

Contaminant Source Apportionment in Brownfield Soils by Combined SEM Image Analyses and Plasma Ionisation, Multi-Collector Mass Spectrometry (PIMMS)

Rona A. R. McGill (rarmc@wpo.nerc.ac.uk)¹, Jonathan Pearce¹, Neil J. Fortey, John Watt³, Randall R. Parrish¹, Scott D. Young³ & Iain Thornton⁴

¹ NERC Isotope Geosciences Laboratory, British Geological Survey, Keyworth, NG12 5GG, UK

² Urban Pollution Research Centre, University of Middlesex, London

³ School of Biological Sciences, University of Nottingham, Nottingham

⁴ Environmental Geochemistry Research Group, Imperial College, London

Brownfield soils can contain lead-enriched particles from natural and anthropogenic sources. Lead is a potentially harmful substance therefore redevelopment of brownfield land, with elevated soil-lead concentrations, requires an understanding of source, pathway and receptor relationships to establish effective risk assessment and remediation strategies. Using a combination of imaging and geochemical techniques, a new procedure is being developed that is capable of determining the source of individual lead-bearing particles from contaminated and brownfield soils.

Soil samples from brownfield sites in Nottingham and Wolverhampton (UK) were analysed by XRF to determine total lead. Each XRF powder pellet was analysed by LA-PIMMS (Laser Ablation - Plasma Ionisation Multi-Collector Mass Spectrometry) to determine the lead isotope signature for the whole soil, a method that considerably reduces sample preparation time. Using the VG-Elemental UV-Microprobe II, each analysis comprised 50 ratios and was rastered over an area of 6 x 3 mm, using a repetition rate of 20Hz, 30-40% power and with a spot size of 400µm. Figure 1 is a plot of the Pb^{206}/Pb^{207} vs Pb^{206}/Pb^{208} ; data have been exponentially corrected to compensate for instrumental mass bias effects. The NBS 981 standard solution gives an external measurement precision of 64 ppm 2σ for Pb^{206}/Pb^{207} and 376 ppm 2σ for Pb^{206}/Pb^{208} , $n=14$. Repeat analyses of one XRF pellet (sample Ng2) determine the performance of a matrix matched standard; 2σ precision for 20 measurements was 254 ppm for Pb^{206}/Pb^{207} and 764 ppm for Pb^{206}/Pb^{208} (2σ precision for Pb^{206}/Pb^{204} measurements are 270 ppm for NBS 981 and 400 ppm for Ng2). All samples lie along an apparent two component mixing line (Figure 1), suggestive of two dominant contamination sources. Lead isotope studies on environmental materials (Sudgen et al., 1993, Croudace & Cundy, 1995, Bacon et al., 1996, Farmer et al., 2000) suggest that the two principal sources of lead contamination in the UK are alkyllead anti-knock additives in petrol (derived from Australian and Canadian ore deposits, Delves, 1988) and Pb from UK ore deposits. The Pb-isotope data obtained by LA-PIMMS from the Nottingham and Wolverhampton soil samples is consistent with lead derived from a range of admixtures of these two end members. Within

this range will lie sub-sources of lead, such as lead from water-pipes, roofing, paint, coal, incinerator ash and smelting slag. To determine which local Pb-sources are significant soil contaminants, a more detailed study of soil particles is required.

Focussing on two samples with high lead levels (Ng4 and Wv6), detailed SEM-imaging of the soil particulate was implemented. Each sample was split into 9 sub-samples, on the basis of particle size and density, and lead concentrations determined for each sub-sample. In both soils a large proportion of the lead was found in the finest (sub- 10µm) size fractions (39% for Ng4, and 63% for Wv6). The dense fractions generally contain higher concentrations of lead than the float fractions, but the dense fractions comprise a smaller proportion of the soil, and are therefore less significant to the soil Pb-budget than was expected. SEM image analysis using backscattered electron imagery coupled with X-ray microanalysis has identified Pb-rich paint flakes (Pb, Ti, Ba and chromate paints), Pb-solder, Pb-rich metal fragments, slag particles and Pb-Ca phosphates. In coarse float fractions, it was observed that sub-10µm Pb-rich particles adhered to larger grains, accounting for much of the lead in these fractions.

Samples Ng4 and Wv6 have very similar elevated lead levels yet the leachability of lead in the two samples has proven to be very different (C. Thums 2000, *pers comm.*). Mineralogy and isotopic analyses offer an opportunity to account for this difference in behaviour, in relation to different types of lead particles. Automated SEM image analysis allows significant populations of Pb-rich grains to be identified and pinpointed for further LA-PIMMS analyses. Individual Pb-bearing particles, of known speciation and derivation, can thus be analysed isotopically by laser ablation, providing the detail required to deconvolute the potential sources of contamination that give each soil its unique isotopic signature. This information is crucial to the determination of the pollution linkage relationships that result in soil contamination, and is thus important to the effective remediation of contaminated brownfield sites. This work forms part of NERC-URGENT project "Studies into metal speciation and bioavailability".

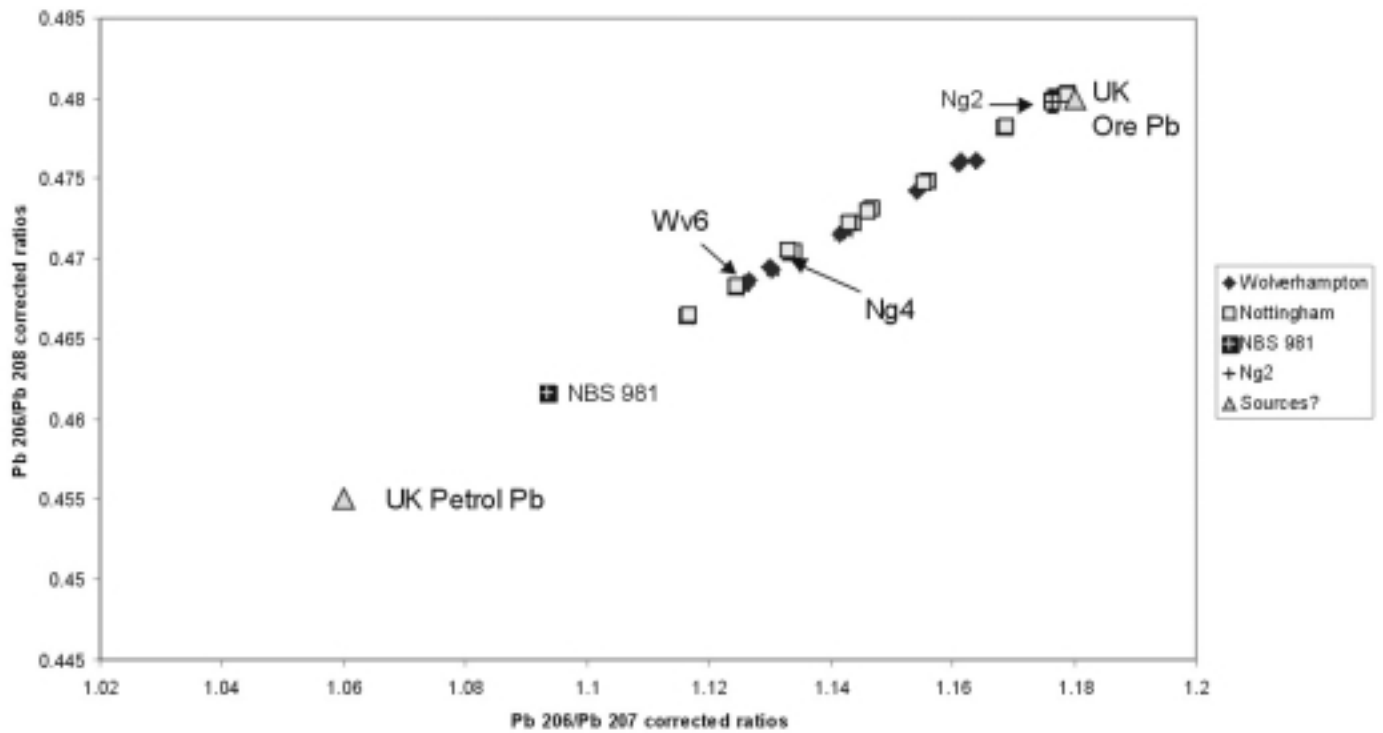


Figure 1, Plot of Pb_{206}/Pb_{207} vs Pb_{206}/Pb_{208} for Nottingham and Wolverhampton soil pellets. Analyses were obtained by LA-PIMMS and results plot along a two component mixing line.

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