Designation: E2927 – 16ε1

Standard Test Method for Determination of Trace Elements in Soda-Lime Glass Samples Using Laser Ablation Inductively Coupled Plasma Mass Spectrometry for Forensic Comparisons[1]

This standard is issued under the fixed designation E2927; 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.

ε[1] NOTE—Editorial corrections were made to 11.1 in December 2017.

INTRODUCTION

One objective of a forensic glass examination is to compare glass samples to determine if they may be discriminated using their physical, optical or chemical properties (for example, color, refractive index (RI), density, elemental composition). If the samples are distinguishable in any of these observed and measured properties, it may be concluded that they did not originate from the same source of broken glass. If the samples are indistinguishable in all of these observed and measured properties, the possibility that they originated from the same source of glass may not be eliminated. The use of an elemental analysis method such as laser ablation inductively coupled plasma mass spectrometry yields high discrimination among sources of glass.

1. Scope

1.1 This test method covers a procedure for the quantitative elemental analysis of the following seventeen elements: lithium (Li), magnesium (Mg), aluminum (Al), potassium (K), calcium (Ca), iron (Fe), titanium (Ti), manganese (Mn), rubidium (Rb), strontium (Sr), zirconium (Zr), barium (Ba), lanthanum (La), cerium (Ce), neodymium (Nd), hafnium (Hf) and lead (Pb) through the use of Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) for the forensic comparison of glass fragments. The potential of these elements to provide the best discrimination among different sources of soda-lime glasses has been published elsewhere (**1-5**).[2] Silicon (Si) is also monitored for use as a normalization standard. Additional elements may be added as needed, for example, tin (Sn) can be used to monitor the orientation of float glass fragments.

1.2 The method only consumes approximately 0.4 to 2 µg of glass per replicate and is suitable for the analysis of full thickness samples as well as irregularly shaped fragments as small as 0.1 mm by 0.4 mm in dimension. The concentrations of the elements listed above range from the low parts per million ($\mu g g^{-1}$) to percent (%) levels in soda-lime-silicate glass, the most common type encountered in forensic cases. This standard method may be applied for the quantitative analysis of other glass types; however, some modifications in the reference standard glasses and the element menu may be required.

1.3 This standard does not replace knowledge, skill, ability, experience, education or training and should be used in conjunction with professional judgment.

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 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.*

1.6 *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.*

[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 a list of references at the end of this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E2330 Test Method for Determination of Concentrations of Elements in Glass Samples Using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) for Forensic Comparisons

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

C162 Terminology of Glass and Glass Products

3. Terminology

3.1 *Definitions:*

3.1.1 *calibration standard, n*—used to determine the quantitative analysis for the analyte elements of interest in the glass matrix. The calibration standard(s) shall have a known elemental composition including a known uncertainty for the reported analytes.

3.1.2 *glass, n*—an inorganic product of fusion that has been cooled to a rigid condition without crystallization. **C162**

3.1.3 *normalization standard, n*—an element that is present in the glass matrix at elevated and relatively homogeneous concentration that may be used to normalize the laser ablation signal to compensate for any variation on the ablated mass or instrumental drift.

4. Summary of Test Method

4.1 The glass fragments usually do not require sample preparation prior to the LA-ICP-MS analysis. However, they may be washed with solvents or pre-ablated if necessary.

4.2 The glass fragment is placed inside an ablation chamber and a laser beam is focused on the surface of the sample. When the ablation is started, the interaction between the pulsed laser and the sample surface produces a cloud of very small particles, which are transported from the ablation cell by a carrier gas into the ICP-MS for analysis.

4.3 An ICP-MS is used to quantify the elements of interest.

4.4 Quantitative analysis is accomplished using well-characterized glass standards whose major elemental composition is similar to the material to be analyzed.

4.5 A comparison between the reported elemental compositions of the known and recovered glass fragments may result in a decision on whether the samples are distinguishable by elemental composition or indistinguishable by elemental composition.

5. Significance and Use

5.1 This test method is useful for the determination of elemental concentrations in the microgram per gram ($\mu g g^{-1}$) to percent (%) levels in soda-lime glass samples. A standard test method may aid in the interchange of data between laboratories and in the creation and use of glass databases.

5.2 The determination of elemental concentrations in glass provides high discriminating value in the forensic comparison of glass fragments.

5.3 This test method produces minimal destruction of the sample. Microscopic craters of 50 to 100 µm in diameter by 80 to 150-µm deep are left in the glass fragment after analysis. The mass removed per replicate is approximately 0.4 to 3.1 µg.

5.4 Appropriate sampling techniques shall be used to account for natural heterogeneity of the materials at a microscopic scale.

5.5 The precision, accuracy, and limits of detection of the method (for each element measured) shall be established in each laboratory that employs the method. The measurement uncertainty of any concentration value used for a comparison shall be recorded with the concentration.

5.6 Acid digestion of glass followed by either Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) may also be used for trace elemental analysis of glass, and offer similar detection levels and the ability for quantitative analysis. However, these methods are destructive, and require larger sample sizes and much longer sample preparation times (Test Method E2330).

5.7 Micro X-Ray Fluorescence (µ-XRF) uses comparable sample sizes to those used for LA-ICP-MS with the advantage of being non-destructive of the sample. Some of the drawbacks of µ-XRF are poorer sensitivity and precision, and longer analysis time.

5.8 Scanning Electron Microscopy with EDS (SEM-EDS) is also available for elemental analysis, but it is of limited use for forensic glass source discrimination due to poor detection limits for higher atomic number elements present in glass at trace concentration levels. However, distinguishing between sources having similar RIs and densities is possible.

6. Apparatus

6.1 *LA-ICP-MS*—A Laser Ablation system coupled to an ICP-MS instrument is employed. Since there are several manufacturers for both laser ablation units and ICP-MS instruments, the instrument maker, model, configuration, and major operational parameters (that is, laser wavelength for the laser and mass selective detector type for the ICP-MS) of both instruments shall be noted within the analysis results. The most common laser wavelengths used for glass analysis are 266 nm, 213 nm, and 193 nm. Either quadrupole or magnetic sector ICP-MS instruments are suitable for this test method.

6.2 Prior to the analysis on the day it is used, the ICP-MS shall be tuned according to the manufacturer's recommendations covering the mass range of the elements to be measured. The instrument shall be adjusted for maximum sensitivity, best precision, and to minimize oxides and doubly charged ion interferences. The use of a glass reference material, such as NIST 612,[4] is recommended during the tuning and performance check. Detector cross calibrations (pulse/analog) shall

[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] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.