Designation: E1967 – 19

An American National Standard

Standard Test Method for the Automated Determination of Refractive Index of Glass Samples Using the Oil Immersion Method and a Phase Contrast Microscope[1]

This standard is issued under the fixed designation E1967; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for measuring and comparing the refractive index (η) at a fixed wavelength (λ) and temperature (*T*) (η_λ^T) of glass from known sources to recovered fragments from a questioned source.

1.2 This test method does not include the measurement of optical dispersion or the measurement of refractive index (η_λ^T) at any other wavelength other than the Sodium D line (η_D^T). This method employs a narrow band pass filter at 589 nm, but other filters could be employed using the described method, allowing the η_λ^T to be determined at other wavelengths, and therefore, also allowing for the dispersion value to be calculated.

1.3 Alternative methods for the determination of η_λ^T are listed in Refs (**1-5**).[2]

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard cannot replace knowledge, skills, or abilities acquired through education, training, and experience and is to be used in conjunction with professional judgment by individuals with such discipline-specific knowledge, skills, and abilities.*

1.6 *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.*

1.7 *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:*[3]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 Oil immersion refractometry is used to determine the refractive index of glass fragments. The glass fragments are mounted on a glass microscope slide in an immersion oil of known refractive index over the temperature range of interest. The glass microscope slide is placed on a phase contrast microscope mounted with a hot stage and a video camera. The phase contrast microscope is employed with illumination at a fixed wavelength (nominally Sodium D) to magnify the image of glass particles immersed in the silicone oil. The microscope is aligned to produce even illumination with maximum contrast. A video camera is attached to a photography port (the output of the image) to observe the immersed glass and measure the contrast between the glass and the oil. The temperature of the oil is changed via the hot stage and an electronic temperature controller until the glass particles' image disappears. The temperature of the oil at which there is minimum contrast between the glass and the liquid then is recorded manually or electronically as the match temperature.

3.2 The match temperature is converted to η_D^T by reference to a calibration curve for the immersion oil. This calibration curve has been previously created by measuring the match temperatures of traceable reference glass standards of known η_D^T within the temperature range of interest. This curve or its mathematical equivalent normally is stored within the computer and used to determine the η_D^T of any glass of interest, whether it is a fragment of known origin or a recovered

[1] This test method is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics.

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

[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.

(questioned) fragment.

3.3 The refractive indices of recovered glass fragments are compared to a test interval defined by the range of refractive index measurements of a broken glass object (known) to determine if they can be excluded as originating from the same source.

3.4 The glass sample is typically crushed prior refractive index analysis. The analyst shall examine the glass fragments for any potential fracture match before altering the physical characteristics of the sample. Some of the crushed glass fragments are immersed in silicone oil. The glass fragments are recovered from the oil for sample preservation, as required.

4. Significance and Use

4.1 This test method is useful for accurate measurement of η_D^T from a wide variety of glass samples, whose η_D^T ranges from 1.48–1.55.

4.2 It should be recognized that measurement of surface fragments, especially from float glass samples, can result in refractive index values which are different than the refractive index values of fragments from the interior of (for example, bulk) the same broken glass source (**5**).

4.3 The precision of this test method shall be established in each laboratory that employs it as part of the validation protocol (see Section 9).

4.4 It should be recognized that this technique measures the refractive index of the glass at the match point temperature, which will be higher than ambient temperature, and thus, may give different η_D^T values from those obtained by other methods, which measure the refractive index at room temperature.

5. Apparatus

5.1 *Microscope*—A microscope outfitted for phase contrast and an appropriate long working distance objective (nominally 10–40×) is required.

5.2 *Temperature Control*—A hot stage is required that connects to a control device with a working range of approximately 26°C to 118°C, having a minimum precision of 0.1°C.[4]

5.3 *Imaging*—A digital video camera is required for the automated measurements and is mounted to an ocular or photography port of the microscope. The output from the camera (image) is used for automated match point determinations.

5.4 *Illumination*—A narrow band interference filter is required. For Sodium D measurements 589 ± 5 nm with a band pass of 10 nm is appropriate. The intensity of the illumination is adjusted to give the brightest image possible, without overloading the video camera.

5.5 *Immersion Oils*—Silicone immersion oils having refractive indices within a specific range are required and are calibrated with the necessary standard reference glasses of known η_D^T.

5.6 *Standard Reference Glasses*—A minimum of three reference η_D^T are required to construct the calibration curve for each silicone oil used. When they are commercially available, three or more traceable reference glasses shall be used to construct the calibration curve. In addition, a minimum of one reference glass (control) not used to construct the calibration curve is required to verify the calibration.

5.7 *Computer*—A computer is required that is equipped with appropriate software and hardware to view the glass fragments, measure edge contrast, and record match temperatures.

6. Procedure

6.1 Select a number of glass fragments from the known glass sample such that the variability in the refractive index throughout the source can be assessed.

6.2 A minimum of ten refractive index replicate measurements per known source for most non-tempered glass and twenty for most tempered glasses is recommended (**6**).

6.3 Collect replicate measurements of the questioned glass to insure that it is adequately characterized. A minimum of three replicates is recommended for the comparison of the refractive index of glass (**7**).

6.4 Arrange the microscope for optimum illumination and phase contrast. To insure maximum contrast, make sure the annular illumination ring from the condenser is aligned properly with the phase contrast shift plate, which is located within the objective. The rings are imaged on the back focal plane of the objective and can be observed in a number of ways, the most convenient of which is the use of Bertrand lens or a phase centering telescope.

6.5 Calibrate the necessary η_D^T oil as indicated in 7.3.

6.6 After using an appropriate cleaning technique, such as a deionized water and alcohol rinse followed by drying, crush a small fragment of the glass to be measured and deposit a small sample on a clean microscope slide. Immerse this sample in the proper calibrated silicone oil and cover with a cover slip.

6.7 Place the covered slide onto the hot stage and focus the image. The phase ring alignment shall be checked each time that a new slide preparation is inserted into the hot stage to ensure that the phase rings are in alignment.

6.8 To minimize analysis time, set the temperature of the hot stage to within several degrees of the estimated match point. To estimate the match point of a glass, vary the temperature of the hot stage either up or down as appropriate until the match point has been reached and passed. The match point is that point at which the contrast is at a minimum, which corresponds to the disappearance of the edge of interest. With computer-controlled units, the match point will be recorded automatically.

6.9 Using the imaging and controller software, analyze localized portions (commonly referred to as "measurement windows") of the edge of the glass for changes in refractive index. Place each measurement window across a portion of a focused glass edge and the immersion oil. Begin the automated analysis, which will change the hot stage temperature in a

[4] Mettler Models FP502 and HS82 have been found satisfactory for this function. Other similar brands/models can be used.