B162 Sampling Considerations in the Analysis of Glass Fragments by Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS)

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After attending this presentation, attendees will have an understanding of the advantages of the new technique of laser ablation ICPMS for the elemental analysis of glass fragments with a focus in the sampling strategies that should be taken into account during analysis.

The presentation will describe the advantages of the laser ablation sampling for ICP-MS analysis of glass evidence, including fast analysis time, excellent discrimination power and minimum consumption of sample. The presentation intends to give continuity to the results previously presented at AAFS. Different studies of interest to the forensic practitioner will be presented including sampling strategies and considerations for glass analysis by LA-ICP-MS.

The authors have previously reported the use of Laser Ablation ICP-MS as a powerful analytical tool for elemental analysis of glass. The advantages of LA-ICP-MS include fast, sensitive determinations of the trace metals in glass with a minimum destruction of the evidence. Glass fragments recovered from crime scenes are typically of 0.1 to 1mm in length and therefore in some cases the amount of sample is very limited to perform conventional digestion methods, which consume at least 2-3 mg of glass per replicate. LA-ICP-MS requires ten thousand times less glass consumption per analysis (~250 ng) and therefore the sample remains practically unaltered. However, due to the minute amount of material removed in LA, the analyst should be aware of special sampling considerations such as characterization of the glass fragments originating from the "known" source, fragment size and selection of the area and surface of ablation.

The fact that glass fragments collected from the crime scene and from suspect(s) and victim(s) are random in size does not affect bulk digestion analysis because sample is crushed and homogenized before it is weighed. During laser ablation, small craters (~50µm) are drilled into the solid material and therefore an investigation of the effect of the size of the fragment on the quantification of glass was conducted in order to determine if the interaction of the laser-surface changes significantly with the size of glass. Standard reference materials SRM 612 and SRM 610 were selected to conduct this part of the work in order to account for different concentration ranges and different opacity of the samples. The set under study was comprised of 7 fragments originating from each standard at different sizes and shapes ranging from 6 mm to 0.2 mm in length. The results show that there is not a significant difference in the elemental composition of fragments of different sizes. In addition, a homogeneity study of the elemental composition of glass samples was performed on glass matrices commonly found in crime scenes such as containers, architectural windows, windshields and headlamps. The set of glasses under study was comprised of 56 samples originating from glass containers, 28 samples from automobile windshields, 20 samples from architectural windowpanes and 20 samples from vehicle headlamps. All fragments were selected with a size smaller than 2 mm in length in order to simulate the typical glass fragments transferred from the crime scenarios. A CETAC LSX 500 Nd:YAG laser, 266nm, flat top beam profile was used in single point mode sampling 50 µm spot size for 50 seconds at 10 Hz (500 shots). The isotope ^{29}Si was used as an internal standard and the standard reference material, SRM NIST 612, was used as a single point for the external calibration. The standard SRM 621 was used as another control for the containers set, SRM 1411 for headlamps and SRM 1831 for the automobile and architectural window sets due to their very similar matrix with the samples of interest. For each set of glasses, the mean values and standard deviations of ten replicates (n=10) of a single fragment were compared with the values obtained from ten (n=10) different fragments of glass within the area of interest in order to evaluate whether or not the variation within a glass was larger than the variation due to the method. An additional subset of tempered glasses was examined to perform an elemental composition profile within different depths of the fragments. Single shot (one laser pulse per analysis) was also evaluated and its limitations for the forensic analysis of glass are also presented. The method presented for the analysis of glass by LA-ICP-MS had previously shown to possess the same or better performance than dissolution –ICP-MS methods in terms of accuracy, precision, limits of detection and discrimination power. The results show that glass is homogenous even at the micro-range level allowing LA-ICP-MS as an alternative technique to perform elemental analysis of glass. However, the variation of elemental composition of headlamps and containers is larger over the source than the instrumental variation due to inherent heterogeneity and therefore different statistical tools are recommended to fully characterize glass originating from the known source before the comparison analysis can be made. The proposed method allows reliable forensic comparisons of glass fragments by LA-ICP-MS independently of the fragment size recovered at scene.

Glass, Laser Ablation ICP-MS, Elemental Analysis

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