

## **A HIGH RESOLUTION MASS SPECTROMETRIC METHOD FOR THE DETERMINATION OF POLYCHLORINATED BIPHENYLS IN SEDIMENT BY ISOTOPE DILUTION**

*C. Peter Taylor, Paul McA. Harvey, Lindsey G. Mackay and Bernard King*

National Analytical Reference Laboratory  
Australian Government Analytical Laboratories, 1 Suakin St, Pymble, NSW, 2073

Polychlorinated biphenyls (PCBs) have been an important environmental concern for many years. The extensive use of PCBs, combined with high environmental persistence, low water solubility, and their potential toxicity to a wide range of organisms has resulted in widespread environmental contamination problems with considerable health concerns. Some of the major environmental sinks for these compounds are marine and lake sediments. The identification and quantitation of PCB congeners at very low concentrations in these sediments, and in the presence of other isomers and interferences is a challenging analytical problem.

As part of an international interlaboratory comparison study for the analysis of PCBs in sediment for the Consultative Committee for the Amount of Substance (CCQM), two marine sediments were studied, one was collected near urban areas in the middle Atlantic states of the USA, the other was a New York/New Jersey waterway sediment.

A congener-specific isotope dilution gas chromatographic/high resolution mass spectral technique was used to determine the accurate concentration of four PCB congeners, IUPAC numbers 28, 101, 153 and 170, in these sediments, and is presented, along with a comprehensive uncertainty budget. Several exhaustive extraction techniques are examined and compared, Soxhlet extraction, supercritical fluid extraction and enhanced solvent extraction. Separation of the PCB congener analytes from other interfering PCB congeners was achieved by the careful choice of specialty GC columns and/or the use of semi-preparative LC columns. The extracts were run on a HP6890 GC connected to a Finnigan MAT95 high resolution double-sector inverse geometry mass spectrometer at a resolution of 6000.

Isotopic Dilution Mass Spectrometry (IDMS)<sup>1,2</sup> analysis of these PCB congeners involves the addition of an isotopically-labelled analogue of each of the PCB analytes as an internal standard and is considered to be a candidate primary method of measurement. In this case <sup>13</sup>C<sub>12</sub>-labelled PCB congener analogues were employed. Provided the labelled analogue is fully equilibrated with the analyte, IDMS does not depend on sample recovery and can be tested for the presence of bias and interferences. By the use of an exact matching technique<sup>3,4</sup> the uncertainties involved in the method are substantially reduced. The uncertainty associated with a primary method such as this should be completely defined, so that the method may be confidently used for the certification of matrix reference materials.

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