B40 Micro-Homogeneity Studies of Trace Elements in Solid Matrices by LA-ICP-MS: Implications for Forensic Comparisons

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After attending this presentation, attendees will understand how to apply micro-homogeneity studies of trace elements in solid matrices such as glass, paint, and soil to define sampling strategies and interpretation of this type of evidence.

This presentation will impact the forensic community and/or humanity by provide forensic examiners with useful information to apply LA-ICP-MS methods for the elemental analysis of glass, paint and soil. During this presentation participants will learn advantages and disadvantages of conducting LA-ICP-MS analysis for these matrices. Microheterogenetiry studies of these samples will assist forensic scientists in developing proper strategies for recovering, sampling, and interpreting the data.

Laser Ablation is a valuable tool for the direct solid sampling of trace evidence within the forensic context. Some of the advantages of LA sampling prior to ICP-MS analysis include elimination for the need of chemical procedures required for dissolution, reduced risk of contamination during the sample storage and sample preparation and vastly reduced consumption of the sample (ng vs mg for solution techniques).

These advantages make LA-ICP-MS a very attractive technique for the analysis of forensic samples, especially for trace examinations where the amount of sample always represents a challenge. The minimum amount of material that should be removed to be representative of the bulk sample may vary from matrix to matrix due to inherent degrees of micro-heterogeneity.

The aim of this work is to provide the forensic community with a critical evaluation of homogeneity of the elemental profile of some forensic samples at a micro-scale, as well as its implications to sampling size requirements, data analysis, and interpretation of results.

The micro-homogeneity of the elemental composition of glass samples (containers, architectural windows, and vehicle windshields) was studied in the types of samples commonly recovered from crime scenes. All fragments were selected with a size smaller than 2 mm^2 in order to simulate the typical glass fragments transferred from the crime scenarios. For each set of glasses, the mean values and standard deviation of 10 replicates (n = 10) of a single fragment were compared with the values obtained from 10 (n = 10) different fragments of glass within the area of interest (i.e., windshield pane, architectural pane and a single container) in order to evaluate whether or not the variation within a glass sample was larger than the variation due to the analytical method. In addition, several samples from a single manufacturer were analyzed. All the fragments were clear float glasses that were periodically gathered between May 1997 and September 2001.

Heterogeneity studies were conducted for the analysis of automotive and architectural paints. Two different sets of automotive paints were analyzed. The first set consisted of five blocks of red automobile paint purchased from ACT Laboratories; each block was ~ 15 cm x 10 cm. The five blocks originated from the same batch of paint and was composed of four layers. The second set was comprised of a piece of green paint (15 x 10 cm) and provided by the Royal Canadian Mounted Police (RCMP), Toronto, Canada). The sample was part of the Paint Database Query (PDQ) and consisted of six layers. For a homogeneity study within a single block, the piece was cut into 15 squared pieces of ~ 2 cm^2 and then 7 of those pieces were randomly selected for analysis. Four replicates per square/piece were analyzed for further statistical comparisons. An additional study was also conducted to determine the heterogeneity between the five blocks of paint. For this study, four replicates were measured on each block.

Raw soil material is significantly more heterogeneous when compared to the man-made glass and paint industrial materials described above. Therefore, homogenization of the soil standards and samples is required prior the analysis by the micro sampling tool of LA-ICP-MS.

The evaluation of homogeneity of soil and sediment matrices was conducted on pressed pellet standards using marine sediment reference material, PACS-2 (National Research Council of Canada, Ottawa, Canada) and soil reference material, SRM NIST 2710, Montana Soil (National Institute of Standards and Technology, Gaithersburg, MD, USA). The standard materials were mixed and homogenized with solid internal standards to improve accuracy and precision of the measurements. Three different ICP-MS instruments were used in standard operation modes: an HP-4500 (Agilent Technologies, Palo Alto, USA), an ELAN DRCII (PerkinElmer LAS, Shelton CT USA), and an Element 2 (Thermo Electron GmbH, Bremen, Germany). Two different laser units were utilized for this work: 1) a *New Wave UP- 213* operating at 213 nm (New Wave Research, USA) and 2) a *CETAC LSX*
500 (CETAC, USA) operating at 266 nm. A Scanning Electron Microscope with EDX detector (SEM/EDX) JSM-5900LV Jeol (JEOL, Japan) was used for the imaging of craters on different matrices and for determining particle size distributions within the sample.

The results demonstrate that float glass is homogenous even at the micro-range level. However, the variation of elemental composition of other glass types such containers, as well as paint and soil, is larger over the source

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than the instrumental variation due to inherent heterogeneity and therefore specific statistical methods are recommended to compare these more heterogeneous samples. Appropriate characterization of the heterogeneity of the control sample and incorporation of this variation into the match criteria allow LA-ICP-MS to be used as a favorable alternative technique to conduct elemental analysis of these matrices for comparison purposes.

Glass, Soil, Trace Evidence

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