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Journal of Chromatography A, 718 (1995) 329–338

JOURNAL OF CHROMATOGRAPHY A

Determination of triorganotin compounds by ion chromatography and capillary electrophoresis with preconcentration using solid-phase extraction

Ewa Poboży, Bronislaw Głód, Joanna Kaniewska, Marek Trojanowicz*

Department of Chemistry, University of Warsaw, Pasteura 1, 02-093 Warsaw, Poland

First received 12 December 1994; revised manuscript received 9 June 1995; accepted 19 June 1995

Abstract

Procedures for the determination of triorganotins using HPLC with a cation-exchange column and capillary electrophoresis (CE) were optimized. In both cases indirect UV detection for trimethyl-, triethyl- and tributyltin and direct UV sensing of triphenyltin were applied. In CE the best separation of tributyl- and triphenyltin was obtained with the use of tartaric acid in the electrolyte. With both procedures a comparable analysis time was found, whereas a better separation of analytes and much lower detection limits were obtained for CE determination. Among five different commercial non-polar sorbents examined for solid-phase extraction of TBT and TPT, the best results were obtained with XAD-2.

1. Introduction

Although at present the determination of the total content of metals at trace level is predominated by atomic spectrometric methods, there is increasing interest in using chromatographic methods for this purpose [1,2]. A substantial part of such a procedure, including all areas of chromatography (GC, HPLC, TLC, SFC), often involves complexation of the metal ion in some form. Chromatographic methods, either with conventional detectors or especially coupled with atomic spectrometric detection, are most commonly applied in the determination of the speciation of trace metals, including organometallic compounds [3].

Awareness of the environmental impact of organometallic compounds has intensified since the 1970s. One of the most hazardous species are organometallic compounds of tin [4]. Industrial applications are based on diorgano- and triorganotins, which are widely used in agriculture as pesticides, in wood preservative formulations, in marine paints as antifoulants and as PVC stabilizers [5]. Owing to their biocidal properties in agriculture, most activity and toxicity of organotin compounds is associated. The most important organotin in the aquatic environment is tributyltin (TBT), the active ingredient in antifouling paint, although pollution by triphenyltin (TPT), used as a non-selective pesticide, is also a serious problem since it accumulates in lipophilic tissues of fishes [6].

Although in the speciation determination of tin, especially organometallic compounds, most often chromatographic methods are used, the

* Corresponding author.

SSDI 0021-9673(95)00696-6

literature also provides numerous examples of other procedures. TBT and TPT can be preconcentrated on carbon black, then eluted and separated on a small silica gel column and eluted with different solvents for electrothermal atomic absorption spectrometer (AAS) detection [7]. Several attempts have been published on the conversion of organotin compounds into the volatile hydride, cryogenic trapping and separation by warming on the basis of different boiling points for different hydrides [7–9]. Different butyltin species can be directly determined in the organic extractant by alternating current polarography [10].

Organotin compounds generally exhibit sufficient volatility for separation by GC, although the drawback of GC separation is the necessity for derivatization of organotin compounds to more stable and less reactive species by alkylation or hydridization. Numerous papers have been published on the coupling of GC with atomic spectrometry (see Ref. [11] and references cited therein).

The HPLC separation of organotin species has definite advantages over GC, such as the lack of need for derivatization, the variety of stationary and mobile phases available and the possibility of separation at ambient temperature, reducing possible losses of analytes during the separation process. The first paper on the HPLC of organotins was published by Brinckman et al. [12]. The HPLC separation of organotin species can also be achieved using reversed-phase systems [12–15] in addition to normal-phase systems with cyanopropyl columns [16–19]. Owing to the ionicity of alkyltin compounds, most often the separation of these species is carried out using strong cation-exchange columns [20–31]. As detection methods in HPLC mostly AAS is used with flame atomization [22], hydride generation [25] and especially with electrothermal atomization [12,18–20,25,27]. Recently several workers have developed HPLC systems coupled with inductively coupled plasma atomic emission spectrometric detection (ICP-AES) for organotin determination [15,26,28,30,31]. Laser-excited atomic fluorescence in a flame has also been employed for detection [26]. Spectrophotometry in the visible [13] or UV [24] region has occasionally been employed, but numerous examples can be found of the use of spectrofluorimetric detection of tin complexes with morin for tin speciation [14,16,17,23,28]. Amperometric detection in the differential-pulse mode has limited application [21].

Among the most advanced techniques of LC, organotins have been extracted and chromatographed under supercritical fluid conditions with formic acid-modified carbon dioxide [32]. Capillary electrophoresis has so far been applied only in the determination of organometallic compounds of lead and selenium [33].

The aim of this work was to compare the resolution and detectability obtained by HPLC separation on a cation-exchange column with indirect UV detection with those obtained by capillary electrophoresis for selected triorganotin compounds. Also, the possibility of preconcentration of those species by solid-phase extraction with several non-polar sorbents was examined.

2. Experimental

2.1. Apparatus

The HPLC equipment consisted of a Knauer isocratic HPLC system with a UV–Vis detector and a Whatman Partisil SCX-10 strong cation-exchange column (250 × 4.6 mm, 10 μm). As the optimum eluent, a 70:30 mixture of methanol with 10 m*M* acetate buffer (pH 5.9) containing 2 m*M* benzyltrimethylammonium chloride (BTMA) was used.

For capillary electrophoretic (CE) measurements, a Model 3850 system from Isco equipped with a quartz capillary (75 μm I.D., total length 60 or 100 cm and length to the detector 35 or 60 cm, respectively), was used with split-vent tubing sample introduction. Measurements were carried out at 10 μA current and a rise time 1.6 s. The effective sample volume was calculated according to the manufacturer's recommendation.

2.2. Reagents

Tributyltin chloride (TBT) and triphenyltin chloride (TPT) were obtained from Schering

Industrie-Chemikalien and trimethyltin chloride (TMT), triethyltin chloride (TET) and benzyltrimethylammonium chloride (BTMA) from Aldrich. The organotin compounds were a kind gift from Dr. N. Buschmann (Department of Chemistry, Westfalian Wilhelms University, Munster, Germany). D-(1S)-(+)-Camphorsulfonic acid was purchased from Sigma and β-cyclodextrin from Kodak. HPLC-purity methanol was obtained from Baker. Other reagents were of analytical-reagent grade from POCh (Gliwice, Poland).

Solid-phase extraction was performed using laboratory-made microcolumns (40 × 7 mm I.D.) with C_{18}, C_8 and phenyl functionalized sorbents from Baker and Amberlite XAD-2 and XAD-4 resins from Aldrich. Elution from the preconcentration column was carried out with 10 ml of methanol and the extract obtained was evaporated to 2 ml. For a 1-l sample volume a 500-fold preconcentration was obtained with such a procedure. The comparison of the effectiveness of preconcentration using different sorbents was conducted at a flow-rate of 7 ml/min during the preconcentration phase.

3. Results and discussion

3.1. HPLC determination of triorganotins

In this study, the simplest possible HPLC system which is possible for the determination of organotins was employed, with isocratic elution, a cation-exchange column and UV detection. Because of all the triorganotins considered only TPT strongly absorbs UV radiation, in all HPLC measurements indirect vacancy detection was employed with addition of a low concentration of BTMA to the eluent solution, with a UV absorbance maximum at 262 nm.

Owing to their environmental importance, the main task in the optimization of the analytical procedure was to establish best conditions for the determination of TBT and TPT. These two species are difficult to resolve by cation-exchange chromatography and in all the publications cited only two provided some results on the determination of TBT and TPT [20,24]. In both of them 10 m*M* ammonium acetate–methanol (70:30) was used as the eluent with a Whatman Partisil 10 SCX column.

The addition of BTMA to the eluent not only allows the indirect detection of trialkyltins, but also, owing to the presence of the large hydrophobic cation, it strongly influences the retention of species to be separated. The reversed order of elution of TBT and TPT after addition of BTMA to acetate–methanol eluent has been reported previously [24]. An increase in the BTMA concentration in the eluent results in a decrease in the retention times of analytes and in some improvement in the resolution (Fig. 1A); however; it is also associated with a significant drop in the magnitude of the signal (Fig. 1B). As the optimum, a BTMA concentration in the eluent of 2 m*M* was assumed. One can expect also that in the separation of triorganotins, some contribution from non-ionic interactions of analytes with the hydrocarbon part of the stationary phase may take place. This contribution depends essentially on the content of the organic solvent in the eluent as was demonstrated earlier [20]. In this study it was observed that the use of either a higher or lower content of methanol, although it changes the retention times, does not improve the separation of TBT and TPT.

The chromatogram of the mixture of triorganotins under optimum conditions is shown in Fig. 2. The separation between TBT and TPT is poor, but for measurements of peak height for both species at different concentrations linear relationships were found between peak height and concentration. In comparison with earlier applications of the same column and similar chromatographic conditions [20,24], at a retention time of about 20 min a large system peak was observed, which did not overlap with any peak of the determined tin species. An increase in BTMA concentration causes a shift towards shorter retention times.

The sensitivity of HPLC determination is very different for different triorganotins. The calibration plots exhibited slopes of $8.1 \cdot 10^{-5}$ and $2.0 \cdot 10^{-5}$ AU/mg · ml^{-1} for TPT and TBT, respectively. The sensitivity for the determination of the two remaining trialkyltins was about one order of magnitude lower with slopes of the

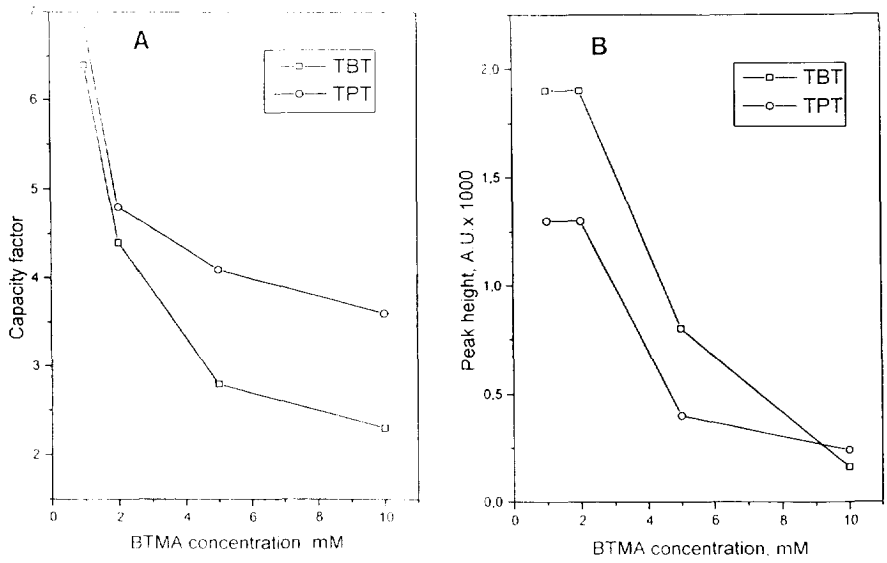

Fig. 1. Effect of BTMA concentration in methanol–10 m*M* acetate buffer (pH 5.9) (70:30 v/v) eluent on (A) retention times and (B) signal magnitude for TBT and TPT. Flow-rate, 1.0 ml/min; injection, 200 μl of solution containing 10 mg/l TBT and 1.5 mg/l TPT.

calibration plots of $4.8 \cdot 10^{-6}$ and $2.6 \cdot 10^{-6}$ AU/mg $\cdot$ ml^{-1} for TET and TMT, respectively. The detection limits calculated in relation to the amplitude of the baseline noise are given in Table 1. They are improved in comparison with earlier work [24], taking into account that previous data were reported for the procedure involving sorbent extraction preconcentration. For comparison, data on the detectability of TBT and TPT obtained by other workers for HPLC procedures with different detectors are shown in Table 2. Significantly lower detection limits for both analytes in HPLC determinations can be obtained by coupling of HPLC with expensive atomic spectrometric instrumentation.

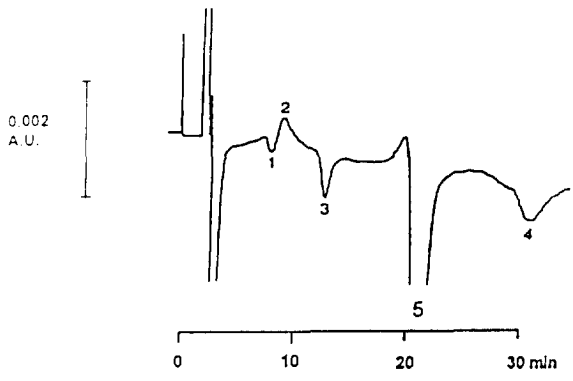

Fig. 2. Chromatogram obtained for a mixture containing (1) 2.0 mg/l TBT, (2) 0.5 mg/l TPT, (3) 15 mg/l TET and (4) 25 mg/l TMT using as eluent a 70:30 (v/v) mixture of methanol and 10 m*M* acetate buffer (pH 5.9) containing 2 m*M* BTMA. Sample volume, 200 μl; flow-rate, 1.0 ml/min. 5 = System peak.

3.2. Capillary electrophoresis

Most reports of the CE of inorganic cations are based on the use of indirect UV detection in

Table 1
Detection limits obtained for the separation of triorganotins for a signal-to-noise ratio of 3 without preconcentration

Analyte	Detection limit (mg/l)	
	HPLC	CE
TMT	2.5	0.16
TET	1.5	0.24
TBT	0.5	0.29
TPT	0.15	0.009

The sample volume used in HPLC was 200 μl and the effective sample volume in CE was 122 nl.

Table 2
Detection limits for TBT and TPT reported for HPLC with various detection methods without preconcentration of analytes

Detection method	Reported limit of detection (mg/l)		Ref.
	TBT	TPT	
Indirect photometry	2.1[a]	0.05[a]	[24]
Fluorimetry	0.09[a]	–	[23]
	0.015	–	[28]
	0.047	0.075	[14]
Flame atomic fluorescence	0.035	–	[32]
Flame AAS	0.1	–	[22]
Graphite furnace AAS	0.08	0.025	[20]
	0.04	–	[27]
Hydride generation AAS	0.02	–	[25]
ICP	6×10^{-5}	–	[27]
ICP-MS	$7.5 \cdot 10^{-6}$	$1.1 \cdot 10^{-5}$	[15]
	0.002	–	[28]
	0.025	–	[30]
	$2 \cdot 10^{-4}$	–	[31]

[a] For procedure with preconcentration step.

the presence of an organic amine as the primary electrolyte component [34–36]. Recently, copper electrolyte was also reported as an inorganic ionophore [34]. The CE determination of organolead and organoselenium compounds was recently performed with on-column direct UV detection [33], but because most triorganotins do not absorb in the UV region, also in CE measurements indirect UV detection with BTMA in the electrolyte was employed.

The electrophoretic separation of cations depends on the equivalent ionic conductivities and the velocity of electroosmotic flow. The latter depends on the charge of the capillary walls, whereas the relative mobilities of ions in solution can be modified by changing the ionic strength and pH by addition of complexing ligands.

Fig. 3 shows examples of electropherograms recorded for the mixture of four triorganotins with different electrolytes. In all cases examined, in a much shorter time than in HPLC a very satisfactory separation of TMT, TET and TPT is observed. However, also with CE it is more difficult to resolve TBT and TPT.

For the electrolyte containing sulfuric acid, no essential effect on the separation of organotins was observed on varying acid concentration from 0.1 to 10 m*M* and the methanol content from 10 to 50%. Under these conditions the separation of TBT and TPT was not achieved, similarly to the electrolyte containing 1–10 m*M* camphorsulfonic acid (Fig. 3A and D).

An efficient method for the modification of the apparent mobility of cations is addition of complexing agents to the electrolyte. A further advantage observed in the presence of complexing ligands is the improvement of the peak symmetry, resulting from the decrease in the mobility of complexed ions, which more closely matches the mobility of the electrolyte [36]. As modifiers for CE separation of cations, α-hydroxyisobutyric acid [34–36], citrate [36] and 18-crown-6 [37] have been used. In this study, for the CE separation of triorganotins satisfactory results were obtained with electrolytes containing tartaric acid. As the initial attempt gave separation of signals for TBT and TPT, a more detailed optimization of CE separation with tartaric acid was carried out.

A decrease in the apparent mobility of complexed cations can be expected with increase in the ligand concentration in the electrolyte. This has been found already, e.g., for 18-crown-6 used for the separation of ammonium, alkali metal and alkaline earth metal ions [37]. Similar results were obtained for the separation of tri-

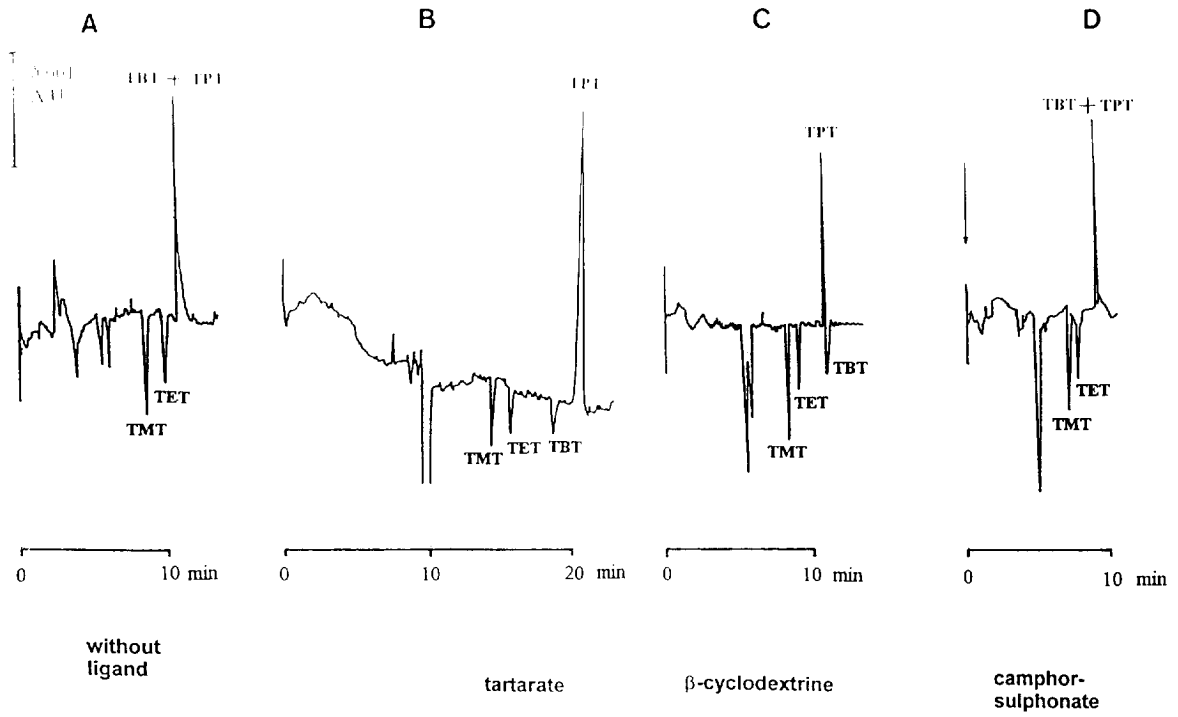

Fig. 3. Electropherograms of a mixture of 10 mg/l each of TMT, TET and TBT and 1 mg/l TPT at 20 kV with UV detection at 220 nm. Electrolytes: (A) 0.5 m*M* sulfuric acid–10% methanol–1 m*M* BTMA; (B) 20 m*M* tartaric acid–20% methanol–4 m*M* BTAC; (C) 20 m*M* β-cyclodextrin–10% methanol–1 m*M* BTMA; (D) 1 m*M* camphorsulfonic acid–1 m*M* BTAC. Effective sample volume, 4 nl; total capillary length, 60 cm.

organotins in the presence of an increasing concentration of tartaric acid in the electrolyte (Fig. 4A). From a 30 m*M* concentration of tartaric acid in the electrolyte, separation of the negative TBT peak from the positive peak corresponding to TBT is observed. The use of a longer capillary (total length 100 cm, 60 cm to the detector) allows a satisfactory separation of all triorganotins at 20 m*M* tartaric acid, and this concentration was adopted in further studies. A further increase in tartaric acid concentration improves the separation but simultaneously increases the migration times.

An increase in tartaric acid concentration also affects the magnitude of the signal (Fig. 4B). Generally, the sensitivity of indirect detection of trialkyltins is much lower than that of direct UV detection of TPT. The increase in the migration time of TPT with increase in tartaric acid concentration results in an improvement in sensitivity for TPT detection. Above 20 m*M* tartaric acid concentration, where a satisfactory separation of TBT and TPT is obtained, an increase in tartaric acid concentration also improves the sensitivity of detection of trialkyltins. More than a twofold increase in the sensitivity of detection of TMT, TET and TBT can be also achieved by increasing the BTMA concentration from 1 to 4 m*M* in the electrolyte. This effect is negligible for TPT. The increase in BTMA concentration in the electrolyte is also accompanied by a slight decrease in the migration times of all separated species.

The wavelength of detection also significantly affects the detection sensitivity; however, at wavelengths shorter than 210 nm a substantial increase in the baseline noise was observed, hence 210 nm was taken as the optimum. The effect of the pH of the electrolyte on sensitivity of detection and migration time is illustrated in Fig. 5. A decrease in the pH of the electrolyte from 3.0 to 2.6 improves the signal magnitude and decreases the migration times, but below pH

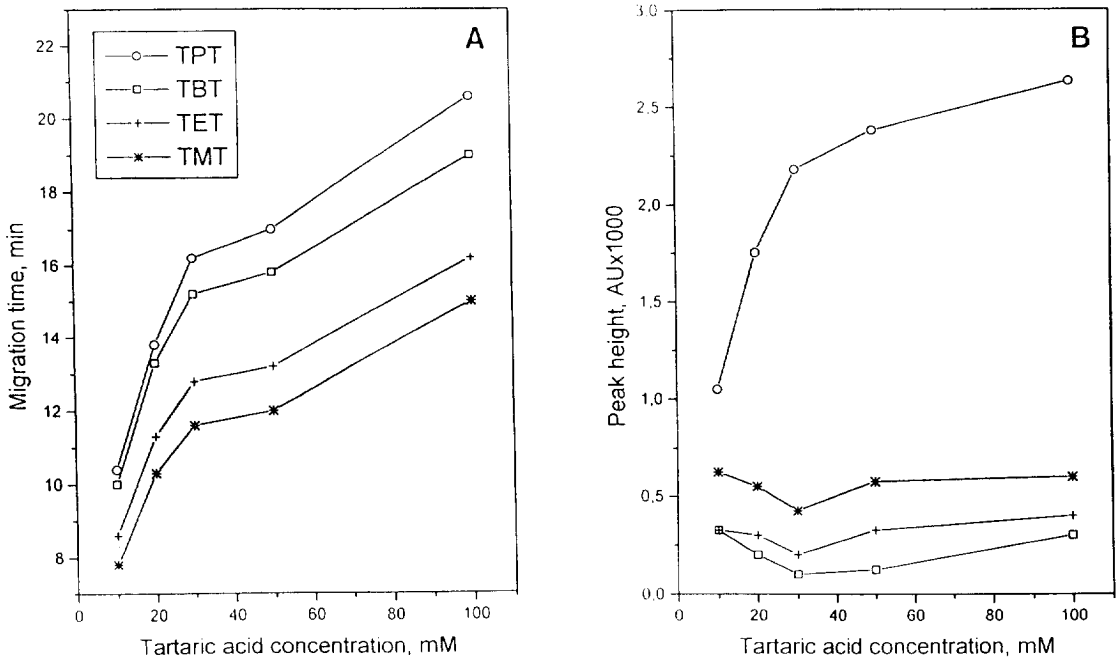

Fig. 4. Effect of the concentration of tartaric acid in the electrolyte on (A) migration times of triorganotins and (B) signal magnitude in CE separation. Mixture analyzed as in Fig. 3. Electrolyte used contained 20% methanol and 1 m*M* BTMA. Voltage, 20 kV; total capillary length, 60 cm.

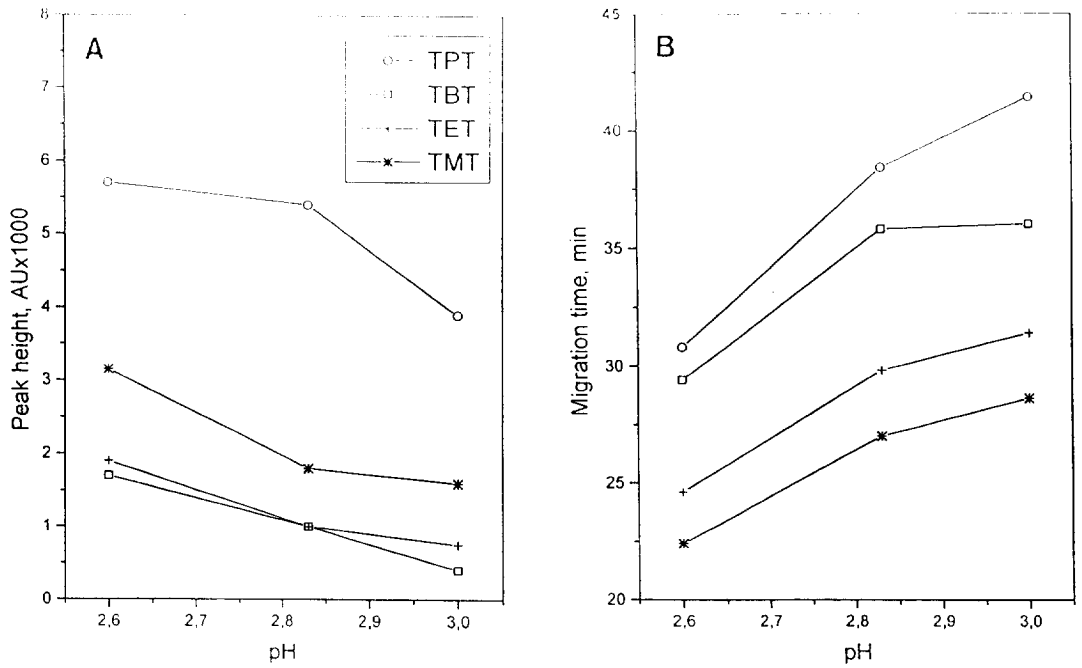

Fig. 5. Effect of pH on (A) peak heights and (B) migration times in electrophoretic determination of organotin compounds with a total capillary length of 100 cm, an effective sample volume of 61 nl and UV detection at 210 nm. Other conditions as in Fig. 3B.

2.6 a substantial increase in the baseline noise was observed, hence pH 2.6 was assumed to be the optimum value.

The apparent migration velocity is inversely proportional to the voltage across the capillary [38]. An increase in voltage from 15 to 30 kV shortens the migration times substantially, but simultaneously causes an increase in the baseline noise, therefore an optimum voltage of 20 kV was adopted.

Under the experimental conditions which were used for obtaining the electropherogram shown in Fig. 6 and for an effective sample volume of 122 nl, the detection limits were calculated for a signal-to-noise ratio of 3 for all separated species and compared with those obtained in HPLC determination (Table 1). The limit of detection for CE determination is better for all the species examined.

Another ligand employed in this study as modifier of the mobility of triorganotin cations in CE determination was β-cyclodextrin (β-CD). Cyclodextrins are now routinely used in HPLC for improving the separation of structurally different compounds including enantiomers via inclusion complexation. Recent applications of β-CD in CE include the improvement of the separation of cationic enantiomers [39] and derivatized amino acids [40]. β-CD has also been applied in the separation of organolead and organoselenium compounds by micellar electrokinetic chromatography to improve the peak shape [33]. In this study of the CE determination of organotins, it was found that the presence of β-CD in the electrolyte also improves the peak shape (Fig. 3C) and, as it also increased the migration time of TBT, it allows a partial separation of TBT and TPT. Addition of β-CD in the concentration range 0.1–20 m*M* did not result in better separation of TBT and TPT than that shown in Fig. 3B in the presence of tartaric acid in the electrolyte. The maximum β-CD concentration used in this study was limited by its solubility.

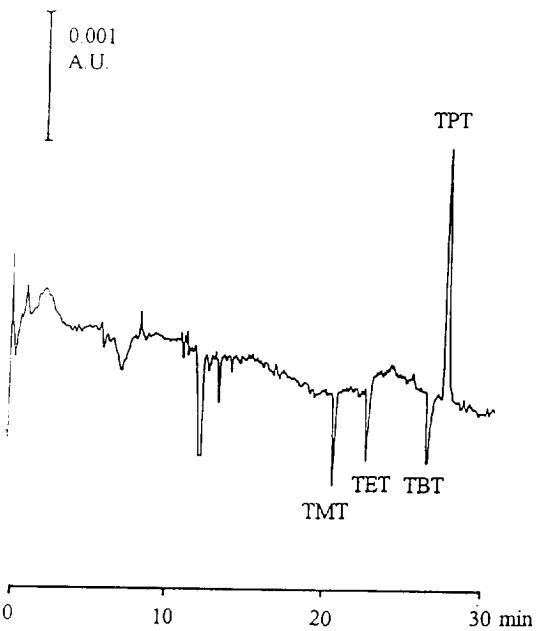

Fig. 6. Electropherogram of a mixture of 10 mg/l each of TMT, TET and TBT and 1 mg/l TPT under optimized conditions with a total capillary length of 100 cm. Electrolyte, 20 m*M* tartaric acid–20% methanol–4 m*M* BTAC (pH 2.6); effective sample volume, 12.2 nl; voltage, 20 kV; detection at 210 nm.

3.3. Preconcentration of triorganotins by solid-phase extraction

As in most other chromatographic procedures, the detectability found in the above-described HPLC and CE determinations is not sufficient for the direct determination of triorganotins in environmental samples, and therefore preconcentration procedures for these analytes are required for real environmental applications. The analytical literature provides a wide variety of preconcentration procedures for organotins by solvent extraction (see Ref. [41] and references cited therein), although in recent years these methods have been increasingly replaced by preconcentration on solid sorbents. For organotins the most often used sorbent for solid-phase extraction is a bonded silica with octadecyl functional groups, either as disposable cartridges [42,43] or extraction discs [44,45]. A satisfactory preconcentration of TBT and TPT was obtained on graphitized carbon black, where elution of analytes with different solvent allows the graphite furnance AAS determination of both

Table 3
Efficiency of preconcentration of TBT and TPT on different non-polar sorbents

Sorbent	Recovery of organotin species (%)	
	TBT	TPT
C_8	19	97
C_{18}	33	100
Phenyl	25	103
XAD-2	100	100
XAD-4	37	50

Preconcentration from 1 l of solution of 30 μg/l TBT and 3 μg/l TPT at a flow-rate of 7 ml/min. Elution with 10 ml of methanol. The values obtained are means of three parallel determinations, of which the bias exceeded ±3% in all cases.

species without a column chromatographic step [46]. A comparison of three sorbents, Carbopack, C_8 and C_{18}, showed the best recoveries of mono-, di- and tributyltin with Carbopack and C_{18} and elution with methanol [47]. Other organotins were not examined in that study. Methanol was also considered to be the best eluent for the preconcentration of TPT on C_{18} sorbent by other workers [43].

The solid-phase extraction study in this work was carried out for the two most important environmentally organotin species, TBT and TPT, with the use of five commercially available non-polar sorbents. The results obtained (Table 3) show a very different behaviour of the sorbents examined. The data in Table 3 are means of three parallel experiments. The earlier data [43] showing a satisfactory recovery of TPT on C_{18} were confirmed, but this sorbent was not appropriate for the preconcentration of TBT, as the mean recovery was 33% only. Other workers found for the preconcentration of TBT from a 1-l sample recoveries as low as 24% [47], although in other work at a much higher flow-rate satisfactory results were reported [42]. The most appropriate sorbent in this work was found to be Amberlite XAD-2, which has not, so far, been used for the preconcentration of organotin species. However, the recovery of TBT substantially decreases with sample flow-rates during preconcentration below 2 ml/min. The recovery of TBT on XAD-2 is not influenced by flow-rate in the range 2–12 ml/min.

The recovery in the preconcentration of TBT and TPT on XAD-2 was also studied in the presence of common inorganic salts occurring in various natural waters except sea water. The presence of 100 m*M* NaCl, 1 m*M* $CaCl_2$ and Na_2SO_4 and 0.8 m*M* $Cu(NO_3)_2$ did not affect the preconcentration of TPT, whereas the recovery of TBT was 71–79%.

The results obtained demonstrate that the use of solid-phase extraction with XAD-2 improves the detectability of both chromatographic and CE determinations by about three orders of magnitude, which offers the possibility of practical application in the environmental analysis of natural waters, except sea water with a high salt content.

Acknowledgement

This study was supported by the Committee of Scientific Research (Grant No. 2 0554 91 01).

References

[1] K. Robards, P. Starr and E. Patsalides, Analyst, 116 (1991) 1247.
[2] K. Robards and P.J. Worsfold, Trends Anal. Chem., 4 (1991) 121.
[3] Y.K. Chau and P.T.S. Wong, Fresenius' J. Anal. Chem., 339 (1991) 640.
[4] J.P. Craig (Editors), Organometallic Compounds in the Environment, Longman, London, 1986.
[5] C.J. Evans and S. Karpel, Organotin Compounds in Modern Technology, Elsevier, Amsterdam, 1985.
[6] T. Tsuda, M. Wada, S. Aoki and Y. Matsui, Toxicol. Environ. Chem., 18 (1988) 11.
[7] R.S. Braman and M.A. Tomkins, Anal. Chem., 51 (1979) 12.
[8] V.F. Hodge, S.L. Seidel and E.D. Goldberg, Anal. Chem., 51 (1979) 1256.
[9] L. Schebek, M.O. Andrea and H.J. Tobschall, Environ. Sci. Technol., 25 (1991) 871.
[10] M. Ochsenkuhn-Petropoulou, G. Poulea and G. Parissakis, Mikrochim. Acta, 109 (1992) 93.
[11] J.A. Stab, W.P. Cofino, B. van Hattum and U.A.Th. Brinkman, Fresenius' J. Anal. Chem., 347 (1993) 247.

[12] F.E. Brinckman, W.R. Blair, K.L. Jewett and W.P. Iverson, J. Chromatogr. Sci., 15 (1977) 493.
[13] W.G. Lakata, E.P. Lankmayr and K. Muller, Fresenius' Z. Anal. Chem., 319 (1984) 563.
[14] W. Kleibohmer and K. Cammann, Fresenius' Z. Anal. Chem., 335 (1989) 780.
[15] U.T. Kumar, J.G. Dorsey, J.A. Caruso and E. Evans, J. Chromatogr. A, 654 (1993) 261.
[16] T.H. Yu and Y. Arakawa, J. Chromatogr., 258 (1983) 189.
[17] W. Langseth, Talanta, 11 (1984) 975.
[18] M. Astruc, A. Astruc and R. Pinel, Mikrochim. Acta, 109 (1992) 83.
[19] M. Astruc, A. Astruc, R. Pinel and M. Potin-Gautier, Appl. Organomet. Chem., 6 (1992) 39.
[20] K.L. Jewett and F.E. Brinckman, J. Chromatogr. Sci., 19 (1981) 583.
[21] W.A. MacCrehan, Anal. Chem., 53 (1981) 74.
[22] L. Ebdon, S.J. Hill and P. Jones, Analyst, 110 (1985) 515.
[23] L. Ebdon and J.I. Garcia-Alonso, Analyst, 112 (1987) 1551.
[24] C.W. Whang and L.L. Yang, Analyst, 113 (1988) 1393.
[25] L. Ebdon, S.J. Hill and P. Jones, Talanta, 38 (1991) 607.
[26] A. Mazzucotelli, R. Frache, E. Magi, P. Rivaro and T. Gerbino, Ann. Chim. (Rome), 82 (1992) 379.
[27] F. Pannier, X. Dauchy, M. Potin-Gautier, A. Astruc and M. Astruc, Appl. Organomet. Chem., 7 (1993) 213.
[28] J.I. Garcia-Alonso, A. Sanz-Medel and L. Ebdon, Anal. Chim. Acta, 283 (1993) 261.
[29] A.P. Walton, G.T. Wei, Z. Liang, R.G. Michel and J.B. Morris, Anal. Chem., 63 (1991) 232.
[30] S. Branch, L. Ebdon, S. Hill and P. O'Neill, Anal. Proc., 26 (1989) 401.
[31] J.W. McLaren, K.W.M. Siu, J.W. Lam, S.N. Willie, P.S. Maxwell, A. Palepu, M. Koether and S.S. Berman, Fresenius' J. Anal. Chem., 337 (1990) 721.
[32] J.W. Oudsema and C.F. Poole, Fresenius' J. Anal. Chem., 344 (1992) 426.
[33] C.L. Ng, H.K. Lee and S.F.Y. Li, J. Chromatogr. A, 652 (1993) 547.
[34] F. Foret, S. Fanali, A. Nardi and P. Bocek, Electrophoresis, 11 (1990) 780.
[35] M. Chen and R.M. Cassidy, J. Chromatogr., 602 (1992) 227.
[36] A. Weston, P. Brown, P. Jandik, W.R. Jones and A.L. Heckenberg, J. Chromatogr., 593 (1992) 289.
[37] J.M. Riviello and M.P. Harrold, J. Chromatogr. A, 652 (1993) 385.
[38] P.D. Grossman, J.C. Colburn and H.H. Lauer, Anal. Biochem., 179 (1989) 28.
[39] C. Quang and M.G. Khaledi, Anal. Chem., 65 (1993) 3354.
[40] T. Ueda, R. Mitchell, F. Kitamura, T. Metcalf, T. Kuwana and A. Nakamoto, J. Chromatogr., 593 (1992) 265.
[41] W.M.R. Drikx, R. Łobiński and F.C. Adams, Anal. Chim. Acta, 286 (1994) 309.
[42] G.A. Junk and J.J. Richard, Chemosphere, 1 (1987) 61.
[43] R. Compano, M. Granados, C. Leal and M.D. Prat, Anal. Chim. Acta, 283 (1993) 272.
[44] O. Evans, B.J. Jacobs and A.L. Cohen, Analyst, 116 (1991) 15.
[45] J. Szpunar-Łobińska, M. Ceulemans, R. Łobiński and F.C. Adams, Anal. Chim. Acta, 278 (1993) 99.
[46] T. Ferri, E. Cardarelli and B.M. Petronio, Talanta, 36 (1989) 513.
[47] S. Chiavarini, C. Cremisini, T. Ferri, R. Morabito and C. Ubaldhi, Appl. Organomet. Chem., 6 (1992) 147.