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Estimation of uncertainty in the sampling and analysis of polychlorinated biphenyls and polycyclic aromatic hydrocarbons from contaminated soil in Brighton, UK



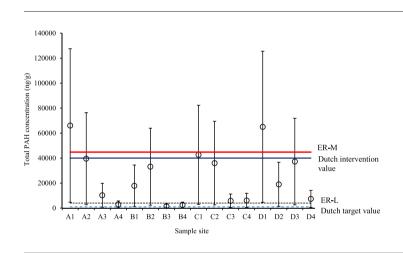
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HIGHLIGHTS

GRAPHICAL ABSTRACT

- A balanced sampling design was used for assessing soil contamination.
 Measurement uncertainty was domi-
- nated by sampling procedure.
- Measurement uncertainty can be reduced by using a high-resolution sampling.
- The soil guideline values were exceeded for PAHs and PCBs at selected sites.



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ABSTRACT

The heterogeneity of environmental samples is increasingly recognised, yet rarely examined in organic contamination investigations. In this study soil samples from an ex-landfill site in Brighton, UK were analysed for polychlorinated biphenyl (PCB) and polycyclic aromatic hydrocarbon (PAH) contamination by using a balanced sampling protocol. The analytical technique of gas chromatography–mass spectrometry was found to be fit for purpose by the use of duplicate samples and the statistical analysis of variances, as well as of certified reference materials. The sampling uncertainty was found to significantly overweigh the analytical uncertainty, by a factor of 3 and 6 for PCBs and PAHs, respectively. The soil samples showed a general trend of PCB concentration that was under the recommended target level of 20 ng/g dry weight. It is possible that one site alongside the main road may exceed the 20 ng/g target level, after taking into consideration the overall measurement uncertainty (70.8%). The PAH contamination was more severe, with seven sites potentially exceeding the effect-range medium concentrations. The soil samples with relatively high PCB and PAH concentrations were all taken from the grass verge, which also had the highest soil organic carbon content. The measurement uncertainty which was largely due to sampling can be reduced by sampling at a high resolution spacing of 17 m, which is recommended in future field investigations of soil organic contamination.

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1. Introduction

Environmental pollution by persistent organic pollutants (POPs) is the focus of attention worldwide, of which organochlorine pesticides, polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) have been widely studied (Zhou et al., 2001; Zhang et al., 2002; Maskaoui et al., 2005; Jiang et al., 2011; Cachada et al., 2012). PCBs are a family of 209 man-made congeners which were manufactured from 1929 until 1977. PAHs are composed of two or more fused benzene rings that have been found to be ubiquitous contaminants in the natural environment. PAHs are mainly derived from the incomplete combustion of organic material (Atkins et al., 2010; Cachada et al., 2012). Due to their chemical stability they are highly persistent and have become a global environment problem. The lower chlorinated PCBs are more soluble, mobile and volatile than the higher chlorinated PCBs. PCBs have been found in human and animal tissues in high levels in the Arctic and Antarctic where there are few if any point sources (Iwata et al., 1994; Glynn et al., 1995; Bard, 1999). It is likely that this contamination is due to the volatilisation of PCBs from the temperate regions, which are then transported by air currents and ocean circulation to the Arctic and Antarctic (Focardi et al., 1992). Weber and Goerke (2003) further suggested that Antarctic fish generally took highly lipophilic POPs primarily up from the food, leading to the high biomagnification factors of PCBs.

In addition to their persistence, PCBs and PAHs are known to be carcinogens in animals when they are given in high doses, and are suspected of being carcinogenic to human as well. This is manifested by the induction of DNA adduct formation and P450 in human, rat and avian liver cells following exposure to PCBs (Dubois et al., 1995). PCBs can also produce neurodevelopmental toxicity in human infants due possibly to interactions of PCBs and sex steroids (Winneke et al., 2002). The US Environmental Protection Agency (EPA, 1993) and Department of Health and Human Services (2011) have classified PCBs and PAHs as probable human carcinogens, in addition to their known adverse effects on the human immune, reproductive, nervous and endocrine systems.

The 62-acre ex-landfill site at Sheepcote Valley, to the east of Brighton, UK, was used by the British Army through the 19th century for manoeuvres and as a firing range. The Valley was first used as a landfill site in 1913 to receive household and highway refuse. In the 1930s and 1970s, incinerators were used on the site to burn wood and paper refuse. The site was never knowingly used for any toxic or industrial waste; however, the sheer volume of rubbish, 1450 m³/day in the 1970s (East Sussex County Council records), suggests that the real contents of the site were unknown. Dumping eventually ceased in 1990 when chalky soil from the excavation of Brighton's storm drains was used as a final capping. Although the site is monitored for gas emissions via the boreholes, there has never been any previous investigation into organic contamination on the site. Currently there are proposals for developing the site into a recreation ground, sports field or community farm, although no assessment has ever been made of the environmental quality in the area, hence the need for this study.

In addition, in any environmental measurement, there is always an element of uncertainty due to systematic and random errors which should be fully considered. Uncertainty may be defined as "a non-negative parameter characterising the dispersion of the quantity values being attributed to a measurand, based on the information used" (BIPM, 2012). Uncertainty may be estimated from calculations of precision and bias. Precision is defined as the closeness of agreement between indications or measured quantity values obtained by replicate measurements

on the same or similar objects under specified conditions (BIPM, 2012). Bias may be defined the estimate of a systematic measurement error (BIPM, 2012). In addition to analytical components, sampling procedure and sample preparation can also contribute to the overall uncertainty due to the inherent heterogeneity of most environmental media (Taylor et al., 2005). As a consequence, environmental measurement for quality assessment often has a degree of uncertainty which can lead to false negative or false positive classification (Taylor et al., 2005; Boon and Ramsey, 2012). To estimate the measurement uncertainty caused by sampling, the Eurachem guide (2007) introduced two approaches, empirical and modelling methods; these approaches can also be used in combination, if desired (Esbensen and Wagner, 2014). In the modelling method, the estimation of uncertainty involves the identification, quantification and summation of each potential source of uncertainty. However, this approach can become increasingly problematic in identifying all of such sources when it is applied to primary sampling (Lyn et al., 2007). The empirical approach to uncertainty estimation involves replicated measurements from either interorganisational trials and/or internal method validation and quality control, with a simpler method involving duplicating sampling and analysis by one organisation for a small proportion of sample population (Lyn et al., 2007). The objectives of this work were therefore to determine the contamination intensity, spatial variation and sources of PCBs and PAHs in the ex-landfill site; to compare the concentrations of PCBs and PAHs found in the soil samples with relevant soil guideline values; and to assess the uncertainty generated by sampling and analytical procedures and its impact on risk assessment.

2. Material and methods

2.1. Chemical standards

The standard solution (TCL PAH mix) containing 16 PAHs including naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo[*a*]anthracene, chrysene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, dibenz [*a*,*h*]anthracene, indeno[1,2,3-*cd*]pyrene and benzo[*ghi*]perylene was obtained from Supelco (UK). A deuterated internal standard (IS) mixture (EPA 525/525.1) containing acenaphthene-d₁₀, phenanthrene-d₁₀ and chrysene-d₁₂ was also obtained from Supelco. For PCBs, 14 PCB congeners (18, 28, 31, 44, 52, 101, 118, 138, 149, 153, 170, 180, 194, 209) were obtained from Supelco, whilst the ¹³C-labelled internal standards (CB-52, CB-153, CB-209) were purchased from Cambridge Isotope Laboratories, USA. Two certified reference materials (CRMs), LGC6113 (Laboratory of Government Chemists, UK) for PCBs, and CRM104 (Resource Technology Corporation, USA) for PAH were obtained. Organic solvents used were of glass-distilled grade (Rathburn Chemicals Ltd, Walkerburn, Scotland). High purity deionised water was taken from a Millipore system.

2.2. Sampling method

A 23,409 m² area of the Sheepcote Valley ex-landfill site was surveyed in May and October 2009 by taking samples using a 51 m regular grid (Fig. 1a). Soil samples from 20 cm deep were taken using a stainless steel auger and were sealed in ashed aluminium foil bags to prevent contamination. Within the sampling area, a total of 8 sites were randomly chosen, where a duplicate auger sample was taken 5 m (10% of the grid spacing as recommended) either north or south from the original locations using a balanced sampling design (Fig. 1b). The samples (48 in total) were all labelled properly, and on return to laboratory they were left in a fume cupboard to air

Fig. 1. a. Map showing the sampling sites and where duplicate samples were taken.b. Balanced experimental design for the estimation of measurement uncertainty.c. High-resolution sampling using 17-m grid. Duplicates are shown in red. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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