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Journal of Chromatography B, 783 (2003) 221–229

JOURNAL OF CHROMATOGRAPHY B

www.elsevier.com/locate/chromb

Comparison of different liquid chromatography conditions for the separation and analysis of organotin compounds in mussel and oyster tissue by liquid chromatography–inductively coupled plasma mass spectrometry

Raimund Wahlen*, Tim Catterick

LGC Ltd., Queens Road, Teddington, Middlesex TW11 0LY, UK

Received 7 May 2002; received in revised form 6 August 2002; accepted 5 September 2002

Abstract

In this paper, a new high-performance liquid chromatography–inductively coupled plasma mass spectrometry (HPLC–ICP-MS) methodology for the analysis of organotin compounds in complex matrices is described. Earlier studies had failed to show baseline resolution between dibutyltin (DBT) and triphenyltin (TPhT). The data presented in this paper show that, by using a different C-18 stationary phase material (Ace C-18) with decreased particle size, baseline resolution of DBT and TPhT can be achieved, with the resultant separation of a third interfering component. In addition, the Ace C-18 stationary phase yields a significant increase in the number of theoretical plates, and, combined with changes in the mobile phase composition, a reduction in run-time by ~25%. It is shown that the minor compounds detected are present in the sample and not artefacts of the analytical procedure. The accuracy and precision of the proposed HPLC–ICP-MS method was demonstrated for the determination of TBT in oyster tissue during the BCR ''MULSPOT'' international interlaboratory certification project.

Keywords: Organotin compounds

1. Introduction

Organotin compounds have been widely used in the production of polyvinylchloride (PVC) materials as heat and light stabilisers and as anti-bacterial and anti-fungal agents in pesticides and anti-fouling applications. Their toxicity [1], especially of tributyltin (TBT), in coastal environments has resulted in deleterious effects on non-target organisms, such as shellfish [2] and dogwhelk populations. Organotin compounds have been detected in most marine and freshwater sediment and water samples [3] as well as seafood destined for human consumption [4]. As a result, a wide range of methods has been developed over the years for extraction, separation and detection of organotin compounds. However, significant variations in results obtained by different methodologies in the literature have been highlighted [5,6] and at present there is still a scarcity of suitable reference materials for quality control purposes.

*Corresponding author. Tel.: +44-208-943-7000; fax: +44-208-943-2767.

E-mail address: raimund.wahlen@lgc.co.uk (R. Wahlen).

1570-0232/02/$ – see front matter
PII: S1570-0232(02)00662-1

Generally, the separation method of choice has been gas chromatography (GC). However, the derivatisation [7] required for GC analysis can result in variation in yields between species and in terms of efficiency depending on matrix components. Organotin separations by high-performance liquid chromatography (HPLC) [8,9] analysis have also been used. Liquid chromatography has been widely employed for organotin analysis and a comprehensive review of HPLC techniques is given by Ebdon et al. [10]. This approach offers the advantage that sample preparation does not involve a derivatisation step, which can eliminate a potential source of uncertainty in the final result. However, the range of compounds that can be analysed in a single run, and the number of detectors that give comparable sensitivity are limited compared to GC. Common HPLC detectors for organotin analysis include fluorescence [11], ultraviolet (UV) [12] and, more recently, inductively coupled plasma optical emission spectrometry (ICP-OES) [13], inductively coupled plasma mass spectrometry (ICP-MS) [8–10] and organic mass spectrometry detectors such as atmospheric pressure chemical ionisation mass spectrometry (APCI-MS–MS) [14] and electrospray ionisation mass spectrometry (ESI-MS) [15].

HPLC separations for organotin applications are more prone to insufficient resolution between analyte peaks [16], because the peak widths are generally broader than those encountered with GC. Therefore, it is imperative that efforts are made to maximise the chromatographic resolution for the HPLC-based methods. This need has become more evident as detection systems with improved sensitivity have revealed additional organotin components in matrix samples. This problem was encountered when the HPLC methodology developed in this laboratory by White et al. [9] was used with more sensitive detectors (MAT Finnigan Element and Agilent Technologies 7500i) and applied to the analysis of mussel tissue CRM477. This paper describes improvements made to the HPLC methodology developed by White et al. [9], based on the need to resolve a previously undetected peak from DBT. The original method employed a Kromasil C-18 reversed-phase column and is capable of separating DPhT, DBT, TPhT and TBT in 13–14 min. Although peak shape and resolution of these compounds was better than some other HPLC methods [13,17], DBT and TPhT were still not separated to achieve baseline resolution.

In this work, an accelerated solvent extraction method has been adapted from Arnold et al. [18], and used for the extraction of a mussel tissue (CRM 477, BCR). The proposed HPLC method uses an Ace C-18 3 μm stationary phase and produces a more efficient separation of the analytes in CRM477. The new conditions also result in a reduced run-time, which minimises solvent use and increases sample throughput, which is of importance for routine laboratories.

To underline the effectiveness of the newly developed method, it was used in an international interlaboratory comparison (MULPSOT study, BCR) with the opportunity to compare the quantitative data with other expert laboratories.

2. Experimental

2.1. Reagents

Acetonitrile (UpS ultra-purity solvent grade) was obtained from Romil (Cambridge, UK). Glacial acetic acid (TraceSelect) was obtained from Fluka (Gillingham, Dorset, UK). Triethylamine (TEA, HPLC grade) was obtained from BDH (Poole, UK). De-ionised water was obtained from a water purification unit at >18 MΩ (Elga, Marlow, UK). Methanol and hexane (HPLC grade) were obtained from Fisher (Fisher Scientific, Loughborough, UK) and anhydrous sodium acetate (Microselect ≥99.5% NT) from Fluka. Sodium tetra-ethylborate ($NaBEt_4$) was obtained from Aldrich (Gillingham, Dorset, UK).

Tributyltinchloride (TBTCl), dibutyltinchloride (DBTCl), triphenyltinchloride (TPhTCl) and diphenyltinchloride (DPhTCl) were obtained from Aldrich and purified according to the procedure described by Sutton et al. [19]. Monobutyltinchloride (MBTCl), di-*tert*.-butyltin dichloride (Tert-DBTCl) and tetrabutyltinchloride (TeBTCl) were obtained from Aldrich, and dioctyltin (DOT), tripropyltin (TPrT) and tetrapropyltin (TePrT) were obtained from Alfa Aesar (Johnson Matthey, Karlsruhe, Germany). The purity of TBTCl was determined to be $>99\%$ by differential scanning calorimetry (DSC).

The other standards were used for species identification purposes only.

The reference material CRM477 (BCR, Brussels), certified for the mass fractions of monobutyl, dibutyl and tributyltin in mussel tissue, was obtained from the Office of Reference Materials at LGC (Teddington, UK). This material was used to evaluate the developed methodologies.

2.2. HPLC–ICP-MS instrumentation

2.2.1. Preliminary work

Initial work was carried out on a Perkin-Elmer Elan 5000A ICP-MS (Beaconsfield, UK) instrument. This was coupled to a stand-alone Kontron Instruments 420 HPLC pump using the chromatographic parameters proposed by White et al. [9]. A MAT Finnigan Element 1 (Bremen, Germany) High Resolution ICP-MS (HR-ICP-MS) was used at the low-resolution, high-sensitivity setting with the same stand-alone pump and chromatographic conditions in order to determine interfering components. Manual injections were carried out using a syringe and a Rheodyne Model 9725 injector (Cotati, CA, USA) with a 50 μL PEEK sample loop. The establishment of these conditions is reported elsewhere [9].

2.2.2. Setup for chromatographic method development

An Agilent Technologies 1100 HPLC system (Palo Alto, CA, USA) was used for HPLC separations. The system consisted of a quaternary pump, a vacuum degasser, an autosampler and a heated column compartment. All stainless-steel parts of the HPLC system that come into contact with the sample were replaced by polyether ether ketone (PEEK) components. A 100 cm length piece of PEEK tubing (red) was used to connect the analytical column to the nebuliser of the ICP-MS.

An Agilent 7500i ICP-MS was used for time-resolved analysis of ^{120}Sn, ^{117}Sn and ^{103}Rh. A second roughing pump was added in-line to increase sensitivity by decreasing the interface vacuum pressure and therefore improving ion transmission. A 100 μL min^{-1} Agilent Micro Flow PFA concentric nebuliser was used to create the aerosol prior to introduction into the plasma. Typical ICP-MS conditions are given in Table 1.

Optimisation of the ICP-MS conditions was achieved prior to HPLC analysis by adjusting the torch position and tuning for reduced oxide and doubly charged ion formation with a standard tuning solution containing ^{7}Li, ^{89}Y, ^{140}Ce and ^{205}Tl in 2% HNO_3. After this preliminary optimisation, the HPLC system was coupled to the nebuliser and a final optimisation was carried out using ^{103}Rh added to the HPLC mobile phase. In order to reduce the solvent loading on the plasma, the double-pass spray-chamber was peltier cooled to −5 °C. Oxygen (0.1 L min^{-1}) was mixed into the make-up gas by a mass-flow controller and added post-nebulisation in order to convert organic carbon to CO_2 in the plasma and avoid carbon build-up on the cones. The final gas flow optimisation was important because the carrier gas and make-up gas flows had to be adjusted (using the tuning software) to ensure plasma stability

Table 1
ICP-MS conditions

	HPLC–ICP-MS	GC–ICP-MS
Cones	Platinum	Platinum
Plasma gas flow (L min^{-1})	14.5–14.9	14.5–14.9
Nebuliser gas flow (L min^{-1})	0.65–0.75	0.80-0.85
Make-up gas flow (L min^{-1})	0.15–0.25	Not used
RF power (W)	1350–1550	1100–1200
Sampling depth (mm)	4.0–7.0	6.5–7.5
Integration time per mass (ms)	300	100
Isotopes monitored	^{120}Sn, ^{117}Sn	^{120}Sn, ^{118}Sn, ^{117}Sn
Other parameters	Injector diameter, 1.5 mm; Peltier-cooled spray-chamber at −5 °C; 0.1 L min^{-1} O_2 added post-nebulisation; shield torch used	0.1 L min^{-1} O_2 added to enhance sensitivity; shield torch used

Table 2
HPLC conditions

Column	C-18 reversed-phase (15 cm×1.0 mm/2.1 mm I.D., 3 μm/5 μm particle size)
Mobile phase	Conditions A: 65:25:10 (v/v) acetonitrile–water–acetic acid with 0.05% TEA, pH 3.4±0.1 Conditions B and C: 65:23:12 (v/v) acetonitrile–water–acetic acid with 0.05% TEA, pH 3.1±0.1
Flow rate	0.2 mL min^{-1} (0.06 mL min^{-1} for 1 mm I.D. column)
Other parameters	All stainless-steel tubing and needle assembly replaced with PEEK (polyether ether ketone)

with the organic mobile phase conditions. The HPLC conditions are given in Table 2.

2.3. Chromatographic conditions

White et al. [9] used a Kromasil C-18 stationary phase (Hichrom, Reading, UK), with a particle size of 5 μm and column dimensions of 15 cm×2.1 mm I.D. (conditions A in Table 2). In order to improve the resolution between DBT and TPhT, several stationary phases and column dimensions were tested:

1. Conditions B—Ace C-18 3 μm particle size with a 15 cm×2.1 mm I.D. column.
2. Conditions C—Ace C-18 3 μm particle size with a 15 cm×1.0 mm I.D. column.

All Ace columns are manufactured by Advanced Chromatography Technologies and were obtained from Hichrom.

All chromatographic peaks were integrated manually using the Agilent Technologies ICP-MS chromatographic software (G1824C Version C.01.00). The column efficiency (theoretical plates), resolution and retention times were calculated for each chromatogram using the integration software of the Agilent Technologies LC–MSD ChemStation (Rev. A. 08.03 [847]).

2.4. GC–ICP-MS setup

GC separations were performed on an Agilent Technologies 6890 gas chromatograph. The GC interface used to couple the GC to the ICP-MS was an Agilent Technologies G3158A and consisted of a heated transfer line and heated injector. An Agilent Technologies 7500i ICP-MS was used for time-resolved analysis of ^{120}Sn, ^{118}Sn and ^{117}Sn. The GC method was based on that described by Rajendran et al. [7]. The column was a HP5 (30 m×0.32 mm I.D., 0.25 μm film thickness) with He as the carrier gas at 2 mL min^{-1} (controlled by electronic pressure control). The analytical column was connected to a length of de-activated fused silica, which was inserted along the ICP transfer line and injector. The injection volume was 1 μL. A pulsed splitless inlet mode was used at 250 °C. The GC interface injector was heated to 240 °C and the transfer line to 280 °C. The argon nebuliser gas for the ICP-MS was heated in a stainless-steel coil in the GC oven and transported along the transfer line and injector into the plasma.

After installation of the interface, the ICP-MS torch position and the ion lenses were tuned using a mixture of 100 ppm xenon in oxygen, which was added to the ICP-MS nebuliser gas at 0.1 L min^{-1} via a T-piece. The isotope monitored for this adjustment was ^{131}Xe. Table 1 shows the ICP-MS conditions used for GC–ICP-MS analysis. After this adjustment pure oxygen replaced the xenon/oxygen mixture.

2.5. Extraction of organotin compounds from CRM477

2.5.1. Accelerated solvent extraction

Accelerated solvent extraction (ASE) was carried out using a Dionex ASE 200 system (Dionex, Camberley, UK). Stainless-steel extraction cells (11 mL) were loaded with an inert diatomaceous material

(hydromatrix) as a dispersing agent. About 0.2 g of CRM477 were accurately weighed into the cells and mixed thoroughly with the dispersing agent. Each cell was extracted after Arnold et al. [18] using five repeat 5 min cycles at 100 °C and 1500 p.s.i. with 0.5 *M* anhydrous sodium acetate–1.0 *M* acetic acid in methanol after a 2 min pre-heat and 5 min heat cycle. Prior to analysis, the extracts were diluted about two- to five-fold in ultra-pure water for HPLC–ICP-MS analysis. For GC separations, 5 mL of the extract solution were derivatised for 10 min with 1 mL of 5% $NaBEt_4$ and shaken with 2 mL of hexane. The hexane layer was transferred to GC vials for GC–ICP-MS analysis.

2.5.2. Microwave extraction

In order to ascertain that the presence of the minor peaks was not a result of analyte transformation during the sample preparation procedure, a variety of extraction solutions (methanol, glacial acetic acid, 0.5 *M* acetic acid in methanol, 0.5 *M* HCl in methanol and the extraction solvent described for ASE extractions) were tested for the extraction of CRM477 by the closed vessel microwave extraction conditions proposed by Yang and Lam [20]. The microwave instrument used was a Paar Physica Multiwave (Perkin-Elmer, Beaconsfield, UK).

3. Results and discussion

Preliminary studies, which detected a number of previously undetected peaks, showed that the original method developed by White et al. [9] could be improved.

3.1. Chromatographic method development

In order to determine the effects of changes in chromatographic parameters, an Agilent Technologies 7500i ICP-MS was used coupled to an Agilent Technologies 1100 HPLC system. This setup provided the sensitivity needed to detect the interfering peaks in the sample, sufficient plasma stability and reproducible HPLC conditions combined with chromatographic software for accurate data interpretation. Chiron and co-workers [8] indicated that the use of methanol instead of acetonitrile resulted in better plasma stability at the expense of broader analyte peaks. This compromise was not necessary using the instrument setup described in this paper.

Fig. 1 shows a chromatogram for HPLC conditions A, coupled to the Agilent Technologies 7500i ICP-MS. Peaks for DPhT, DBT, TPhT and TBT, as well as two minor unidentified peaks [labelled ''Unknown 1'' (UK1) and ''Unknown 2'' (UK2)] are detected. Peak UK1 elutes on the tailing side of DBT and interferes with the quantitation of this compound. TPhT and TBT are sufficiently well resolved from UK1 and UK2 to allow accurate peak integration.

Attempts were made to increase the resolution between DBT and UK1 by sharpening the peak shape of DBT using a stationary phase with a lower nominal silanol activity, and a reduced particle size. An Ace C-18 3 μm, 15 cm×2.1 mm I.D. was selected for this purpose. Initial experiments showed that this approach yielded improved resolution between DBT, UK1 and TPhT as well as decreasing their retention times significantly. The composition of the mobile phase was then altered by increasing the volume of acetic acid and decreasing the volume of water, so as to decrease the pH from 3.4±0.1 to 3.1±0.1 in order to reduce retention times still further. Fig. 2 shows the result of the combined changes on the chromatography of the CRM477 extracts. The peak widths for the butyltin compounds are reduced significantly (~30 s) and there is a reduction in the retention times of all compounds

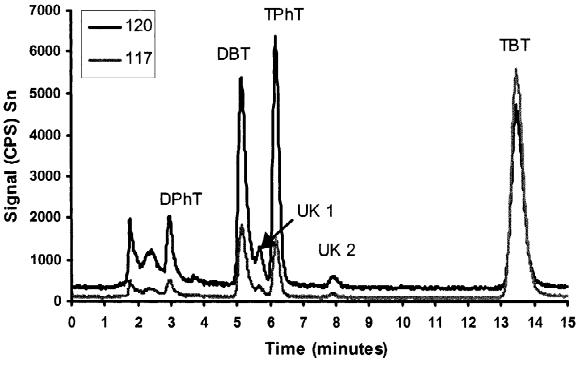

Fig. 1. Chromatogram of CRM477 using Kromasil C-18 5 μm, 15 cm×2.1 mm with an Agilent 7500i ICP-MS detector. Amount of TBT injected: 38 pg as Sn.

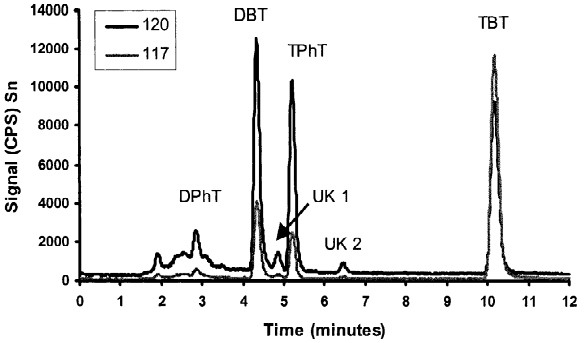

Fig. 2. Chromatogram of CRM477 using ACE C-18 3 μm, 15 cm×2.1 mm coupled to an Agilent 7500i ICP-MS. Amount of TBT injected: 32 pg as Sn.

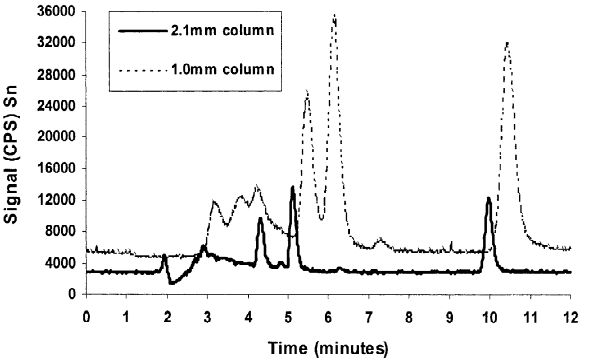

Fig. 3. Chromatogram of 20 μL injections of the same sample onto 15 cm Ace columns of 2.1 and 1.0 mm internal diameters. Amount of TBT injected: 46 pg as Sn.

(Table 3). The TBT detection limit for this method based on injections of a single standard calibration solution was estimated as 40 pg g^{-1} (as Sn).

Based on these improvements in resolution and reduced retention time, an attempt was made to increase sensitivity by using the same Ace stationary phase with a reduced column diameter of 1.0 mm. Experiments using this column (conditions C) showed that, after optimisation of the mobile phase flow-rate to 0.06 mL min^{-1}, there was a significant increase in the analyte signal (Fig. 3). However, the improvements in resolution between DBT and TPhT were reversed, and this approach was therefore not pursued for the separation of the analytes in this sample. In order to evaluate the differences between the HPLC conditions tested in quantitative terms, the column efficiency (theoretical plates), resolution and retention times were calculated for each chromatogram using the Integration software of the Agilent Technologies LC–MSD ChemStation (Rev. A. 08.03 [847]). These data are summarised in Tables 3–5.

Table 3
Retention times (min)

	Kromasil C-18 (2.1 mm) Conditions A	ACE C-18 (2.1 mm) Conditions B
DBT	5.14	4.34
UK 1	5.68	4.86
TPhT	6.18	5.21
UK 2	7.94	6.46
TBT	13.48	10.17

3.1.1. Column efficiency and peak resolution

The number of theoretical plates gives an indication of the column efficiency and was calculated by the half-width method. Table 4 shows a significant increase in the number of theoretical plates calculated for each of the compounds when the sample is analysed using HPLC condition B. The column efficiency is 1.4–2.1 times greater for the main

Table 4
Theoretical plates (half-width method)

	Kromasil C-18 (2.1 mm) Conditions A	ACE C-18 (2.1 mm) Conditions B	Factor increase
DBT	2257	4352	1.9
UK 1	2245	6118	2.7
TPhT	4973	6932	1.4
UK 2	5798	10 615	1.8
TBT	6662	14 134	2.1

Table 5
Resolution from preceding peak

	Kromasil C-18 (2.1 mm) Conditions A	ACE C-18 (2.1 mm) Conditions B	Factor increase
DBT	–	–	–
UK 1	1.19	2.04	1.7
TPhT	1.20	1.39	1.2
UK 2	4.57	5.00	1.1
TBT	10.27	12.48	1.2

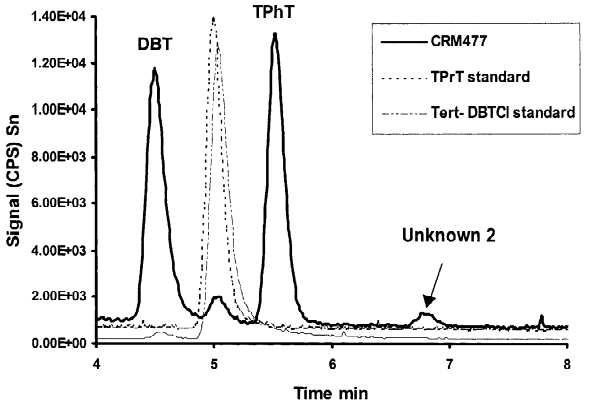

Fig. 4. Retention time matching of peak UK1 with standard solutions.

analytes in CRM477 using the ACE 3C-18 2.1 mm I.D. column, compared to the Kromasil C-18 stationary phase tested. This increase is probably due to a combination of the different C-18 material and the smaller particle size of the stationary phase.

Table 5 shows that resolution is increased for all compounds using conditions B compared to conditions A. The most significant increase in resolution is seen between DBT and Peak UK1 from 1.19, using the HPLC method by White and co-workers, to 2.04, using the ACE 3C-18 column.

3.2. Identification of the unknown compounds detected

The experiments carried out to ascertain whether the minor peaks were an artefact of the sample preparation using five different extraction solvents and two different extraction techniques showed that peaks UK1 and UK2 were present in all of the sample extracts, but not in the blank or standard extracts. In order to help identify peaks UK1 and UK2, a variety of single organotin standards were analysed using HPLC conditions B and GC–ICP-MS in an attempt to obtain a retention time match. Single standard solutions of MBTCl, Tert-DBTCl, TeBTCl, DOT, TPrT and TePrT were injected. The only retention time matches obtained by HPLC–ICP-MS were Tert-DBTCl and TPrT for peak UK1, as shown in Fig. 4. No retention time matches were obtained for peak UK2. However, when using GC–ICP-MS, TPrT and Tert-DBTCl both eluted with a retention time of 5.90 to 5.92 min and neither the extracted standard nor the CRM477 extract showed a signal at that retention time. Fig. 5 shows GC–ICP-MS

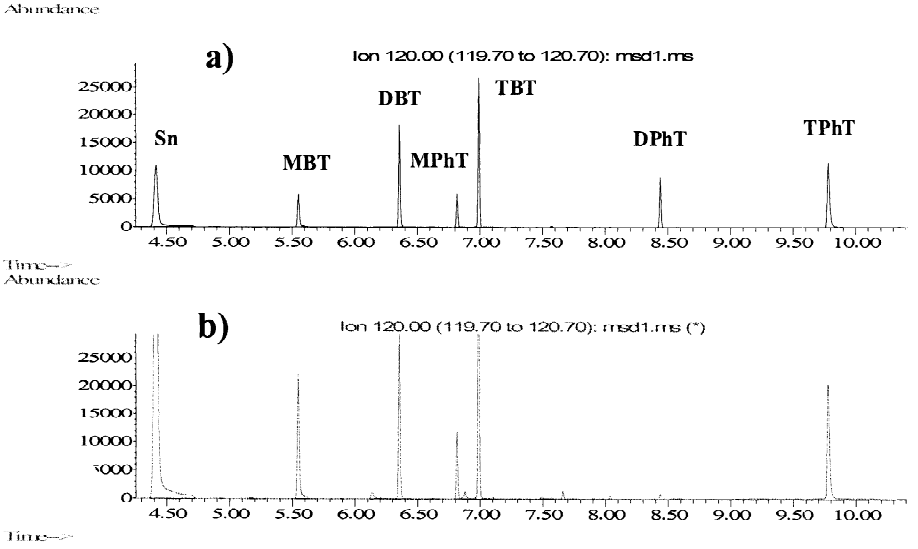

Fig. 5. GC–ICP-MS chromatogram of (a) a standard solution containing MBT (29 ng), DBT (21 ng), TBT (27 ng), MPhT (29 ng), DPhT (21 ng) and TPhT (14 ng) as Sn after ASE extraction, and (b) a CRM477 extract (*x*-axis, time (min); *y*-axis, ^{120}Sn counts).

chromatograms for (a) a mixed standard solution containing MBT, DBT, TBT, MPhT, DPhT and TPhT after extraction by ASE and (b) an ASE extract of CRM477. From Fig. 5a it can be seen that the extraction and derivatisation of the six organotin species in the standard solution does not lead to formation of the unidentified species. However, as shown in Fig. 5b, four minor peaks with retention times of 6.1, 6.9, 7.6 and 8.0 min were detected in the CRM477 extract.

The identification of an unknown compound can be achieved by the use of ESI-MS–MS, which can provide structural information by means of the molecular and product ions of a particular analyte. Unfortunately, the sensitivity of ESI-MS–MS systems is still generally inferior to ICP-MS for most organometallic speciation applications and this in combination with the small signals obtained for the unknown peaks by ICP-MS made this approach unsuitable. No conclusive identification of the unidentified compounds was therefore achieved.

3.3. Quantitative method validation data

The main purpose of this paper was to offer a solution to difficulties encountered during the chromatographic separation of organotin compounds in the mussel tissue CRM477. However, its ability to form the basis for sound quantitation has been evaluated in an international laboratory intercomparison. Using accelerated solvent extraction, the HPLC–ICP-MS conditions proposed in this paper were successfully employed for the determination of TBT in oyster tissue using species-specific isotope dilution analysis during the BCR ''MULSPOT'' certification project[1]. The mean value and standard deviation obtained by the described method was 131 ± 3 μg kg^{-1} (as TBT^+) ($n=7$) compared to a mean value and standard deviation obtained by nine different European laboratories of 133 ± 25 μg kg^{-1} (as TBT^+) ($n=68$). Thus the quantitative value reported using this method shows excellent agreement with the consensus mean.

4. Conclusions

The HPLC conditions proposed for the separation of DBT, TPhT, TBT and two unknown compounds and subsequent determination by ICP-MS are shown to yield increases in resolution for all five peaks analysed in CRM477 when compared to the conditions proposed by White et al. [9]. In addition, the column efficiency as measured by the number of theoretical plates is increased by a factor of 1.4–2.7. The retention times for all compounds are also reduced and this leads to a decrease in the total run-time of ~25%.

The presence of two to four minor unidentified organotin compounds in CRM477 was verified using a combination of two separate extraction techniques, five separate extraction solvents and chromatographic separation of the extracts by both HPLC and GC. No conclusive identification of the unknown compounds was achieved.

The accuracy and precision of the HPLC–ICP-MS method was demonstrated by the results obtained for TBT during the BCR ''MULSPOT'' interlaboratory certification study using an oyster tissue, where the value obtained by the reported method [131 ± 3 μg kg^{-1} (as TBT^+)] agreed extremely well with a consensus mean of 133 ± 25 μg kg^{-1} (as TBT^+) for nine other European expert laboratories.

Acknowledgements

Paul Norris (LGC, Teddington) kindly prepared samples by accelerated solvent extraction. Philippe Quevauviller and Soren Bowadt from the European Commission and Roberto Morabito (coordinator of the MULSPOT Project) from ENEA (Italy) are acknowledged. The contract number for the work carried out during the BCR ''MULSPOT'' certification programme was SMT4-CT98-2232. The work described in this paper was supported under contract by the UK Department of Trade and Industry as part of the National Measurement System Valid Analytical Measurement (VAM) program.

[1]The ''MULSPOT'' (Preparation and certification of an oyster reference material for species of Sn, Hg and As) project has been financed by the SM&T Programme (EU) (contract SMT4-CT98-2232) and coordinated by ENEA (IT). The project is at the certification stage and the material is not yet available on the market.

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