

HIGH ACCURACY ANALYSIS OF BUTYLTIN SPECIES IN SEDIMENT BY ISOTOPE DILUTION MASS SPECTROMETRY

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The National Analytical Reference Laboratory (NARL) has participated in several international intercomparisons with other national measurement institutes to demonstrate comparability in reference measurements at an international level. This comparability underpins Australia's claims for the measurement and provision of reference values for proficiency schemes or the certification of reference materials.

Tributyltin (TBT) chloride is a highly toxic chemical and environmental pollutant. Around 90% of all TBT produced is used in the maritime industry as an anti-fouling paint additive. TBT has been found to accumulate in marine organisms and sediment, especially in areas with high maritime traffic such as harbours and marinas. Current ANZECC guidelines specify a maximum of 70 ng/g as Sn of TBT in marine sediment (ANZECC, 2000).

An international intercomparison involving the analysis of TBT and its dibutyl equivalent, DBT, is currently being conducted between national measurement institutes, including NARL. Most of the participating laboratories are using an isotope dilution mass spectrometry (IDMS) technique, recognised as a potential primary ratio method capable of producing high accuracy results with traceability to the SI. IDMS involves the addition of a labelled internal standard prior to any sample preparation. After sample preparation and extraction, the ratio of labelled to unlabelled analyte in the sample is measured against the ratio in a standard, and the concentration of the analyte is then calculated. The IDMS technique adopted by NARL involves an exact-matching double-IDMS approach, which can minimise mass bias and linearity effects on the concentration determination.

NARL is developing two complementary analytical techniques that utilise isotopically enriched TBT and DBT species (eg ^{117}Sn -TBT and ^{119}Sn -DBT). Liquid Chromatography-Inductively Coupled Plasma/Mass Spectrometry (HPLC-ICP/MS) allows the direct measurement of organotin species, measuring the $^{120}/_{117}$ and $^{120}/_{119}$ ratios. Gas Chromatography/Mass Spectrometry (GC/MS) requires derivatisation of the sample extracts before analysis but affords a greater confirmation of identity. In this case ratios within the TBT and DBT cation clusters for SnBu_3^+ and SnBu_2^+ can be measured.
