B37 LA ICPMS and IRMS Isotopic and Other Investigations in Relation to a Safe Burglary

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After attending this presentation, attendees will appreciate the strong discriminating power of isotopic technique such as (LA) ICPMS and IRMS, not only for these specific safe burglary investigations but for a wide spectrum of other forensic investigations.

This presentation will impact the forensic community and/or humanity by making forensic community further aware of the strong power of LA ICPMS and IRMS isotopic techniques.

Safe wall filling material was released when an old safe was burglarized. During a search at the suspect's residence, visually similar material (mixture of glass like particles and sawdust) was retrieved from a plastic bag containing money. Small iron-containing particles were also retrieved from both samples. The safe wall filling material was found to consist of a mixture of alum or potassium alum ($KAl(SO_4)_2.12H_2O$) crystals (XRF/XRD) and sawdust (vis, FT-IR). All three material fractions (alum, sawdust, metal particles) were intercompared for the two samples.

The material combination of alum and sawdust appears very rare. Information was collected on the history of its use as safe wall filling, alternative applications of this material combination and how often Dutch police organisations encountered this material in the last two years. It was concluded that this material combination was only used for safe wall filling in the period before the 2nd World War, that no alternative applications were found and that no police organisation reported encountering this material combination.

Alum investigations: The glass like particles in both material samples were classified as alum. ICP AES results were obtained using a PE OPTIMA 3000 instrument. Apart from the major elements Al and K (S was not measured) Cr and Tl were observed at similar levels in both samples. Other elements observed (Fe, Sr,..) were present at much higher concentrations in the red-brown powder so that these are not confidently attributed to the alum samples.

LA ICPMS results using a PE ELAN 6100 DRC PLUS instrument were obtained on freshly cleaved inner alum crystal surfaces. Elements observed in both samples are Mg, Si, P, Ti, Cr, Cu, Zn, Ga, Rb, Sr, Sn, Ba, Tl and Pb.

IRMS services were provided by Iso-Analytical Ltd (Sandbach, UK) using standard methods on a Europa Scientific Geo 20-20 instrument with an EA-IRMS interface. Different isotope ratios were measured in separate experiments using different experimental configurations of the instrumentation. The mean $\delta^{34}S$ values for the suspect and crime scene alums (both 2.2 ± 0.1 ‰, n = 6) were undistinguishable using an experimental uncertainty of 0.24 ‰ (2s). This is compared to a variation range of -3.9 to +30.6 ‰ as reported [1] for $\delta^{34}S$ values obtained for a large number of alum minerals from various locations throughout the world and a variation range of 1.7 to 9.8 ‰ for a small test set of four different alum samples as obtained from alum suppliers in the Netherlands.

Sawdust investigations: The suspect and crime scene sawdust samples are visually similar and exhibit the same red brown color. A mixture of wood species, both soft and hard woods, was observed in both samples during botanical species investigations at the National Herbarium of the Netherlands. Over five species were identified in the suspect sample.

IRMS isotope ratios δ^2H, $\delta^{13}C$ en $\delta^{18}O$ for both the suspect and the crime scene sawdust fractions agree within the experimental uncertainty (2s, n=6). $\delta^{13}C$=-25,71+ 0,15 is e.g. compared to a variation range of -32 to -22 ‰ as reported for the $\delta^{13}C$ values of C3 plants [2]. Applicability of this information for the local situation is tested by collecting a limited set of sawdust samples in the Netherlands market and analysing these by IRMS. For $\delta^{13}C$ e.g. a variation range of -23 to -28 ‰ is measured.

Metal particles investigations: In both samples many minute metal particles were observed that were magnetically separated from the sample matrix. Corresponding variations in morphology of the particles (round balls of various sizes and curved lint like particles) indicate that metal particles in the two samples were formed through a similar process consistent with abrasive cutting of a safe [3]. µ-XRF (EAGLE) results for the round balls and lint particles show a high abundance of Fe as well as the presence of other elements such as Al, Si, P, S, Mn and Cu. Similar results were obtained for the fine metal particle fractions in both samples.

Conclusions: Combining the information from the various sources and the analytical results for the various material fractions (sawdust, alum, metal particles) provides a strongly discriminating method to compare the (combination of) material fractions as found in the two samples at the suspect's residence and at the scene of crime.

References

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ICPMS, IRMS, Safe

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