



Designation: C1648 – 12 (Reapproved 2023)

# Standard Guide for Choosing a Method for Determining the Index of Refraction and Dispersion of Glass<sup>1</sup>

This standard is issued under the fixed designation C1648; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 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  $\mu\text{m}$  to 780  $\mu\text{m}$ ); 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<sup>2</sup> 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, health, and environmental 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.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards*:<sup>3</sup>

[E167 Practice for Goniophotometry of Objects and Materials \(Withdrawn 2005\)](#)<sup>4</sup>

[E456 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.

<sup>1</sup> 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.

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<sup>2</sup> Metricon is a trademark of Metricon Corporation 12 North Main Street, P.O. Box 63, Pennington, New Jersey 08534.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

**TABLE 1 Spectral Lines for Measurement of Refractive Index<sup>A</sup>**

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 <sup>B</sup>	656.3 <sup>C</sup>	643.8 <sup>D</sup>	589.3	587.6	546.1	486.1	480.0 <sup>D</sup>	435.8	434.0	404.7

<sup>A</sup> From Ref (1).

<sup>B</sup> A later reference (identification not available) lists 789.9 nm for the potassium A' line, although referring to Ref (1). 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.

<sup>C</sup> The wavelength of the corresponding deuterium line is 656.0 nm.

<sup>D</sup> The two cadmium lines have been recognized for refractometry since Ref (1) was published.

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<sup>-5</sup> method.

3.2 *Symbols: n* = index of refraction

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

*v<sub>D</sub>* = Abbe-number determined with spectral lines *D*, *C*, and *F*

*v<sub>e</sub>* = 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<sup>-4</sup> (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<sup>-6</sup>. 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<sup>-4</sup> 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 *v*-value), calculated by:

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

where *v<sub>D</sub>* is defined in 3.2 and *n<sub>D</sub>*, *n<sub>F</sub>*, and *n<sub>C</sub>* are the indices of refraction at the emission lines defined in 3.2.

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.

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

where *v<sub>e</sub>* is defined in 3.2 and *n<sub>e</sub>*, *n<sub>F'</sub>*, and *n<sub>C'</sub>* are the indices of refraction at the emission lines defined in 3.2.

4.2.2 A consequence of the defining equations (Eq 1 and 2) is that smaller *v*-values correspond to larger dispersions. For *v*-values accurate to 1 % to 4 %, index measurements must be accurate to 1×10<sup>-4</sup>; therefore, citing *v*-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 *v*-values represent larger physical dispersions, the term constringence is used in some texts instead of dispersion.

## 5. Precision, Bias, and Accuracy (see Terminology E456)

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  $1 \times 10^{-6}$ , 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  $1 \times 10^{-6}$ , the bias must be not greater than  $5 \times 10^{-7}$ . 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  $1 \times 10^{-6}$  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.

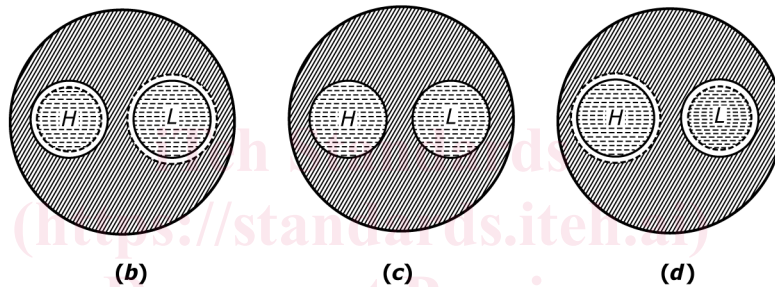
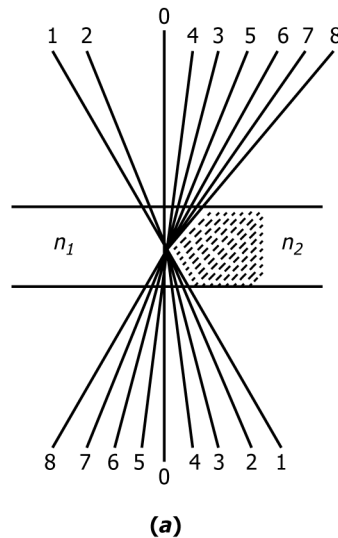
**6.2 Advantages and Limitations**—This method uses the smallest amount of specimen-material and it has the simplest and least expensive method of sample-preparation. Costs of apparatus and materials, too, are moderate, as is the time needed to make a determination; however, the accuracy of the method is limited to about  $5 \times 10^{-4}$  (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  $1 \times 10^{-4}$  (optical density 4.0); it occurs near 589 nm. Its refractive index was determined to  $1 \times 10^{-3}$  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 $\times$  that has a sub-stage condenser with a



(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

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<sup>2</sup> 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 *v*-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.

6.3.4 *Calibrated Immersion Oils*—Sets of calibrated index oils are available with indices over the range 1.300 to 2.31.<sup>5</sup>

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  $2 \times 10^{-4}$ , 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  $2 \times 10^{-4}$  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

<sup>5</sup> Cargille Laboratories, Inc., 55 Commerce Road, Cedar Grove, NJ 07009-1280, Tel. 973-239-6633, www.cargille.com



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 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 (“standardiza-

tion”) 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 (2)<sup>6</sup> 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:

$$n_g \approx t/d \quad (3)$$

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 in **Appendix X1**.

### 7.2 Apparatus and Materials:

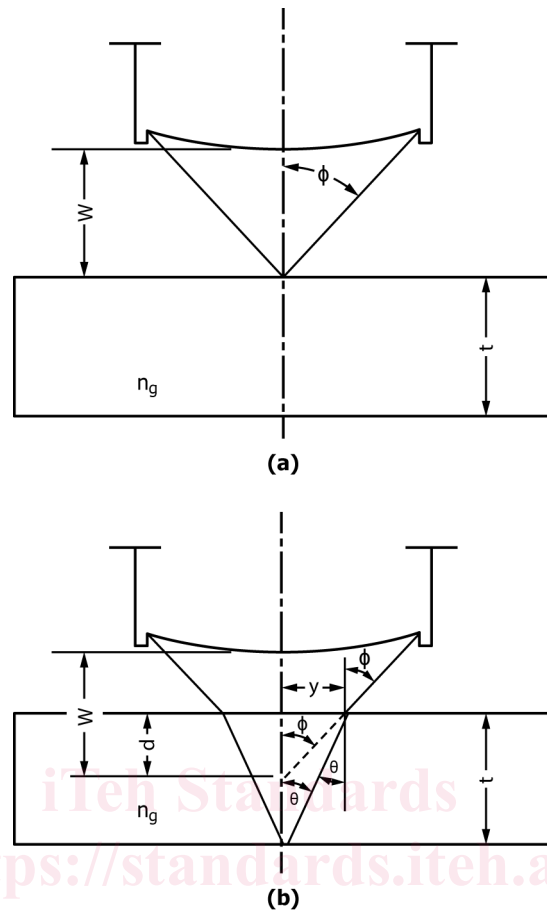
**7.2.1 Microscope**—The microscope should have a total magnification of about 100× and the objective lens should provide about 10× of that. (See **Appendix X1** for discussion of effect of magnification of the objective lens.) The stage should have provision for fine adjustment of its elevation.

**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

<sup>6</sup> 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

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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 cm 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 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

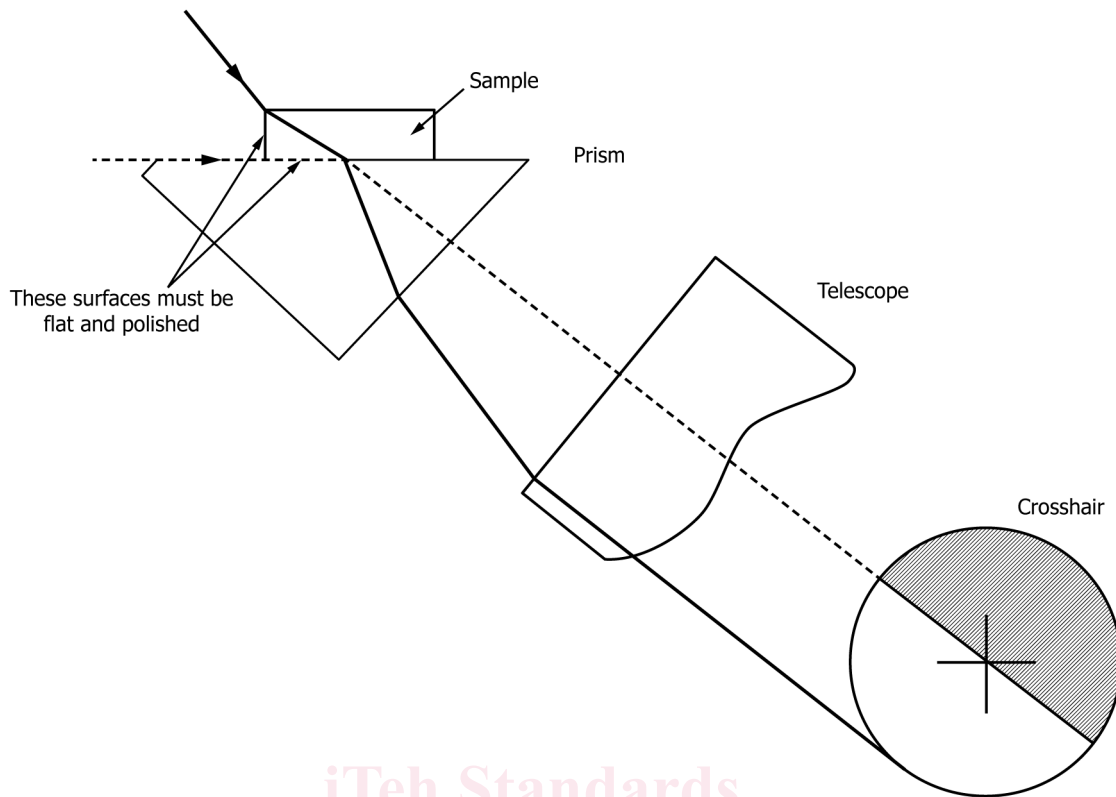


FIG. 3 Principle of Critical Angle Refractometers

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 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 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 (3) analyze accuracy requirements for fifth decimal place refractometry. Tilton (4) 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  $\theta_c$  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