

## **AN ISOTOPE DILUTION REFERENCE METHOD FOR THE DETERMINATION OF CADMIUM AND LEAD IN SEDIMENT BY ICP-MS**

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The development of a reference method for the analysis of lead and cadmium in sediment using isotope dilution mass spectrometry (IDMS) is presented. IDMS is considered to be a primary ratio method of measurement allowing high accuracy analysis to be performed with a well-defined uncertainty.<sup>1,2</sup>

Measurement was performed by exact matching isotope dilution mass spectrometry<sup>3</sup> using a Finnigan MAT Element high resolution ICP-MS, in low-resolution mode. Measurement precision was optimised using a low flow rate nebuliser (Glass Expansion, Micromist) and scanning parameters were set to minimise time spent between each isotope, therefore simulating simultaneous detection with a single detector instrument.

A complete digestion of sediment samples involving a rigorous microwave assisted HF/HNO<sub>3</sub> digestion was used. The HF/HNO<sub>3</sub> solution was evaporated after digestion, to reduce matrix effects and limit the dilution of the analytes in the solution. Of the many isobaric interferences for cadmium, the high levels of tin in sediment can be the most problematic. Therefore, a resin (AGX-10) clean-up method was employed to separate cadmium from tin.

Results for sediment certified reference materials were within the uncertainty on the certified values. The above method was used to quantify lead and cadmium in estuarine sediment during participation in an international interlaboratory comparison for reference laboratories. Results from this comparison will be presented.

1. P. De Bièvre, Fresenius J. Anal. Chem., 1990, **337**, 766-771.
  2. P. De Bièvre, H.S. Peiser, Fresenius J. Anal. Chem., 1997, **359**, 523-525.
  3. T. Catterick, B. Fairman and C. Harrington, J. Anal. Atom. Spectrom., 1998, **13**, 1009-1013
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