[B]	656.3[C]	643.8[D]	589.3	587.6	546.1	486.1	480.0[D]	435.8	434.0	404.7

[A] From Ref **(4)**.
[B] A later reference (identification not available) lists 789.9 nm for the potassium A' line, although referring to Ref **(4)**. The Handbook of Chemistry and Physics lists 789.9 nm as a very strong line, and it does not list a line at 786.2 nm at all.
[C] The wavelength of the corresponding deuterium line is 656.0 nm.
[D] The two cadmium lines have been recognized for refractometry since Ref **(4)** was published.

ν = Abbe-number; a representation of particular relative partial dispersions

ν_D = Abbe-number determined with spectral lines *D*, *C*, and *F*

ν_e = Abbe-number determined with spectral lines *e*, *C′*, and *F′*

D = the spectral emission line of the sodium doublet at nominally 589.3 nm (which is the mid-point of the doublet that has lines at 589.0 nm and 589.6 nm)

C = the spectral emission line of hydrogen at 656.3 nm

F = the spectral emission line of hydrogen at 486.1 nm

e = the spectral emission line of mercury at 546.1 nm

C′ = the spectral emission line of cadmium at 643.8 nm

F′ = the spectral emission line of cadmium at 480.0 nm

4. Significance and Use

4.1 *Measurement*—The refractive index at any wavelength of a piece of homogeneous glass is a function, primarily, of its composition, and secondarily, of its state of annealing. The index of a glass can be altered over a range of up to 1×10^{-4} (that is, 1 in the fourth decimal place) by the changing of an annealing schedule. This is a critical consideration for optical glasses, that is, glasses intended for use in high performance optical instruments where the required value of an index can be as exact as 1×10^{-6}. Compensation for minor variations of composition are made by controlled rates of annealing for such optical glasses; therefore, the ability to measure index to six decimal places can be a necessity; however, for most commercial and experimental glasses, standard annealing schedules appropriate to each are used to limit internal stress and less rigorous methods of test for refractive index are usually adequate. The refractive indices of glass ophthalmic lens pressings are held to 5×10^{-4} because the tools used for generating the figures of ophthalmic lenses are made to produce curvatures that are related to specific indices of refraction of the lens materials.

4.2 *Dispersion*—Dispersion-values aid optical designers in their selection of glasses (Note 1). Each relative partial dispersion-number is calculated for a particular set of three wavelengths, and several such numbers, representing different parts of the spectrum might be used when designing more complex optical systems. For most glasses, dispersion increases with increasing refractive index. For the purposes of this standard, it is sufficient to describe only two reciprocal relative partial dispersions that are commonly used for characterizing glasses. The longest established practice has been to cite the Abbe-number (or Abbe ν-value), calculated by:

$$\nu_D = (n_D - 1)/(n_F - n_C) \quad (1)$$

4.2.1 Some modern usage specifies the use of the mercury e-line, and the cadmium *C′* and *F′* lines. These three lines are obtained with a single spectral lamp.

$$\nu_e = (n_e - 1)/(n_{F'} - n_{C'}) \quad (2)$$

4.2.2 A consequence of the defining equations (Eq 1 and 2) is that smaller ν-values correspond to larger dispersions. For ν-values accurate to 1 to 4 %, index measurements must be accurate to 1×10^{-4}; therefore, citing ν-values from less accurate test methods might not be useful.

NOTE 1—For lens-design, some computer ray-tracing programs use data directly from the tabulation of refractive indices over the full wavelength range of measurement.

NOTE 2—Because smaller ν-values represent larger physical dispersions, the term constringence is used in some texts instead of dispersion.

5. Precision, Bias, and Accuracy (see Terminology E 456)

5.1 *Precision*—The precision of a method is affected by several of its aspects which vary among methods. One aspect is the ability of the operator to repeat a setting on the observed optical indicator that is characteristic of the method. Another aspect is the repeatability of the coincidence of the measurement scale of the instrument and the optical indicator (magnitude of dead-band or backlash); this, too, varies among methods. A third aspect is the repeatability of the operator's reading of the measurement scale. Usually, determinations for a single test specimen and for the reference piece should be repeated several times and the resulting scale readings averaged after discarding any obvious outliers.

5.2 *Bias (Systematic Error)*:

5.2.1 *Absolute Methods*—Two of the test methods are absolute; the others are comparison methods. The absolute methods are the prism spectrometer and the apparent depth of microscope focus. These yield measures of refractive index of the specimen in air. In the case of the prism spectrometer, when used for determinations of 1×10^{-6}, correction to the index in vacuum (the intrinsic property of the material) can be calculated from the known index of air, given its temperature, pressure, and relative humidity. The accuracy of the apparent depth method is too poor for correction to vacuum to be meaningful. Bias of the prism spectrometer depends upon the accuracy of its divided circle. The bias of an index determination must not be greater than one-half of the least count of reading the scale of the divided circle. For a spectrometer capable of yielding index values accurate to 1×10^{-6}, the bias must be not greater than 5×10^{-7}. Bias of the apparent depth method depends on the accuracy of the device for measuring the displacement of the microscope stage; it is usually appreciable smaller than the precision of the measurement, as explained in 7.6.

5.2.2 *Comparison Methods*—All of the comparison methods rely upon using a reference material, the index of which is known to an accuracy that is greater than what can be achieved by the measurements of the given method itself; therefore, the bias of these methods is the uncertainty of the specified refractive index of the reference material, provided that the instrument's scale is linear over the range within which the test-specimen and the reference are measured. The bias introduced by non-linearity of the scale can be compensated by calibrating the scale over its range with reference pieces having indices that are distributed over the range of the scale. A table of scale-corrections can be made for ready reference, or a computer program can be used; using this, the scale reading for a single reference piece is entered and then corrected indices are generated for each scale reading made for a set of test specimens. For a single measurement, scale correction can be made by first measuring the test specimen and then measuring the calibrated reference piece that has the nearest index. In this case, the scale is corrected only in the vicinity where the readings are made.

5.2.3 *Test Specimen*—Deviations of a test specimen from its ideal configuration can contribute a bias. For 1×10^{-6} refractometry, specimen preparation must be of the highest order and specimens are tested for acceptability for use. Bias introduced by a test specimen varies in its manifestation with the type of test method and nature of the deviation from ideal. This consideration is addressed in the descriptions of individual test methods.

5.3 *Accuracy*—The limiting accuracies of the several test methods are given. The operator must estimate whether and how much a given test measurement deviates from that limit. The estimate should take into account the observed uncertainty of identifying where to set on the optical indicator (see 7.6, for example) as well as the precision of such settings. Specific considerations are given in the descriptions of the test methods.

NOTE 3—The Subcommittee did not conduct an Inter-laboratory Study (as normally required) to quantify the Precision and Bias of Methods discussed in this Standard. The cited accuracies of the test methods are based on experience.

TEST METHODS

6. Becke Line (Method of Central Illumination)

6.1 *Summary of the Method*—Not-too-finely ground particles of the glass for testing are immersed in a calibrated refractive index oil and are examined with a microscope of moderate magnification. With a particle in focus, if the indices of the oil and the glass match exactly, the particle is not seen; no boundary between oil and glass is visible. If the indices differ, a boundary is seen as a thin, dark line at the boundary of the particle with either the particle or the oil appearing lighter. The line appears darker as the indices differ more; however, which material has the higher index is not indicated. When the focal plane of the microscope is moved above or below the plane of the particle (usually by lowering or elevating the stage of the microscope), one side of the boundary appears lighter and the other side appears darker than the average brightness of the field. When the focus is above the plane of the glass particle, a bright line next to the boundary appears in the medium of higher index. This is the "Becke line"; conversely, when the focus is below the plane of the particle, the bright line appears in the medium of lower index. Successive changes of oil, using new glass particles, lead by trial and error to a bracketing of the index of the particle between the pair of oils that match most closely (or to an exact match). Visual interpolation can provide resolution to about one fourth of the difference between the indices of the two oils. The physical principle underlying the method is that of total internal reflection at the boundary, within the medium of higher index. This is illustrated by a ray diagram, Fig. 1*(a)*. Visual appearances are illustrated in Fig. 1*(b)*, Fig. 1*(c)*, and Fig. 1*(d)*, where different densities of cross-hatching indicate darker parts of the field of view. Although calibrated indices are provided for the C- and F-lines, enabling an estimate of a dispersion-value, it must be taken not to be very accurate.

6.2 *Advantages and Limitations*—This method uses the smallest amount of specimen-material and it has the simplest and least expensive method of sample-preparation. Costs of apparatus and materials, too, are moderate, as is the time needed to make a determination; however, the accuracy of the method is limited to about 5×10^{-4} (index-values are less accurate for $n < 1.40$ and $n > 1.70$).

NOTE 4—A related test method, the method of oblique illumination, is described in Annex A1.

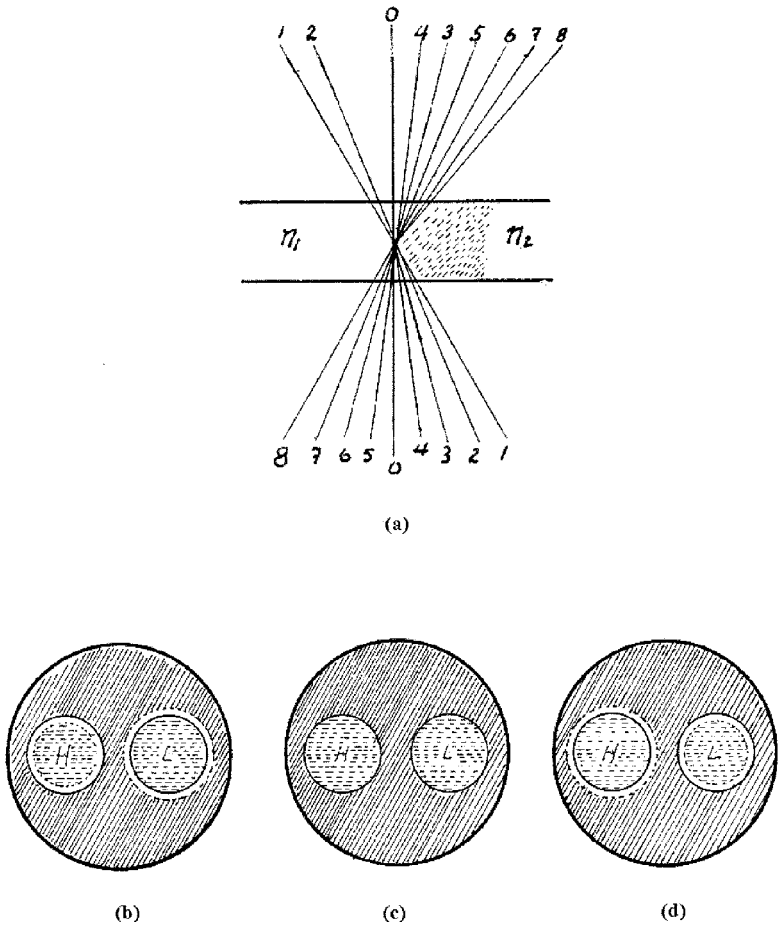
NOTE 5—Because the test specimen is very small, the Becke line method can be used to determine the refractive index of highly absorbing glasses. For example, for a 2-mm thick piece of Corning-Kopp color filter CS 7-58, the maximum spectral transmittance is about 1×10^{-4} (optical density 4.0); it occurs near 589 nm. Its refractive index was determined to 1×10^{-3} by the Becke line method. Appreciably higher absorption can result in there being too little distinction when the indices of specimen and liquid are nearly alike. In this case, the bracketing liquids that can be identified will be more widely separated. Use the mean of their given indices and assign an appropriately larger uncertainty to the result.

6.3 *Apparatus and Materials*:

6.3.1 *Microscope*—Use a microscope having a total magnification of at least 80× that has a sub-stage condenser with a variable-aperture iris diaphragm. (A 10× objective lens and a 10× ocular are very satisfactory.)

6.3.2 *Microscope Slides and Cover Glasses*—Use standard glass microscope slides, 1×3-in., 1-mm thick, and microscope cover glasses, 18 mm (preferred) or 22 mm^2 and 0.35-mm thick (#1½).

6.3.3 *Bandpass Filters*—Narrow spectral bandpass filters, about 1-nm FWHM (full width at half maximum transmittance), should be used (measurement with white light reduces the accuracy of a result). These can be commercial interference filters. Owing to the bandwidth of about 10 nm, the wavelengths of the transmittance maxima of the filters need not fall at exactly the wavelengths of the spectral lines that are specified for determining dispersion-numbers. For the Abbe ν-value, standard interference filters with nominal peak wavelengths of 490 nm, 590 nm, and 650 nm or 660 nm would work well. The filter should be mounted close to the substage condenser assembly. This will avoid focusing dirt or surface defects of the filter onto the plane of the specimen.



(a) ray diagram showing the principle of the method, $n_1 < n_2$; *(b)* appearance of Becke lines for specimens of higher *(H)* and lower *(L)* refractive index than that of the immersion liquid with the microscope-focus above the plane of the specimen-particles; *(c)* in the plane of the particles; *(d)* below the plane of the particles

FIG. 1 Becke Line

6.3.4 *Calibrated Immersion Oils*—Sets of calibrated index oils are available with indices over the range 1.300 to 2.31.[5] Partial sets, by preset groupings or by custom selections, can be purchased according to particular need. The label of each bottle has the index for the sodium D-line at 25°C, standardized to 2×10^{-4}, the temperature coefficient of index, and the indices for the hydrogen C and F-lines. Liquids with indices above 1.70 require special handling, as taught by the manufacturer. The oils are supplied in 7.4-cc (¼ fl oz) bottles; the caps have small glass rods for transfer of fluid by the drop. The refractive indices of the oils depend on their temperature; therefore, store the oils at room temperature and measure the temperature at the time of testing. Temperature-corrections of the indices of the oils must be made.

NOTE 6—"Standardized to" is the manufacturer's statement of the accuracy of the stated index of n_D at 25°C. Standardization to 2×10^{-4} is for the range 1.300 to 1.700. Larger tolerances are specified for lower and higher range oils.

6.3.5 *Mortar and Pestle*—A small mortar and pestle of agate or of a hard ceramic is used to prepare the specimens for observation.

6.3.6 *Thermometer*—A thermometer that is sensitive and accurate to 0.5°C is needed.

6.4 *Hazards*—The immersion oils are somewhat toxic. They should be used in a well ventilated space, and contact with the skin should be avoided. The latter is particularly important for the high index liquids ($n > 1.70$). Manufacturer's guidelines should be followed.

6.5 *Specimen Preparation*—Use a small piece of the glass to be tested. Clean it with alcohol and water (or other solvent, if necessary). Rinse it with water and dry it with a tissue. One or two cubic millimetres will be more than enough for testing with a dozen or so oils; therefore, enough to complete a test, even of an initially completely unknown sample. Put the sample into the mortar and crush it into small pieces by pressing down with the pestle. Use a rocking motion and do not slide the pestle against the mortar as specimens for measurement should not be too small. A text specifies that they should pass through a 100-mesh sieve and be held back by a 170-mesh screen; however, screening is not necessary: the appropriate size will be learned by a few trials.

6.6 *Procedure*—Transfer about 10 particles of glass to the microscope slide using a spatula with a small tip. Three piles can be placed on a slide, spaced about 20-mm apart, to speed the course of measurements. Spread the particles over an area

[5] Cargille Laboratories, Inc., 55 Commerce Road, Cedar Grove, NJ 07009-1280, Tel. 973-239-6633, www.cargille.com

about 10-mm diameter and remove any exceptionally large particles. Lay a cover slip on the spread and dispense one drop of a calibrated index oil by touching the tip of the rod to the edge of the cover slip and the surface of the slide. (Second or third drops, applied to other edges, might be needed for adequate immersion of the particles.) Capillary action will draw the liquid in and immerse the glass particles. Place the slide on the stage of the microscope. Close the iris diaphragm appreciably. Bring a particle into focus and adjust the iris diagram and the focus until the boundary between particle and oil is sharp (Fig. 1*(c)*). Note the darkness and breadth of the particle-oil boundary for estimating whether a small or a large change of index for the next oil is needed. Raise the focal plane of the microscope above the plane of the particle while observing the formation of the bright Becke line and its motion into one medium, whether glass or oil. Repeat these observations for several particles and act according to the indication of the majority. For the next trial, choose an oil with index closer to that of the glass. Repeat the procedure until a match is achieved or until the two closest (bracketing) oils are found. If it is desired to have an estimate of the dispersion of the glass, repeat the procedure with bandpass filters for the C and F-lines.

6.7 *Calculation*—Estimate the index by interpolating between the indices of the bracketing oils using relative contrasts of the boundary when the particle is in focus. The estimate can be as good as one-fourth of the step of index between the two oils. The estimate must also be whether to assign the exact index of one oil (for a close match) or to assign the value of the nearest quarter-step. Multiply the difference between 25°C and the temperature of the oil (that is, room temperature) by the temperature coefficient of index-variation and add (algebraically) to obtain the correct index. Because the rate of variation of index is very much larger for the oils than it is for glass, no adjustment is needed for the glass.

6.8 *Precision and Bias*—Precision can be slightly better than one-fourth of the size of the step between adjacent oils of a set. Bias is limited to the stated adjustment (“standardization”) of index (that is, the accuracy of the index) of the oils of a set. Manufacturer’s instructions must be followed to preserve the integrity of accuracy. Cross-contamination of the applicator rods must be avoided. Bottles must be capped except for the brief time that a transfer of liquid is being made.

7. Apparent Depth of Microscope Focus (the Duc de Chaulnes’ Image Displacement Method)

7.1 *Summary of the Method*—This method has poor accuracy; for example, about 0.05 for a glass with $n = 2.00$; however, Miller (**1**)[6] describes technique and calculation that can provide accuracy of 0.002 for a glass with $n = 1.50$. The accuracy would be poorer for higher index glasses. The utility of the method lies in the relative ease of specimen-preparation and in its convenience for a quick test of higher index glasses ($n > 1.70$) when higher index calibrated oils are not at hand or are not wanted to be used; therefore, it can be a useful tool where experimental melting of higher index glasses is being done and quick results are desired. Because of the poor accuracy, the method is not suitable for determining dispersion. The principle of the method is illustrated in Fig. 2*(a)* and Fig. 2*(b)*. The specimen is a flat parallel-sided piece of glass, both sides polished. Marks are placed on top and bottom surfaces and the piece is examined with a moderate-power microscope. The mark on the top surface is brought into focus and an index of the elevation of the specimen relative to the objective lens is recorded. Then, the mark on the bottom surface is brought into focus and the index of the elevation of the specimen is again recorded, thus providing a measure of the displacement of the specimen relative to the position of the objective lens. The simplified, often used, but rather inaccurate calculation of the refractive index of the glass n_g is given by:

$$n_g \approx t/d \tag{3}$$

where:

t = thickness of the specimen, and
d = displacement of the stage of the microscope.

7.1.1 The derivation of Eq 3 and explanation of the sources of error are given is Appendix X1.

7.2 *Apparatus and Materials*:

7.2.1 *Microscope*—The microscope should have a total magnification of about 100× and the objective lens should provide about 10× of that. (See Appendix X1 for discussion of effect of magnification of the objective lens.) The stage should have provision for fine adjustment of its elevation.

7.2.2 *Marker*—Use a felt-tipped marker capable of making a very thin (in the thickness dimension) line on the polished glass surface.

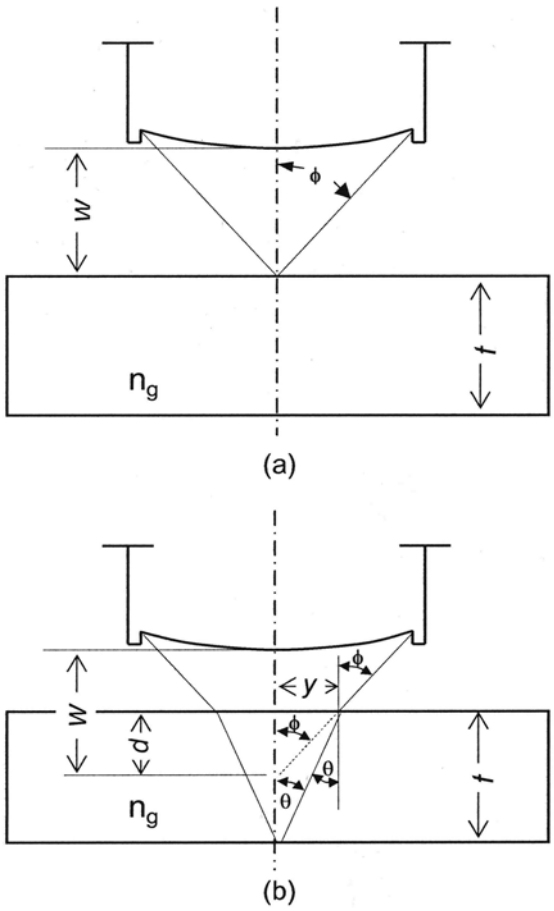
7.2.3 *Narrow Bandpass Filter*—Use a narrow bandpass filter, such as described in 6.3.3, chosen for either n_D or n_e.

7.2.4 *LVDT or Dial Gauge*—Either a linearly variable differential transformer (LVDT) or a dial gauge is used to measure the vertical displacement of the stage of the microscope relative to the objective lens. Consult the manufacturer’s instructions for mounting the LVDT and for measuring displacement with it. A dial gauge mounted on a stand can be placed with its axis vertical and the tip of its shaft on and near the edge of the stage of the microscope. The dial gauge should be divided into 0.01-mm markings, spaced such that interpolation to 0.002-mm can be made. The dial gauge should be tapped gently at each setting and an electrical vibrator (buzzer) should be fastened to the base of the mounting of the dial gauge. These are to overcome sticking of the gauge which occurs because motion in adjusting the focus is very slow. A step-down transformer and momentary contact switch are needed for operating the buzzer.

7.2.5 *Micrometer Caliper*—A micrometer caliper capable of being read to 0.002-mm by interpolation must be used for measuring the thickness of the specimen.

7.3 *Specimen Preparation*—The cross-section of the specimen should be large enough for convenient grinding and polishing flat and parallel surfaces: 21 by 2 cm (2 cm square) or diameter is satisfactory. Measure the thickness of the specimen to an accuracy of 0.002 mm. Clean the surfaces with alcohol and distilled water. Mark a line on one surface, near the center of the piece, and then a line on the other surface such

[6] The boldface numbers in parentheses refer to the list of references at the end of this standard.



(a) focus on mark on top of specimen; *(b)* focus on mark on bottom surface of the specimen, with ray diagram and definition of symbols

FIG. 2 Apparent Depth of Microscope Focus

that an X is seen when viewed perpendicularly. Make the mark as thin as possible but still easily seen with the microscope.

7.4 *Procedure*—Position the specimen on the stage such that the axis where the marks cross is at the center of the field of view. Focus onto the mark on the top surface and record the elevation of the stage as indicated by the LVDT or dial gauge. Repeat at least five times; eliminate obvious outliers; and calculate the average of several readings. Raise the stage to bring the mark on the bottom surface into focus and record the elevation, repeating as above. Tap the dial gauge or use the vibrator to home-in the dial gauge at each setting.

7.5 *Calculation*—Use Eq 3 for a first approximation of the index. Use Eq 4 as a refinement that eliminates the error from replacing tangents of angles with their sines (see Appendix X1).

$$n_g = \{(t/d)^2 - NA^2\,[(t/d)^2 - 1]\}^{1/2} \quad (4)$$

where:

NA = numerical aperture of the objective lens,
t = thickness of the specimen, and
d = displacement of the stage of the microscope.

NOTE 7—The significance of using this correct formula is illustrated by these examples. *(1)* For t/d = 1.60, by Eq 3, n_g = 1.6, and by Eq 4, n_g = 1.50; *(2)* for t/d = 2.19, by Eq 3, n_g = 2.19, and by Eq 4, n_g = 2.00.

7.6 *Precision and Bias*—Precision must be determined by the operator for each test, as it can vary with thickness of the specimen and its refractive index, and on the ability to repeat the focusing on a mark. Precision can be improved by making several replicate measurements and by a using a microscope objective lens with higher magnification (Note 8). Also, for a lens of given magnification, precision can be greater with an objective lens that has a higher numerical aperture. The accuracy of determining the displacement is better with a calibrated LVDT than with a dial gauge. Bias depends on the accuracy of the gauges used. Lack of parallelism of the faces of the test specimen will introduce a small bias. Bias will ordinarily be smaller than the errors from imprecision of setting the focus.

NOTE 8—A lens of higher magnification will have a shorter working distance; therefore, the thickness of the specimen will affect how high a magnification lens can be used. See Appendix X1.

8. Critical Angle Refractometers (Abbe Type and Pulfrich Type)

8.1 *Summary of the Method*—The principle of critical angle refractometry is illustrated in Fig. 3. It was first realized by Abbe. The modification by Pulfrich is the (near-) linearization of the measurement scale of the refractometer as a function of

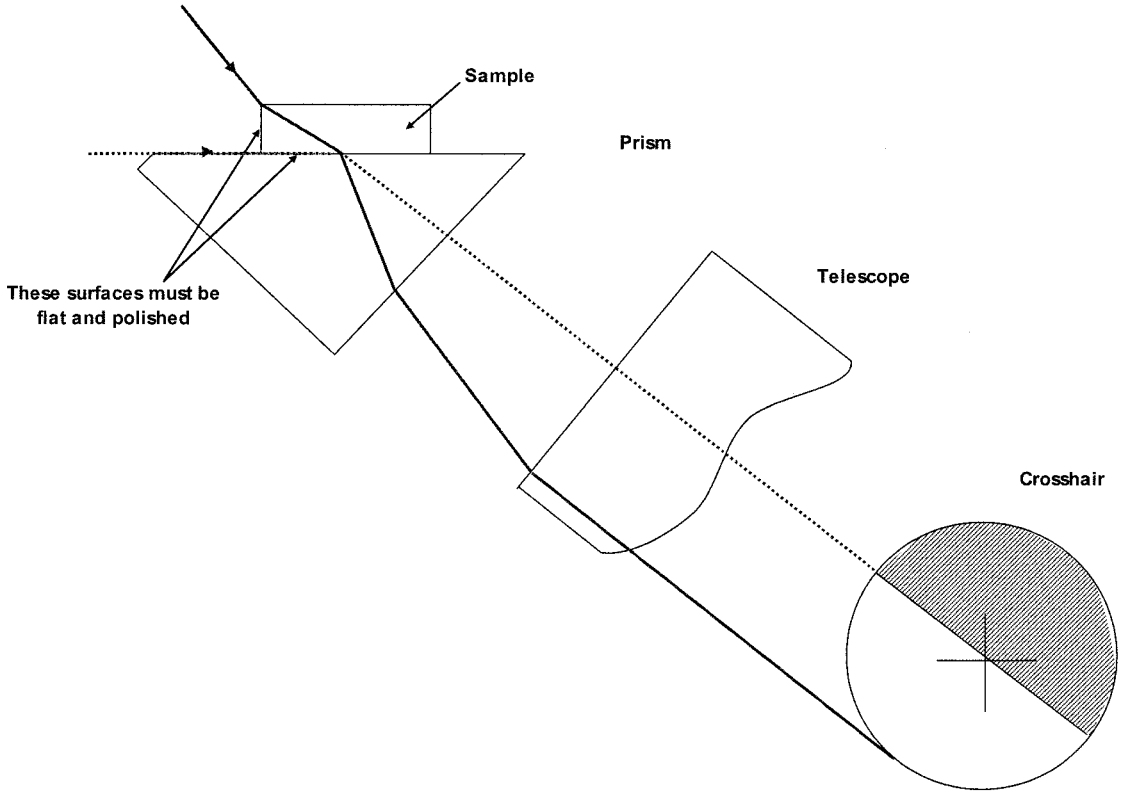


FIG. 3 Principle of Critical Angle Refractometers

the index of the test specimen. In order to cover a very wide range of indices, measurement prisms having different indices are used. The index of the measurement prism must be higher than that of the test specimen. Excellence in the preparation of a test specimen is critical in order to realize accuracies in the fifth decimal place (Note 9). Straat and Forest (**2**) analyze accuracy requirements for fifth decimal place refractometry. Tilton (**3**) provides valuable instruction as well. A glass specimen with an optically flat polished surface is held onto the prism-face by capillary attraction of a coupling liquid which must have a higher index than that of the glass. The interface is illuminated by a spectral lamp such that rays fall at grazing incidence along the interface and at a small range angles above grazing. They enter the glass through a polished face that is perpendicular to the contact face. The back face of the prism is viewed with a simple telescope that focuses emerging rays onto cross hairs; these are viewed through an eyepiece. The light that is incident at grazing incidence enters the prism at the critical angle θ_c given by:

$$\theta_c = \sin^{-1}(n_g/n_p) \quad (5)$$

where:

n_g and n_p = refractive indices of the glass specimen and the measurement prism, respectively, for the wavelength of the spectral line.

8.1.1 Light incident from above grazing incidence enters the prism at angles less than the critical angle. Thus, the field viewed through the telescope appears to be divided by a more-or-less sharp boundary, light on one side and dark on the other. The prism is rotated to bring the demarcation line into coincidence with the cross hairs. A scale related to the angular rotation of the prism (or of a mirror located between the prism and the telescope) is read and converted to the refractive index of the glass by reference to tables provided by the manufacturer. Before measuring new test specimens, the scale is first checked with one or more reference specimens of excellent optical finish and known refractive index; if needed, either an adjustment is made to the scale by shifting the cross hair slightly, or by rotating the prism relative to the scale if need be, in accordance with the manufacturer's instructions. If the error of the scale reading is small, it may be used as a correction without making mechanical adjustments. See 5.2.2 concerning scale corrections

NOTE 9—The Bausch & Lomb Precision Refractometer, a Pulfrich-type instrument, is no longer manufactured commercially; however, a great many of these instruments are still in use. The Reference Manual provided with the Bausch & Lomb Precision Refractometers is a very good guide for the preparation of glass specimens and for measurement procedures (although it is obsolete in its information on spectral lamps).

8.2 *Apparatus and Materials*:

8.2.1 *Refractometer*—A commercial Abbe or Pulfrich refractometer with calibrated reference test pieces.

8.2.2 *Coupling Liquids*—The B&L instruction manual specifies the requirements for the coupling liquid that attaches the test specimen to the measurement prism: "The first criterion for choice of liquid is that its index be greater than that of the sample being read. The second is that its index be fairly well removed from that of either prism or sample." The index of the liquid may lie between those of the two glasses, or it may be higher than that of the prism, but the intermediate choice is

preferable. A part of the procedure is to view the interference fringes set up within the liquid layer between the two glasses; the second criterion is intended to ensure good visibility of the fringes. Three liquids suggested in the B&L manual are Methylene Iodide, $n_D = 1.74$; 1-Bromo-Naphthalene, $n_D = 1.66$; and Anise Oil, $n_D = 1.55$. Other liquids can be selected from commercial sets of calibrated oils.

8.2.3 *Spectral Lamps*—Spectral lamps of several elements or combinations of elements are available commercially. A mercury-cadmium lamp provides three spectral lines (F', e, and C'). Provided that prism indices are known, measurements can be made through the visible spectrum from Hg, 404.7 nm to K (Potassium), 769.9 nm. Table 1 lists eleven spectral lines recommended for refractometry (report of the International Commission of Optics (**4**)). When using dim lines or those near the ends of the visual range, it may be helpful to use an isolating filter to reduce stray brightness in the field of view.

8.3 *Specimen Preparation*—Dimensions of a specimen are not critical. A typical size is 1 cm wide by 2 cm long by 2 to 3 mm thick. Pieces as small as one-half these values in width and length can be used. Two surfaces, a large flat and an end, must be polished, nearly optically flat, and nearly perpendicular. Gunter and Kobeissi (**5**) show that the angle should be 90° or obtuse up to 91°. The other surfaces may be matte. The flatness of the large face should be within one fringe of green light, tested with a small optical flat (see Note 10 and 8.4). Instead of a polished end, a fine matte end-face may be used at the cost of the loss of some light (**3**). Specimen preparation may be accomplished more easily this way. It is imperative that the edge of intersection between the polished face and the end toward the light source be sharp in order to achieve the limiting accuracy of a given instrument (Note 10).

NOTE 10—This is best accomplished with pitch polishing. Felt polishing tends to roll the surface at the edges, and flatness to three fringes is what is customarily obtained, but care is required even for this. The effect is to reduce the accuracy of a measurement slightly. This can be estimated by noting the precision of setting on a somewhat diffuse demarcation line.

8.4 *Procedure*—Start by cleaning the faces of the prism and the specimen meticulously, using a soft tissue wetted with alcohol or xylene. Do not use acetone or similar solvents. Dry the surfaces and be certain that no grit, fine dust, or lint remain. Repeat this cleaning each time a new test piece or a calibrated reference piece is to be mounted. Put a small drop of coupling liquid on the polished face of the test specimen and press the specimen onto the prism. Squeeze the liquid out and remove any excess with a tissue. Minimize sliding of the specimen on the face of the prism: scratching of a prism-face severely affects the sharpness of the edge between dark and bright areas of the field viewed by the telescope. Be especially careful to remove all liquid at the edge of the specimen that is toward the light source. Adjust the lamp to illuminate the full end of the specimen. A large and diffuse source is desirable. The B&L Precision Refractometer has an auxiliary lens in the telescope tube. When rotated into place, the specimen-prism interface is in focus, and interference fringes between the prism and the specimen can be examined. "It is helpful to see a few of these fringes, indicating good mounting." (**3**). Producing a slight wedge in the liquid is recommended. Fringes running parallel to the direction of the light beam indicate a wedge in the vertical direction, and this will not affect the indicated refractive index; however, vertical fringes indicate a wedge in the direction of that of the light beam. This can introduce an error of the indicated index. "For viewing fringes in the exit pupil of the telescope, the tolerance is always ⅓ fringe of yellow light" for accuracy of 1×10^{-5} (**3**). When the specimen is suitably mounted, rotate the measurement prism to bring the demarcation line into view and center it on the cross hairs. If the line is not sharp, make the best estimate possible of the middle of the transition region and set there. Diffuse demarcation lines result from a scratched prism-face, from a liquid wedge, from inhomogeneities (scatterers) in the specimen, and from a specimen surface that is not flat enough (which introduces a liquid wedge). Read the scale and translate the reading into a value of refractive index. Always clean and dry the face of the measurement prism at the end of the measuring session. Place a double-layer of dry tissue on the surface and close the "illuminating prism" over it.

8.5 *Precision and Bias*—Precision depends on the sharpness of the demarcation line and how well the operator can reset to that line. With a good line, the precision can be as good as the least count that the scale can be read (Note 11). Several repetitions of the setting should be made, reading the scale for each and averaging for the best estimate of the correct setting. Bias depends upon how well the instrument has been adjusted with the calibrated reference pieces. In principle, accuracy to 1×10^{-5} is possible, but claimed limiting accuracies of commercial instruments are 3×10^{-5} (a Pulfrich refractometer) and 4×10^{-5} (an Abbe refractometer) for measured indices of glass near 1.5 (Note 12).

NOTE 11—"Least count" means the limit of readability with visual interpolation between adjacent scale divisions. This is about one fifth of a division of the scale of the B&L Precision refractometer.

NOTE 12—Many commercial Abbe-type instruments are intended only for measuring liquids; their accuracies are not as good. For measuring glass, a commercial instrument should be specified as suitable for that purpose and its accuracy should be stated.

9. The Metricon[2] System

9.1 *Summary of the Method*—The Metricon system,[2] too, is critical angle refractometry and Eq 5 applies. The principle of the method is illustrated in Fig. 4. Differing from the Abbe-type refractometers, illumination of the interface between the test specimen and the measurement prism is from within the prism, as illustrated in Fig. 4*(a)*. The accuracy is about 1×10^{-4}, but with special configuration, $\pm\ 1\times10^{-5}$ can be achieved, contingent on using a reference piece with refractive index that is known to a higher degree of accuracy. The light sources are lasers with small divergences and very small diameter beams. Multiple sources are available by custom configuration. For other than standard configuration, the manufacturer must be consulted; construction of the system might be the responsibility of the user. Advantages of the system are: *(1)* specimen preparation is relatively easy; *(2)* detection is by a small solid-state photosensor, so extension to near infrared measurements is possible; and *(3)* the apparatus is readily adapted to enclosure in a thermo-regulated chamber, so the temperature coefficient of variation of index can be measured. The polished

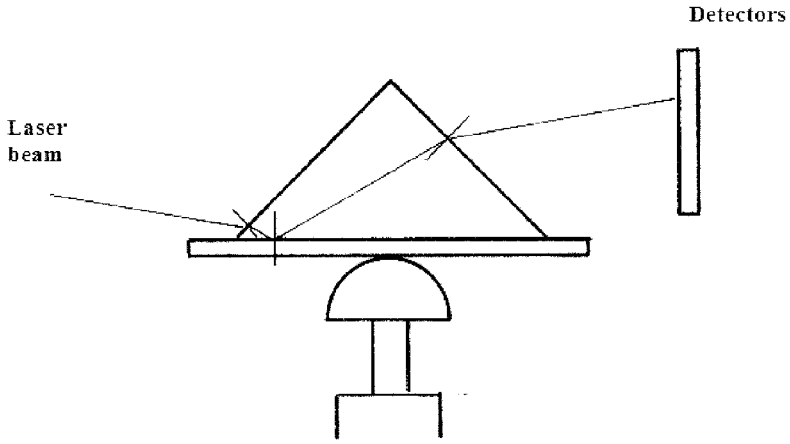


FIG. 4 Principle of Metricon System

face of the test specimen is pressed against the surface of the measurement prism and held there by a pneumatic piston. The measurement is carried out under computer control and the identification of the critical angle is determined automatically from the record of the response of the photodetector as a function of the angle of rotation, as shown in Fig. 4*(b)*. Scanning the angle of incidence of the laser beam on the prism-specimen interface is by a stepper motor. A choice of step-size can be made according to the desired accuracy.

NOTE 13—The Metricon[2] system is suitable for determining the index of thin and thick films. Thick films are treated in essentially in the same way as are bulk specimens.

9.2 *Apparatus and Materials*—Commercial Metricon[2] system with laser (HeNe) and prism suitable to the index range of interest. Consult the manufacturer on using other lasers and prisms (Note 14). Calibrated reference pieces, suitable for the index range covered by a given prism, are necessary.

NOTE 14—A standard prism supplied with the instrument covers the index range 1.3 to 1.8. Precision with this prism is poor and accuracy is from 5×10^{-4} to 1×10^{-3}. Standard prisms that yield accuracy of 1×10^{-4} cover relatively small ranges of index.

9.3 *Specimen Preparation*—The specimen must have one polished surface, flat to within one fringe of green light when tested with an optical flat. Felt polishing, which rolls off the near the edges of a specimen, is satisfactory because the prism is not contacted by any but the central very flat portion. A specimen size of 1 by 1 cm (1 cm square) is usable, but sizes of 1 by 2 cm or 2 by 2 cm are recommended for ease of handling. Glass specimens should not exceed 1-mm thick for good coupling to the prism.

9.4 *Procedure*—Carefully clean the contact surface of the prism and that of the specimen. Be sure that no grit, dust, nor lint remains. Manually center the specimen on the prism-face and activate the pneumatically activated coupling head (Fig. 4*(a)*). Adjust the pressure for good optical contact, but be careful not to press excessively hard. Rotate the prism-assembly to have the laser beam enter near the corner of the prism. Take special note of the critical contact area between specimen and prism (Fig. 4*(a)*). It is close to the corner of the prism, near where the laser beam enters, and it is not at the center of the prism-face. Start the scan under computer control. Repeat the measurement several times and calculate the average value of index that is obtained. Similarly, measure a reference piece with index appropriate to the chosen prism or as near to that of the specimen as available. Calculate the average of several repetitions and use the result to correct the indicated index of the specimen. Clean and dry the prism-face carefully after the last measurement.

9.5 *Precision and Bias*—Determine the precision of a set of measurements by repetition. The bias is limited to the accuracy of the index of the reference piece used. The accuracy is about 1×10^{-4}, but with special configuration, $\pm$ 1×10^{-5} can be achieved, contingent on using a reference piece with index known to a higher accuracy.

10. Vee-Block Refractometers

10.1 *Summary of the Method*:

10.1.1 The principle of vee-block refractometry is illustrated in Fig. 5. The vee-block is made of optical quality glass of known refractive index for all of the wavelengths of interest. The open vee has polished faces set at 90°. When a specimen having faces at 90° is inserted into the vee, using a coupling liquid, the collimated beam from a spectral lamp is deflected by an amount and in a direction determined by the relative refractive indices of the vee-block and the specimen. Light passing through the vee-block below the apex of the vee provides the reference from which the deviation angle is measured (Fig. 5). Provision is made to measure the angular deflection and this is converted to the refractive index of the specimen. Determinations for different wavelengths are possible. The accuracy can be better than 1×10^{-5}. Particular merits are the simplicity of specimen preparation and the speed of conducting a test. Although large specimens are desirable to provide more light, claim is made that a specimen as small as a couple of mm^2 in cross-section can be measured with good accuracy. To cover a wide range of indices, multiple prism-blocks usually are used, when the cost of a refractometer system can be large.

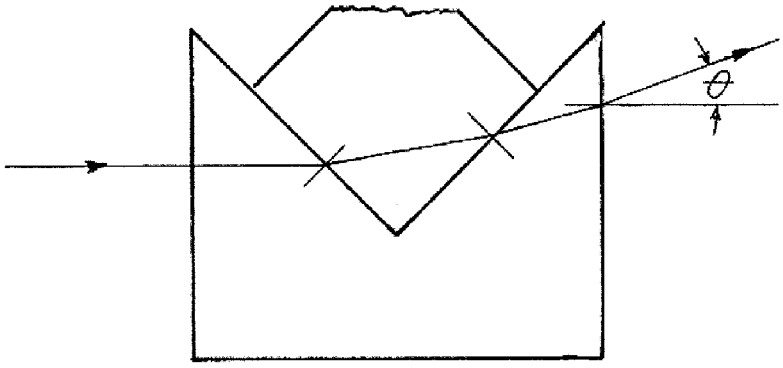


FIG. 5 Principle of Vee-Block Refractometers

10.1.2 Vee-block refractometers have been realized in several ways. Grauer (**6**) describes a system that can be adapted for measurement over a wide range of indices with a single vee-block, but it is preferable to use several vee-blocks for covering a wide range of indices. For small deviations, the linear displacement along the scale of the filar eyepiece of the observing telescope provides the magnitude of the angle by the approximation ϕ = tangent ϕ. The Grauer principle is realized in refractometers using variations on the methods of illumination and observation. Simmons and Potter (**7**) describe a system in which a mirror that can be rotated about a horizontal axis returns the beam to the collimator/telescope. The calibrated angle of rotation of the mirror to center the returned image on the fiducial cross hairs of the telescope provides the measure for calculating the index of the specimen. Variants of the Grauer system are used for production control and product assurance, where a large collection of vee-blocks might be maintained for testing a similarly large number of different product glasses.

10.1.3 The Chance design of vee-block refractometer was realized commercially as the Hilger-Chance refractometers, manufactured by Hilger and Watts, Ltd.[7] This design provides for measuring over a range of angles from about 30° below the horizontal to about 28° above. A single vee-block can cover a range of indices from 1.40 to about 1.85; however, for greatest accuracy it is better to use a vee block that has an index nearer that of the specimen, thereby reducing the amount of deviation. Accuracies to 1×10^{-5} are possible except at extremes of the range.

10.2 *Apparatus and Materials*—A vee-block refractometer and a set of refractive index liquids. The liquids are for coupling the finely ground matte surfaces of a specimen to the vee-block. A reasonably close match of the liquid to that of the specimen is necessary to minimize scattering and consequent fuzziness of the image. For very high index glasses, where good high-index liquids that are a close enough match might not be available, the quality of the specimen's surface must be a semi-polish.

10.3 *Specimen Preparation*—A specimen is to have one angle between adjacent faces very close to 90°. Deviation up to 5 arc-min is permissible, provided that two pieces having exactly complementary angles are used. Then, the liquid wedges are in opposite sense, and the average of the indices determined for the two is the true value for the specimen. A block of glass is ground flat on one surface. The block is then divided with a right-angle saw cut. The two flat, ground surfaces are held together and the sawn ends are ground to form a continuous surface. This produces the required complementary angles of the corners that fit into the apex of the prism opening. Another way to prepare a test specimen is to saw a prism of rectangular cross-section. The four surfaces of the prism are ground smooth. Provided that each of the four corner-angles is within 5 arc-s of 90°, measuring the deviations for each of the four edges and averaging the four measurements yields the true index of the specimen. Use a very fine-grit grinding abrasive; 400-mesh aluminum oxide is good.

10.4 *Procedure*—Put a few drops of (closely) matching index liquid on the first flat face of one piece of the specimen and press it against the surface of the prism that is away from the spectral light source. Slide it down carefully to contact the other face of the prism and add some coupling liquid to that interface. In the case that the two faces are not exactly perpendicular, a small wedge of liquid is formed between the specimen and the other face (nearer the light source) of the vee-block. Read the deviation of the transmitted beam from the reference position. Repeat with the other piece of the specimen. A liquid wedge produced this time will be in the opposite sense from the first. For each piece, repeat the readings at least five times and calculate an average displacement for conversion to index. Follow a comparable procedure when measuring a block with four edges. Be sure that the faces of the vee-block prism are scrupulously clean and free from any grit, dust, or lint, before inserting pieces of the specimen. Clean and cover at the end of the measurement session. Scratched prism faces hinder precise observation of the location of the image of the slit.

10.5 *Calculation*—Tables of refractive index of the test specimen for measured displacement angles are provided for each vee-block prism of the Hilger-Chance Refractometer.

[7] Hilger and Watts vee-block refractometers are no longer available commercially. Many are still in use.

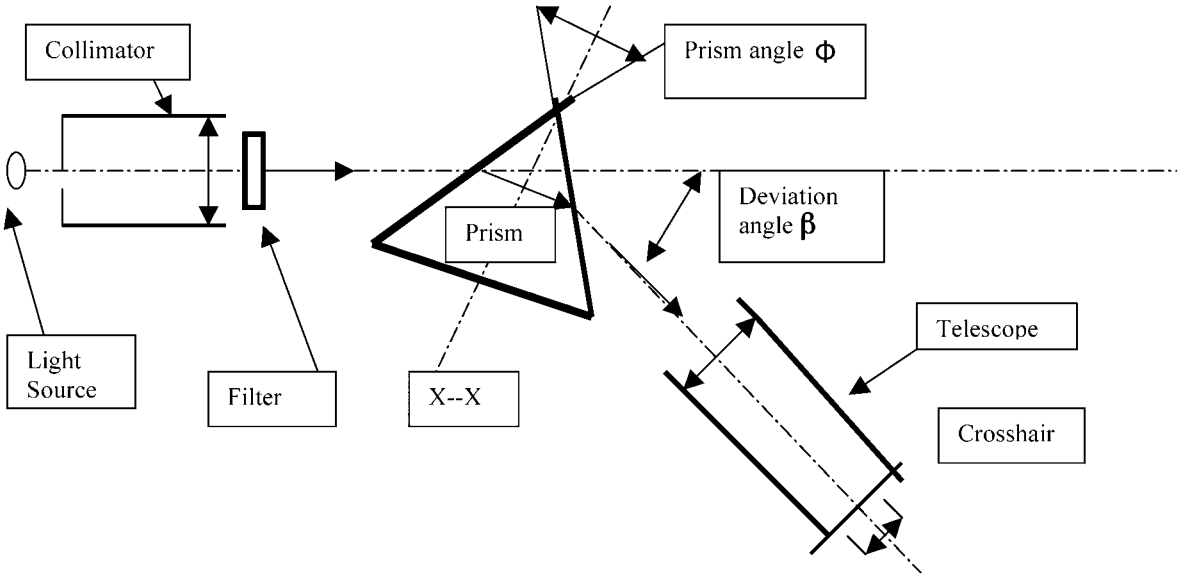


FIG. 6 Principle of Prism Spectrometer

Calculate the average of the indices determined for the two pieces of the specimen. The basic equation of the Grauer refractometer is:

$$n_g = \phi/2 + n_p \quad (6)$$

where:

n_g = refractive index of the test specimen,
n_p = index of the vee-block prism, and
ϕ = angle of deviation of the refracted beam, measured in radians.

10.5.1 The tangent of ϕ is the displacement measured by the filar eyepiece of the viewing telescope divided by the focal length of the objective lens of the telescope.

10.6 *Precision and Bias*—Precision must be determined by repetition of measurements of each of the two pieces of the specimen (or of the four edges of a rectangular prism). Bias will depend upon the accuracy of the 90° angle of the vee-block prism and on the accuracy of its known indices. The accuracy of the measurement angles of the pieces of the specimen will affect bias if they are not exactly complementary. Well-made 90° reference prisms having known indices of refraction can be used to check the alignment of the system and the accuracy of the scale of angular measurement.

11. Prism Spectrometer

11.1 *Summary of the Method*:

11.1.1 The principle of the prism spectrometer for determining refractive index is illustrated in Fig. 6. A specimen is cut to form a prism with two polished faces that intersect in the angle ϕ. The prism is mounted on a rotary stage with its plane of symmetry oriented along the centerline of the rotary stage. The line of intersection of the polished faces is made parallel to the axis of rotation of the stage (see 11.2.4). A collimated beam of light is used to determine the magnitude of the angle ϕ. A collimated beam of monochromatic light is then made to cross the specimen-prism. Refraction at the entrance and exit faces causes the exit beam to deviate from its original direction by an angle β. The stage is rotated to minimize the deviation-angle β. Using a telescope, the operator adjusts the specimen's rotation to achieve the condition of minimum deviation precisely. The angular displacement of the telescope from the direction of the undeviated beam is read from the scale of the goniometer, yielding the angle of minimum deviation β.

NOTE 15—The following text addresses two aspects of realizing the measurement apparatus. One is the "prism spectrometer" that is self-contained with respect to the autocollimator, prism table, telescope, and calibrated goniometric scales. The other treats these components as assembled from separate units. Measurement procedures can vary in some particulars between these two systems.

11.1.2 This method is absolute, because only the angular measurements of the prism-angle ϕ and of the deviation β are used to determine the refractive index of the specimen; a calibrated reference specimen is not required, and no index-matching liquids are used.

NOTE 16—A prism spectrometer capable of accuracy to about 1×10^{-6}, a common requirement for optical glasses, is extremely expensive. Preparation of the test prism, too, is expensive, and the size of the required test material may be large. For example, the apertures of the objective lenses of the collimator and the telescope of one such spectrometer are about 75-mm diameter The faces of the test prism would need to have even larger cross-sections.

11.2 *Apparatus*:

11.2.1 The arrangement of the essential components of a prism spectrometer for determining refractive index is shown in Fig. 6. Practice E 167 provides further information on the names and nature of the components (Note 13). The "source arm" has a light source, entrance slit, and collimator. The "receptor arm" has a telescope.

NOTE 17—Practice E 167 relates to the measurement of the angular distribution of light transmitted through or reflected from objects or materials, for which the requirements for the apparatus might not be as strict as needed for high accuracy refractometry.

11.2.2 *Light Sources*—For highest accuracy work, spectral lamps are used. A white light source may be used with

narrow-bandpass interference filters at the input to the collimator. Additional filtering may be used at the output of the telescope, if needed to improve seeing. For measurements at more closely spaced wavelengths (if, for example, a curve showing $n = n(\lambda)$ is desired), a monochromator with a white light source may be used to illuminate the entrance slit of the collimator (or actually to provide the entrance slit).

11.2.3 *Collimator*—An autocollimator is used in the source arm for measuring the prism angle ϕ and for setting the faces of the prism exactly perpendicular to the horizontal optical axis of the system. (These can be accomplished with the telescope if it has provision for autocollimation.)

11.2.4 *Specimen Mounting Stage (Prism Table)*—This rotatable stage should have provision for tilting the specimen-prism in order to adjust both of the polished prism-faces to be exactly perpendicular to the horizontal optical axis of the system. Calibrated angular rotation of this stage is provided in some prism spectrometers for the measurement of the prism-angle ϕ.

11.2.5 *Receptor Arm*—Unless incorporated in a prism spectrometer, the viewing telescope must be mounted on a precision rotary table with a vernier scale that is readable to 1 arc-s (or better, for 10^{-6} refractometry), or with a calibrated electrical readout of the same degree of resolution. For visual observation, a crosshair-equipped recticle is placed in its focal plane. For greater sensitivity, a position-sensitive photodetector may be used in place of the cross-hair.

11.2.6 *Specimen Preparation*—For high accuracy refractometry, the specimen should be cut from an annealed homogeneous block of glass in the form of a roughly finished prism. Two intersecting faces are ground flat and polished; other faces are not polished. The usual prism-angle is about 60°; however, a smaller angle can be used if material is limited; the result will be less accurate. A mounting-base should be cut approximately perpendicular to the polished faces.

11.3 *Procedure*:

11.3.1 With the specimen-prism removed, rotate the telescope to a position where collimated light from the source enters at the center of the aperture of the telescope's objective lens; the image of the collimator slit should be centered on the cross-hair. This establishes the zero position for angular deviation. Record the scale reading.

11.3.2 Place the specimen-prism on the rotary sample stage (prism table) with its mid-plane along the centerline (Fig. 6: X-X) of the stage. Rotate the stage to make one face perpendicular to the collimated light beam; use the tilt adjustments of the stage to make the face perpendicular along its vertical axis. Rotate the other face into the beam and make similar adjustments (use the autocollimating feature of the collimator). Iterate these adjustments until both faces are perpendicular to the light beam in both their vertical and horizontal axes.

11.3.3 Rotate the stage and observe the motion of the refracted beam, using a white screen. Continue rotating until the deviation angle β is seen to be a minimum.

11.3.4 Rotate the receptor arm to place the telescope in position to receive the refracted beam. If the refracted beam is not centered on the entrance pupil of the telescope, shift the mid-plane of the specimen prism slightly to bring it more closely into alignment with the centerline of the stage (Fig. 6: X-X). Then repeat the setting of the minimum deviation.

11.3.5 Observe the image of the source slit on the recticle of the eye-lens of the telescope. Make the final adjustment of the angle β of the receptor arm by placing the center of the slit-image at the cross-hair of the reticle. Read the angle-scale, using the vernier for greatest accuracy. Calculate the value of β as the difference between the zero-point scale reading and the final scale reading. (Alternatively, use the corresponding readings of the electrical readout.) Repeat the procedure five times and calculate the average of the minimum deviation angles, β_{min}.

11.3.6 Verify the angular position of the prism stage. The angle of the mid-plane to the goniometer axis should equal $\beta/2$.

11.4 *Calculation*—(See Fig. 6 for identification of the angles represented by the symbols ϕ and β.) The index of refraction measured for wavelength λ is calculated by:

$$n_\lambda = \{[\sin(\phi + \beta_{min})/2] / [\sin \phi/2]\} \quad (7)$$

11.5 *Precision*—Using a goniometer that can resolve 1 arc-s, a precision of better than 1×10^{-5} can be obtained, provided that the prism-angle ϕ is known to the same accuracy.

12. Specular Reflectance

12.1 *Summary of the Method*—The specular reflectance of a polished glass surface, for irradiation and detection at normal incidence, is related to the refractive index of the glass by the Fresnel relationship:

$$R = [(n_g - 1)/(n_g + 1)]^2 \quad (8)$$

where:

R = specular reflectance, and
n_g = refractive index of the glass.

12.1.1 This equation is inverted to express the refractive index as a function of the specular reflectance:

$$n_g = (1 + R^{1/2})/(1 - R^{1/2}) \quad (9)$$

12.1.2 The reflectance is measured with a spectrophotometer with a specular reflectance accessory. Any wavelength within the range of the spectrophotometer can be used, provided that operating parameters can be set for adequate signal-to-noise ratio.

12.2 *Apparatus*—Spectrophotometer with specular reflectance accessory and reference mirror. Because the reflectance of glass is small, greater accuracy is obtained by using a reference mirror of comparably small reflectance. A glass of known refractive index at the measurement wavelength, prepared in the manner described in 12.3, and preferably one that has appreciable absorption at that wavelength, is the best reference mirror. Calculate its reflectance by Eq 8.

12.3 *Specimen Preparation*—Use an approximately rectangular block of glass with one flat, polished face having an area commensurate with the requirements of the specular reflectance accessory (for example, about 20 mm by 30 mm) and as thick as the accessory will accommodate, or as thick as available material permits. The back surface of the block should be treated to minimize reflected radiation from it being included in the measurement beam. A glass that absorbs highly

at the measurement-wavelength does not need special treatment. Grind the back side to provide a coarse matte finish and paint it with black paint. Making the specimen somewhat wedge-shaped (if that is commensurate with mounting in the reflectance accessory) can deflect second-surface reflected radiation from the measurement beam.

12.4 *Procedure*—Follow the manufacturer's instructions for using the specular reflectance accessory. Set the spectrophotometer at a desired wavelength. Insert the reference mirror and adjust the transmittance (reflectance) scale of the spectrophotometer to 100 % or, for a digital spectrophotometer, store the signal as the baseline. Replace the reference mirror with the specimen and record its indicated reflectance. In the case of a digital instrument, the correction of the specimen's signal to be displayed as its value of reflectance might be done automatically.

12.5 *Calculations*:

12.5.1 *Reflectance*—Calculate the reflectance of the specimen by multiplying its indicated reflectance by the known reflectance of the reference mirror. In the case of a digital spectrophotometer, multiply the corrected, displayed result by the known reflectance of the reference mirror.

12.5.2 *Refractive Index*—Calculate the refractive index of the specimen by using Eq 9.

12.6 *Precision and Bias*:

12.6.1 The precision of the method depends upon the reproducibility of the reflectance measurements, which must be determined by repeated trials; however, because the accuracy of the method is poor, determination of precision is not likely to be necessary.

12.6.2 Bias will depend upon the performance of the spectrophotometer and the alignment of the specular reflection accessory. The radiation beam of a spectrophotometer is partially polarized and the angle of incidence set by the specular reflectance accessory is, usually, from 6 to 10°. These considerations may be ignored as sources of error, because the precision of the method is relatively poor. The uncertainty Δn_g of the refractive index is:

$$\Delta n_g = R^{-1/2}\,(1 - R^{1/2})^{-2}\,\Delta R \tag{10}$$

where:

ΔR = uncertainty of the measured value of R.

Examples

Example (1): $R = 0.040$, $\Delta R = 0.002$: $n_g = 1.500 \pm 0.013$
Example (2): $R = 0.111$, $\Delta R = 0.002$: $n_g = 2.000 \pm 0.009$

12.7 *Discussion*—The examples show that the accuracy of this method is about 0.01, and this represents a very favorable determination of the value of the reflectance. Because the accuracy of this method is poor, the calculation of a dispersion-value is not likely to be useful. The main value of this method is that it can be used to determine refractive indices in the ultraviolet and infrared spectral regions.

13. Keywords

13.1 Abbe-number; dispersion; glass; index of refraction; refractive index; refractometer

ANNEX

(Mandatory Information)

A1. FOUR ADDITIONAL METHODS FOR MEASURING REFRACTIVE INDEX

A1.1 *Immersion Refractometer*:

A1.1.1 Faick and Fonoroff (**8**) describe the concept of the immersion refractometer. The parts include a small tank (about 20 by 10 by 10 cm: L×W×H, for example) with parallel glass windows sealed in the opposite sides; a spectral lamp; slit; collimating lens; and telescope with a knife-edge for schlieren observation. The refractive index of the immersion liquid must be very close to that of the glass to be tested. It is evident from the large volume of liquid required that this method has its principal application for production monitoring of a single glass, although the liquid can be changed for production runs of other glasses. Accuracy may be set by that of an Abbe-type refractometer used to measure the index of the liquid when a match with a test specimen has been made, but it is possible to achieve accuracies of 1×10^{-5}. The index of the liquid can be adjusted by adding, drop-by-drop, either a higher or a lower index liquid until a match with an immersed specimen has been found. A drop of the well stirred liquid is transferred to the Abbe refractometer and measured. Temperature correction must be made. A different way to adjust the index of the liquid is to change the temperature of the bath and to use a calibration curve of its index as a function of temperature. For higher accuracy, a stack of several calibrated pieces that vary over a small range of indices is immersed with the test specimen. Their indices are known to at least the accuracy desired for the test. Changing composition or temperature of the bath is done until the index of the liquid matches simultaneously that of the test specimen and of one of the members of the stack.

A1.2 *Interferometry*:

A1.2.1 Apparatus requirements are a spectral lamp, collimator, precision goniometer, telescope, and means of counting successive bright and dark transitions (fringes) as the specimen is rotated in the collimated beam. The specimen must have flat, polished parallel sides. It acts as a Fabry-Perot interferometer. The length of the light path through the specimen changes as it is rotated. From measured thickness and fringe-count as a function of angle of rotation, the refractive index can be calculated.

A1.3 *Ellipsometry*:

A1.3.1 The principle of ellipsometry is that the optical constants (the real and imaginary parts of the complex refractive index) can be determined by measuring the specular reflectance of polarized light at two angles of incidence. There are commercial ellipsometers fully configured for this type of analysis.

A1.4 *Method of Oblique Illumination*:

A1.4.1 This is a variation of the Becke-line method of central illumination (Section 6). The apparatus and materials are the same, and the preparation, mounting, and immersion of the test specimen are the same. After the immersed glass particles have been brought into focus of the microscope with the iris diaphragm nearly closed, the diaphragm is opened wide. Then, an opaque card is inserted into the light beam from one side, blocking about one half of the beam. As the brightness of the field diminishes, glass particles will readily be seen, shaded toward one side and bright on the other. If the index of a particle is greater than that of the immersion liquid, the bright side will be opposite from the side where the card is inserted. Conversely, if the index of a particle is lower than that of the liquid, its bright side will be on the same side as the card.

APPENDIX

(Nonmandatory Information)

X1. ANALYSES FOR THE APPARENT DEPTH OF MICROSCOPE FOCUS METHOD (SECTION 7)

X1.1 The depth of focus of a microscope objective lens is the distance through which the focal point can be moved without the observer being able to recognize a change in the appearance of the observed object. The depth of focus is smaller as the focal length of the objective lens is smaller (and its magnification is higher). For visual observation (in contrast to photographic), the effective depth of focus is greater than theoretical, because the eye accommodates to changes of apparent distance. Fig. 2*(a)* shows a microscope focussed on a mark on the top surface of a specimen being tested for refractive index by the apparent depth of focus method. The distance w between the surface of the objective lens and its focal point is called the working distance of the lens. It is evident from Fig. 2*(b)* that the working distance of the objective limits the optical thickness of a specimen, if a mark on its bottom surface is to be brought into focus. (Optical thickness is the product $n \times t$, where n is refractive index and t is physical thickness.) There are, then, two conflicting interests. The precision of focusing on a mark is improved with a lens of higher magnification, but because the working distance is less, the thickness of the specimen that can be measured is less. An error analysis shows that the contribution to the uncertainty n_g by an error of measurement of thickness is appreciably less than that caused by errors of focussing. The suggested (7.2.1) magnification of the microscope objective of about 10× is a very good compromise.

X1.2 The tangent of the angle ϕ shown in Fig. 2*(a)* and Fig. 2*(b)* is the ratio of the radius of the objective lens to its focal length. The sine of ϕ is designated the numerical aperture, *NA*, of the objective. The symbols used in the derivations of Eq 3 (7.1) and Eq 4 (7.5) are shown in Fig. 2*(b)*.

$$y = t \tan \theta = d \tan \phi \qquad \text{(X1.1)}$$

$$t/d = \tan \phi / \tan \theta$$

where:

t = thickness of the test specimen, and

d = displacement of the microscope focus on moving from the top to the bottom surface.

X1.2.1 The small-angle approximation that the sine and the tangent are equal is applied, when:

$$t/d = \sin \phi / \sin \theta \qquad \text{(X1.2)}$$

by Snell's law:

$$n_g \sin \theta = \sin \phi \qquad \text{(X1.3)}$$

when:

$$n_g = t/d \qquad \text{(X1.4)}$$

which is Eq 3 (7.1).

X1.2.2 This approximation introduces a large error, which can be avoided.

$$t/d = \tan \phi / \tan \theta = (\sin \phi / \cos \phi) / (\sin \theta / \cos \theta) = n_g (\cos \theta / \cos \phi) \qquad \text{(X1.5)}$$

$$n_g = t/d\,(\cos \phi / \cos \theta) = t/d\,[1 - \sin^2 \phi]^{1/2} / [1 - \sin^2 \theta]^{1/2}$$

$$n_g = t/d\,[1 - NA^2]^{1/2} / [1 - n_g^{-2} \sin^2 \phi]^{1/2}$$

$$= t/d\,[1 - NA^2]^{1/2} / [1 - n_g^{-2} NA^2]^{1/2}$$

$$n_g [1 - n_g^{-2} NA^2]^{1/2} = t/d\,[1 - NA^2]^{1/2}$$

$$n_g^2 - NA^2 = (t/d)^2 [1 - NA^2]$$

$$n_g^2 = (t/d)^2 [1 - NA^2] + NA^2 = (t/d)^2 + NA^2 [1 - (t/d)^2]$$

$$n_g = \{(t/d)^2 - NA^2 [(t/d)^2 - 1]\}^{1/2}$$

which is Eq 4 (7.5).

REFERENCES

(1) Miller, A., "Duc de Chaulnes' Method for Refractive-Index Determination."

(2) Straat, H. W., and Forrest, J. W., "The Accuracy Requirements in Fifth Place Refractometry," *Journal of the Optical Society of America*, Vol 29, No. 6, June 1939, pp. 240-247.

(3) Tilton, L. W., "Testing and Accurate Use of Abbe-Type Refractometers," *Journal of the Optical Society of America*, Vol 32, No. 7, July 1942, pp. 371-381.

(4) International Commission of Optics, "Recommendations for the Standardization of Sign Conventions and Symbols in Geometrical Optics, and for the Definition of Refractive Indexes of Optical Glass," *Journal of the Optical Society of America*, Vol 41, No. 2, February 1951, pp. 140-141.

(5) Gunter, R. C., and Kobeissi, M. A., "Tolerated Range of the Refracting Angle of a Glass Sample Whose Index *n* is Measured on a Refractometer," *Applied Optics*, Vol 10, No. 8, August 1971, pp. 1966-1967.

(6) Grauer, O. H., *National Bureau of Standards Notes, Journal of the Franklin Institute*, Vol 256, November 1953, p. 463.

(7) Simmons, J. H., and Potter, K. S., *Optical Materials*, Academic Press, San Diego, CA, 2000, pp. 136-137.

(8) Faick, C. A., and Fonoroff, B., "A Precision Apparatus for the Rapid Determination of Indices of Refraction and Dispersion by Immersion," *Journal of Research of the National Bureau of Standards*, Vol 32, 1944, p. 67.