



The optimization of microwave digestion procedures and application to an evaluation of potentially toxic element contamination on a former industrial site

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ABSTRACT

The optimization of a microwave dissolution procedure for potentially toxic elements in a contaminated soil sample has been evaluated using a central composite design approach. By considering the operating parameters of temperature, digestion time, microwave power and acid volume it was possible to identify the following optimum conditions: temperature, 160 °C; power, 750 W; digestion time, 25 min; and, an aqua regia volume of 13 mL. These microwave digestion conditions were then applied to 19 samples obtained from a former industrial site in Newcastle upon Tyne. Of the range of potentially toxic elements present at the site as a consequence of former industrial activity (As, Cd, Cr, Cu, Mo, Ni, Pb and Zn), the majority of topsoil samples indicated elevated concentrations of one or more of these elements. In particular, data obtained using either inductively coupled plasma mass spectrometry or flame atomic absorption spectroscopy indicates the high and wide concentration of Pb on the site (188–60300 mg/kg). Comparing the resulting potentially toxic elements data with UK soil guidelines values (SGVs) suggests at least parts of the site represent areas of potential human health risk. It was found that lead soil values exceeded the SGV on 17 out of the 19 sampling sites; similarly for As 6 out of 19 sampling sites exceeded the SGV. While for Cd, Cr and Ni the soil levels were below the stated SGVs.

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1. Introduction

Data on potentially toxic element (PTE) pollution in urban soils has increased over the last few decades as their significance in human health related issues has gained wider public and media attention [1–3]. Of particular concern in this paper is the importance of a former industrial site and the legacy that remains with respect to its current use as a country park for recreational activity. The site of the former St. Anthony's Lead Works lies to the east of Newcastle upon Tyne City Centre with an area of 52,651 m² [4]. The factory which operated from around 1840 to the mid 1930s smelted lead ore imported from Europe, mainly Spain and processed this along with United Kingdom produced lead. The factory refined lead ore which was then manufactured locally into white and red lead, sheet metal, and lead pipe. Historical maps also confirm a range of other industrial activities on and adjacent to the site including a copper/arsenic works, railway and tram lines, St. Anthony's Chemical works/Tars works (to the east) and Northumbrian Chemical/Paint works (to the SW on the opposite banks of the River Tyne). Consequently, the range of contaminants potentially present in sufficient concentrations to pose a risk to humans and

the wider environment includes As, Cd, Cr, Cu, Mo, Ni, Pb and Zn. The site was landscaped in the mid 1960s by Newcastle City Council and Newcastle University [4]. This involved a surface dressing of topsoil, tree planting on slopes and the establishment of grass on the flatter areas. The current uses of the site include: fishing on the riverside, ball games, walking, and dog exercise. The site is currently occupied by Walker Riverside Park, an area of public open space to the east of the city centre. Site surfaces include hard standing (footpath, road and car park) grassed and wooded areas. Although the majority of the site is vegetated, areas of bare ground are exposed, predominantly under the wooded slopes, where ash and clinker debris are evident.

Inductively coupled plasma mass spectrometry (ICP-MS) is a widely used analytical technique for the analysis of trace metals in samples whereas flame atomic absorption spectroscopy (FAAS) is often used for major elements. Prior to elemental analysis however, the soil samples must first be converted into a liquid form by acid digestion. Acid digestion procedures are often employed for the determination of elements in solid samples in order to transfer the analytes into solution prior to the determination step (e.g. ICP-MS, AAS) [5–12]. Acid digestion involves the use of acid and heat to destroy the matrix of a sample to liberate the metal content. It measures the (pseudo) total concentration of the metal present in the sample matrix. The goal of every digestion process is (a) dissolution of the sample matrix (but the totality can depend on the

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Table 1
Site description of St. Anthony's Lead Works [4].

Site name	The former St Anthony's Lead Works
Description of location	The site is situated next to both Felling View and Pottery Bank in the Walker area of Newcastle. (Grid reference: 428685E 562987E)
Site boundaries	North – disused railway track, east – Pottery Bank, south – River Tyne, west – open ground
Site setting	The site is situated on the banks of the River Tyne in a predominantly recreational area and consists of public open space
Adjacent land uses	Adjacent land uses includes educational, recreational and residential
Site shape	The site is roughly rectangular in shape
Site surfaces	This include hard standing (footpaths, car park and road) grassed, landscaped and planted areas
Vegetation	Vegetation present on the site includes grassed areas, shrubs and trees
Subsurface structures	This may include foundations, structures and various tanks associated with the sites former land uses
Relevant features	The River Tyne is located to the south of the site

choice/combination of acid), (b) avoidance of element losses and minimisation of contamination risk, and (c) reduction of handling and process times [11]. There are different approaches to heating the acid and sample in the digestion procedure including use of a hot plate/sample digestion block, and a microwave oven. The choice of acid and their combination is an important aspect. In this study acid digestion using aqua regia ($\text{HNO}_3:\text{HCl}$) (1:3, v/v) in combination with a microwave oven was used to extract the metals of interest from topsoils collected at the St. Anthony's Lead Work site (SAL) prior to determination.

The work presented in this paper proposed to (a) optimize the influence of microwave oven operating parameters on PTE recovery: acid volume (V), digestion time (t), microwave power input (P) and temperature (T) and (b) use the optimized microwave operating conditions and apply the approach to a range of soil samples from the former industrial site.

2. Experimental

2.1. Chemicals and apparatus

All chemicals used were of analytical grade. Concentrated hydrochloric acid and concentrated nitric acid were obtained from Fisher Scientific UK Ltd. (Loughborough, Leicestershire). A multi-element standard for As, Cd, Cr, Cu, Mo, Ni, Pb and Zn and an internal standard solution containing Sc, In, and Tb were purchased from SPEXCertPrep (Middlesex, UK). Ultra pure water of resistivity $18.2 \text{ M}\Omega \text{ cm}$ was produced by a Direct QTM Millipore System (Molsheim, France). Soil samples were collected from the former St. Anthony's Lead Works in Newcastle upon Tyne (Table 1). Two soil certified reference materials were used (GBW 07041 and SRM 2711) obtained from LGC-Promochem Ltd., London; SRM 2711 was originally sourced from agricultural soils close to a former smelting plant in Montana, USA. ICP-MS measurements were carried out with an ICP mass spectrometer X series II (Thermo Electron Corporation, Cheshire, UK). Flame atomic absorption spectroscopy (FAAS) measurements were carried out using an A. Analyst model 100 supplied by Perkin-Elmer Corporation, Norwalk, CT, USA. All digestions were carried out using a Start D Multiprep 42 High Throughput Rotor microwave digestion system supplied by Milestone Microwave Laboratory Systems UK.

3. Methodology

3.1. Sample collection and sample handling

Samples of soil were collected from 19 sample points on the site by digging a rectangular hole of about 10 cm deep to collect the topsoil. The sample points were selected to provide wide geographical coverage both within the known boundaries of the former site and its immediate periphery. The soil was put inside a sample bag, closed and labelled to include the sample point, date of collection and sample location (Fig. 1). During sampling, sample handling and

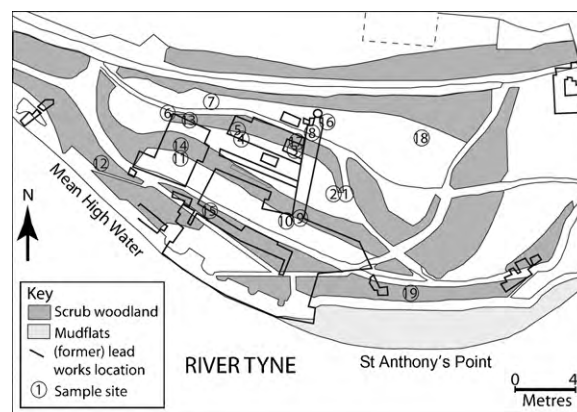


Fig. 1. Site map of St. Anthony's Lead Works (The map highlights the current surface topology. In addition, the historic position of the former Lead Works is highlighted. Sampling points are identified via the numerical key).

sample preparation polyethylene gloves were worn. The soil samples in the sample bag were dried in an oven at a temperature of 37°C for 6 days.

3.2. Sample preparation

The dried soil samples were first passed through a plastic sieve of mesh size $<2 \text{ mm}$ and stored. Sub-samples of the $<2 \text{ mm}$ fraction were powdered in a rotating ball mill and powdered aliquots were digested using the microwave digestion technique and then stored in a fridge (4°C) prior to metal analysis using either ICP-MS or FAAS.

3.3. Microwave digestion protocol

Central composite design (CCD) is required in order to establish the optimum conditions and also to obtain information about the inter-relationships between the variables [13]. A randomised central composite design was set up to obtain the various experimental conditions for each run. A total of 28 standard runs were obtained from the CCD. A maximum and minimum limit was chosen for each of the variables i.e. temperature, time, power and volume (Table 2). The use of a CCD allows all operating parameters to be investigated individually, as quadratic effects, and to consider interaction effects. The results of the CCD were evaluated using multilinear regression. The range of PTEs for which this technique has been applied are Cr, Cu, Mo, Ni, Pb and Zn.

A soil sample (0.5 g) was accurately weighed into a PFA (65 mL) vessel pre-cleaned with concentrated HNO_3 . Then aqua regia ($\text{HCl}:\text{HNO}_3$, 3:1, v/v) was added to the soil. The vessel was sealed with the TFM cover and placed inside a rotor of the microwave digestion system and submitted to a microwave dissolution program. The microwave oven was operated with a nominal capacity of 4 vessels per run. A ventilation (cooling) time of 30 min was allowed while the vessels were still in the oven. After cooling, the digest

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