



D16 Bulk and Micro-Scale Trace Element Analysis of Glass Standard Reference Materials (SRMs) Using Modern Nuclear Analytical Methods and Laser Ablation-Inductively Coupled Plasma/Mass Spectrometry (LA-ICP/MS)

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Learning Overview: After attending this presentation, attendees will understand the importance of fit-for-purpose National Institute of Standards and Technology (NIST) SRMs for calibration of instrumentation for the forensic analysis of glass samples. The goal of this project is to provide the forensic science community with additional data about the suitability of currently available NIST SRMs for micro-scale analyses. This effort will also generate new concentration values for trace elements in existing SRMs. Additionally, the developed techniques for bulk and micro-scale analysis will be used to certify new trace-element glass reference materials specifically designed for use in forensic practice, such as the Organization of Scientific Area Committees (OSAC)-approved American Society for Testing and Materials (ASTM) E2927-16e1 standard test method for analysis and comparison of glass evidence.¹

Impact on the Forensic Science Community: This presentation will impact the forensic science community's understanding by providing additional information on currently available NIST SRMs, such as highlighting elements affected by potential heterogeneity biases and providing additional trace element abundance data.

Analysis of glass using micro-sampling techniques, such as micro X-Ray Fluorescence (μ -XRF) and LA-ICP/MS, is a key capability for many forensic laboratories. However, the NIST standard reference glasses that are currently available for instrument calibrations and quality control, such as NIST 612/4 and 1830/1, were not designed for forensic purposes. They were designed for chemical analysis of lunar material, such as the 61x series, or for major element analysis, 183x. Both types of analyses were performed on the bulk-scale, with hundreds of mg of material used for each analysis.^{2,3} When these materials are used as calibration standards for analyses conducted on the micro-scale, there are potential heterogeneity biases that will inherently affect the precision and accuracy of subsequently reported data.⁴

This study's goal is to re-analyze widely used NIST glass SRMs so that their trace element abundances are reported on a scale that is useful and appropriate for micro-scale forensic glass analyses through a combination of multiple analytical techniques. The first will involve the use of nuclear analytical techniques, such as instrumental neutron activation analysis and prompt gamma activation analysis, to analyze the bulk glass materials and determine additional forensically useful elements.^{5,6} The use of LA-ICP/MS will allow us to determine the degree of heterogeneity present on the glass wafers at the micro-scale.⁷ The combination of these two techniques will allow for the modification of existing, and provide new, trace-element data that is appropriate for the sample size of forensic analysis. The methods developed during this work will ultimately be used to analyze and report on the trace element abundance of new float glass standards designed to supplement the existing float glass standards (FGS glasses), with the goal to certify the new material as a NIST SRM specifically for the forensic community.³

Reference(s):

1. E2927-16-e1, Standard Test Method for the Determination of Trace Elements in Soda-Lime Glass Samples Using Laser Ablation Inductively Coupled Plasma Mass Spectrometry for Forensic Comparisons. *ASTM International*, (2016) DOI: 10.1520/E2927.
2. National Institute of Standards and Technology (NIST) *SRM 612 Trace Elements in Glass*, Department of Commerce, United States of America, 2012.
3. National Institute of Standards and Technology (NIST) *SRM Soda-Lime Sheet Glass*, Department of Commerce, United States of America, 2012.
4. Latkoczy, Christopher et al. Development and evaluation of a standard method for the quantitative determination of elements in float glass samples by LA-ICP-MS. *Journal of Forensic Science* 50.6 (2005): JFS2005091-15.
5. Koeberl, C. Instrumental neutron activation analysis of geochemical and cosmochemical samples: a fast and reliable method for small sample analysis. *Journal of Radioanalytical and Nuclear Chemistry* 168.1 (1993): 47-60.
6. Lindstrom, R. M. et al. Neutron capture prompt gamma-ray activation analysis at the NIST Cold Neutron Research Facility. *Journal of Radioanalytical and Nuclear Chemistry* 167.1 (1993): 121-126.
7. Trejos, T. et al. Sampling strategies for the analysis of glass fragments by LA-ICP-MS Part I. Micro-homogeneity study of glass and its application to the interpretation of forensic science. *Talanta* 67 (2005): 388-395.

Trace Elements, Instrumental Analysis, Glass