
Pilot Study

CCQM-P18: Tributyltin in sediment

R. Sturgeon and R. Wahlen

Abstract. Pilot study CCQM-P18 was undertaken to assess the current capabilities of interested national metrology institutes (those which are members of the Consultative Committee for Amount of Substance) and selected outside 'expert' laboratories to quantitate $(C_4H_9)_3Sn^+$ (tributyltin, TBT) in a prepared marine sediment. This exercise was sanctioned by the 7th Meeting of the CCQM in April 2001 as an activity of the Inorganic Working Group, and was jointly piloted by the Institute for National Measurement Standards of the National Research Council of Canada and the Laboratory of the Government Chemist (LGC, UK). A total of fourteen laboratories initially indicated interest (ten NMIs and four external participants), from which results were submitted by seven of the NMIs and the four external laboratories. Two of the latter utilized a standard calibration approach based on a natural abundance TBT standard, whereas all NMIs relied on isotope dilution mass spectrometry for quantification. For this purpose, a species-specific ^{117}Sn -enriched TBT standard was supplied by the LGC. No sample-preparation methodology was prescribed by the pilot laboratories and thus a variety of approaches was adopted by the participants, including mechanical shaking, sonication, accelerated solvent extraction, microwave-assisted extraction and heating in combination with Grignard derivatization, ethylation and direct sampling. Detection techniques included ICP MS (with GC and HPLC sample introduction), GC-MS, GC-AED and GC-FPD. Recovery of TBT from a control standard (NRCC CRM PACS-2 marine sediment) averaged $(93.5 \pm 2.4) \%$ ($n = 14$). Results for the pilot material averaged $(0.680 \pm 0.015) \text{ nmol/g}$ ($n = 14$) with a median value of 0.676 nmol/g . Overall, performance was comparable to state-of-the-art expectations and the satisfactory agreement among participants allows a follow-up key comparison to be considered.

Main text

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R. Sturgeon: Institute for National Measurement Standards,
National Research Council of Canada, Ottawa, Canada.

R. Wahlen: Laboratory of the Government Chemist, Teddington,
Middlesex, UK.

This Final Report has been peer-reviewed and approved for
publication by the CCQM.