Characterizing Landfill Contamination

By Pb Isotope Ratio LA-MC-ICP-MS

In many parts of the UK, urban regeneration projects and reclamation of building and industrial land are key strands in strategies directed towards the provision of new housing developments. Landfills, mining waste deposits and other relics of past industrial activity have also become the focus of decontamination research and development. The work described here describes lead isotope multi-collector (MC) -ICP-MS analyses of landfill and brown-field sites samples.

Features of laser ablation (LA) - MC - ICP - MS:

- **Analysis of soil pellets, for bulk and trend analysis within the site.**
- **Lower DLs than solution methods, which have high extraction procedure blanks.**
- **Higher throughput than solution analysis.**

Landfill and brown-field samples

A common difficulty in landfill or brown-field site analysis is the number of different sources of lead contamination. Sampling and analysis methods are very important in achieving representative analysis. A range of urban soils (Fig.1) and contamination types were analysed as part of the UK Natural Environment Research Council funded Urban Regeneration (URGENT) Thematic Programme.

The maturity of the site influences the heterogeneity of the contamination. In some sites, the bio-available lead may reflect the contribution of petrol lead or UK ore lead at the site. In several mature sites, ore lead was the general background signature, but bio-available petrol lead still influenced the upper layer of the soil profile.

In immature contaminated sites, a complete mix of lead types can be found. Lead contamination can be derived from lead in petrol, paint, solder, pipes etc. In such sites, it is almost impossible to obtain a representative analysis in either solid or solution mode techniques, since the compositions of these sites are too heterogeneous.

Analysis strategies

(1) Sequential leaching and solution analysis.

The aim of this study was to identify the composition and bioavailability of different lead sources and compare them to the natural background signature. The soil samples were leached, using sequential leaching techniques and analysed by solution MC-ICP-MS.

Figure 1: A brown-field site in Nottingham and a landscaped landfill in Wolverhampton

(2) LA-MC-ICP-MS

Laser sampling was used to analyse individual particles and pressed powder pellets to identify end-member and dominant Pb-isotope signatures within the site. Particles were generally very complex (Fig.2), often containing a mixture of lead oxides, phosphates, elemental lead, silica calcite and graphite. As such, individual particles proved too heterogeneous to clearly resolve end-member components.

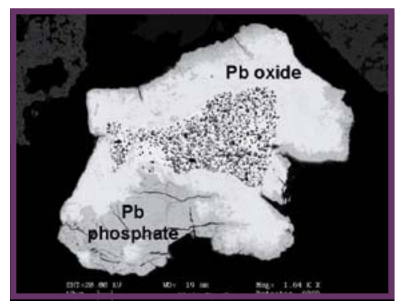

Figure 2 : WV6 -250+63 mm heavies

Bulk-sampling using pressed powder pellets proved very successful. The powder pellets were prepared exactly as for XRF analysis. The soils were dried and ashed, to eliminate organic compounds, then ground down to 250μm in order to homogenise the soil. The powders were then pressed, following normal XRF powder pressing routines. Smaller 5mm diameter pellets can be made in order to fit the maximum number of samples into the laser ablation cell. When preparing small pellets, the applied pressure can be very high and most materials can be crushed and compressed with no binder. Using smaller samples enhances the sample throughput from 8 samples per day to 20 per day.

NEW WAVE RESEARCH

To verify the viability of laser sampling, signatures obtained by solution and laser analysis were compared and showed good correlation across the range from Australian and UK petrol Pb, through to typical UK ore lead (background) isotopic compositions.

Samples for LA-ICP-MS

A large number of soil samples were taken from landfill and brown-field sites around Nottingham and Wolverhampton, as part of the NERC-URGENT programme. As expected, soil samples needed to be well homogenised, prior to pressing pellets for bulk analysis, so that a lead isotope composition analysis of a bulk material will represent all of the components present in that soil.

Standardization

A New Wave Research model UP266 (266nm Nd:YAG) laser was coupled to a Multi-collector ICP-MS. Instrument calibration was achieved using an international reference solution (NBS 981). Mass bias correction for each sample was achieved by simultaneously aspirating a thallium solution via a micro-concentric nebuliser, during ablation of the sample. Ablated and aspirated aerosols were introduced to the ICP source at the same time.

A secondary reference material was also required as an internal ablation standard. One of the analysed samples, NG2, a graveyard soil, proved to be very homogenous in its lead content and composition. It was therefore used as a quality control sample in between the analyses of unknowns and proved to be the most homogeneous of all the samples measured.

Each time a batch of samples was placed in the laser cell, NG2 was analysed first to verify system calibration. Repeatability of other sample analyses was generally greater than this check sample, due to the heterogeneity of the samples.

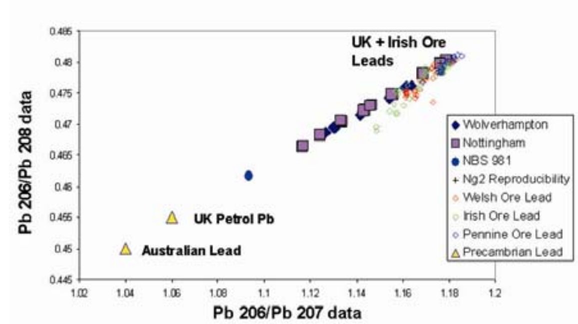

Figure 3: LA-PIMMS analysis of pressed powder pellets (1)

Summary

The study showed good potential for representative analysis of mature sites and can help establish the extent of lead source variability for a heavily contaminated site (McGill et al, 2003; Fig.3).

The advantages of LA-MC-ICP-MS include improved sample throughput, leading to fast, cost-effective and representative isotope ratio data. Sample preparation is simpler using laser ablation, compared to solution analyses and their associated extraction and dissolution procedures. High reagent blank Pb levels are also a serious disadvantage of extraction methods whilst laser ablation contributes virtually no blank to the analysis.

In this study, only lead isotopes were investigated, but other elements and isotope systems could be included in comparable methods.

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References

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