Designation: E1967 – 11a

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 the refractive index ($\eta_\lambda{}^t$) of glass samples, irregularly shaped and as small as 300 µg, for the comparison of fragments of a known source 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 ($\eta_\lambda{}^t$) at any other wavelength other than the Sodium D line ($\eta_D{}^t$). This method employs a narrow band pass filter at 589 nm, but other filters could be employed using the described method and allowing the $\eta_\lambda{}^t$ to be determined at other wavelengths, therefore, also allowing for the dispersion value to be calculated.

1.3 Alternative methods for the determination of $\eta_\lambda{}^t$ 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 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 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 A 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 silicone oil. The microscope is aligned to produce even illumination with maximum contrast and a video camera is attached to an eyepiece (the output of the image) to observe the immersed glass and measure the contrast of the image of the glass. The temperature of the oil is changed via a hot stage and an electronic temperature controller until the glass particles' image disappears. The temperature at which there is minimum contrast between the glass and the liquid then is recorded manually or electronically.

2.2 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 $\eta_D{}^t$ by reference to a calibration curve for the immersion oil previously created from the match temperatures obtained on reference glass standards. This calibration curve is obtained from reference glasses of known $\eta_D{}^t$'s within the range of interest. This curve or its mathematical equivalent normally is stored within the microprocessor and is employed to determine the $\eta_D{}^t$ of any glass of interest, whether it is a fragment of known origin or a recovered (questioned) fragment.

2.3 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.

3. 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.

3.2 This test method is useful for accurate measurement of $\eta_D{}^t$ from a wide variety of glass samples, where most glasses of interest have $\eta_D{}^t$ in the range between 1.48 – 1.55 in $\eta_D{}^t$ units.

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

Current edition approved Dec. 1, 2011. Published December 2011. Originally approved in 1998. Last previous edition approved in 2011 as E1967 – 11. DOI: 10.1520/E1967-11A.

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