Designation: ~~E1967 – 11a~~ E1967 – 19

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(η)$_\lambda^T$) of glass samples, irregularly shaped and as small~~ at a fixed wavelength (λ) and temperature (*T* ~~as~~) (~~η_λ^T 300 µg, for the comparison of fragments of a known source~~) 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($_T\eta_\lambda^{T^t}$)~~ at any other wavelength other than the Sodium D line ~~(η($_D\eta_D^{T^t}$)~~. This method employs a narrow band pass filter at 589 nm, but other filters could be employed using the described ~~method and~~ method, allowing the ~~η$_\lambda^T$~~ 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$_\lambda^T$~~ 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 ~~test method~~ 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~~ safety, health, and ~~health~~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 ~~A~~ 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 ~~while these are~~ immersed in ~~a~~the silicone oil. The microscope is aligned to produce even illumination with maximum ~~contrast and a~~ contrast. A video camera is attached to ~~an eyepiece~~ a photography port (the output of the image) to observe the immersed glass and measure the contrast ~~of~~between the ~~image of~~glass and the ~~glass.~~oil. The temperature of the oil is changed via ~~a~~the hot stage

[1] This ~~practice~~ 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.

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.~~electronically as the match temperature.

3.2 ~~A microprocessor or other handling station, such as a personal computer, employs a video camera interfaced by appropriate software and hardware to view the glass fragments. These commercial electronics result in a digital count representing a preselected edge feature's contrast being determined. This edge or contrast measurement is updated with every frame of video as the temperature of the hot stage, oil, and sample are ramped up or down. The software automatically registers the match point by taking the average of the minimum contrast measurements for both the cooling and the heating cycles. This match temperature can be converted to η~~The match temperature is converted to $\eta_{\lambda D}^{T\ t}$ by reference to a calibration curve for the immersion ~~oil previously created from the match temperatures obtained on reference glass standards.~~ oil. This calibration curve ~~is obtained from reference glasses of known η~~has been previously created by measuring the match temperatures of traceable reference glass standards of known $\eta_{\lambda D}^{T\ t}$~~'s~~ within the temperature range of interest. This curve or its mathematical equivalent normally is stored within the ~~microprocessor~~computer and ~~is employed~~ used to determine the ~~$\eta\eta_{\lambda D}^{T\ t}$~~ of any glass of interest, whether it is a fragment of known origin or a recovered (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 ~~Precise control and measurement of the immersion liquid temperature is achieved by use of a microscope hot stage. A precision of 0.05°C for the hot stage is desirable, but a precision of 0.1°C is the requirement for interlaboratory comparisons.~~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

~~3.1 This technique modifies the sample, in that the glass fragment must be crushed, if it is too large, and immersed in oil for the analysis. Some sample handling, however, would enable the analyst to recover the sample in the crushed form, if necessary.~~

4.1 This test method is useful for accurate measurement of ~~$\eta\eta_{\lambda D}^{T\ t}$~~ from a wide variety of glass samples, ~~where most~~whose n_D^T ~~glasses of interest have η~~ranges from 1.48–1.55.~~$_D^{\ t}$~~ ~~in the range between 1.48 – 1.55 in $\eta_D^{\ t}$ units.~~

~~3.3 The objective nature of the match point determination allows for a better standardization between laboratories, and therefore, allows for the interchange of databases between laboratories.~~

4.2 It should be recognized that measurement of surface fragments, especially from float glass samples, can result in ~~η~~refractive~~$_D^{\ t}$~~ ~~s measurably higher than~~ index values which are different than the refractive index values of fragments from the ~~bulk of~~ interior of (for example, bulk) the same broken glass source ~~((~~**5**~~).).~~

4.3 The precision ~~and bias~~ of this test method ~~should~~shall be established in each laboratory that employs ~~it. Confidence intervals or a similar statistical quality statement should be quoted along with any reported η~~it as part of ~~$_D^{\ t}$~~ value. For instance, a laboratory ~~may report that the error for the measurement,~~the validation protocol (see Section 9~~using a reference optical glass is 0.00003 units.~~).

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 ~~$\eta\eta_{\lambda D}^{T\ t}$~~ 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 ~~objective (nominally 10× – 40×) with a~~ long working distance ~~condenser is employed.~~objective (nominally 10–40×) is required.

5.2 *Temperature Control*—A hot stage ~~connected~~ 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 is employed.~~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 ~~the image processing for~~ automated match point determinations.

5.4 *Illumination*—A narrow band interference filter is ~~employed as a monochromatic source.~~ 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 ~~for the glasses under~~

[4] Mettler Models FP502 and ~~FP82~~HS82 have been found satisfactory for this function. Other similar brands/models can be used.

~~study~~ and are calibrated with the necessary standard reference glasses of known ~~$\eta_{D}^{T}{}_{D}^{t}$~~.

5.6 *Standard Reference Glasses*—A minimum of three reference ~~$\eta_{D}^{T}{}_{D}^{t}$~~ are ~~used, when possible, for~~ required to construct the calibration ~~of~~ curve for each silicone oil ~~to be used for the actual measurements.~~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 ~~Prior to crushing the glass sample for the η~~Select a number of glass fragments from the known glass sample such ~~$_{D}^{t}$~~ ~~measurement, one should be certain that the possibility of obtaining a physical match has been explored and other examinations requiring larger sample size, such as density have not been precluded.~~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 by viewing the superimposition at~~objective. The rings are imaged on the back focal plane of the ~~objective. This alignment~~ objective and can be ~~accomplished~~ observed in a number of ways, the most convenient of which is the use of ~~Bertrand™~~Bertrand lens or a phase centering telescope.

6.5 Calibrate the necessary ~~$\eta_{D}^{T}{}_{D}^{t}$~~ oil ~~from a set~~as indicated in 7.3~~of three oils represented by oils of approximately 1.50, 1.53, and 1.55 using reference glasses of known $\eta_{.D}^{t}$ to ± 0.00001. At least three glasses for each oil should be employed for the calibration. Once calibrated, the η_{D}^{t} of the oils can be plotted against the match temperatures to produce a calibration curve for each oil. The preprogrammed protocol within the automated system to perform this function can be used.~~

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 ~~studied~~measured and deposit a small sample on a ~~clean, flat~~ 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 ~~must~~shall be checked each time that a new slide preparation is ~~made~~ inserted into the hot stage to ensure that the phase rings are in alignment.

6.8 ~~Vary the temperature by ramping up, or down, past the match point and then cooling down, or heating up, past the match point. Record~~ 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 ~~temperature in both directions and calculate the average. With microprocessor controlled units, recording will be performed automatically.~~ 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.

~~5.7 Determine the η_{D}^{t} of the glass fragment measured by reading the η_{D}^{t} from the calibration curve (η_{D}^{t} versus match temperature) for the average match temperature. For the microprocessor-controlled units, this calculation is displayed and printed automatically. The η_{D}^{t} value will represent the η_{D}^{t} of the sample at the match point temperature. To obtain the η_{D}^{t} at ambient temperature the value must be corrected using the dn/dt for that glass. Note that this is not usually known for casework glass samples. The match point temperature must be noted in the final report.~~

6.9 Using the imaging and controller software, analyze localized portions (commonly referred to as "measurement windows") of the ~~entire image,~~ 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 controlled manner. Alternatively, record the video image of the heating and cooling cycles before setting the measurement windows. ~~Record~~ The computer automatically records the match point temperatures for both cooling and heating cycles for each measurement ~~window. The match point is that point at which the contrast is at a minimum, which corresponds to the disappearance of the edge of interest. Calculate the~~ window and calculates the average of the heating and cooling match point temperatures ~~of~~for every measurement window separately.

6.10 The η_{D}^{T} of the glass fragment is measured by calculating the η_{D}^{T} from the calibration curve (η_{D}^{T} versus match temperature) based on the average match temperature. For computer-controlled units, this calculation is performed and displayed automatically.