ELSEVIER

Journal of Chromatography A, 840 (1999) 21–30

JOURNAL OF CHROMATOGRAPHY A

Determination of chlorophenolics in waters by membrane solid-phase extraction: comparison between C_{18} and activated carbon membranes and between modes of extraction and elution

Luis E. Sojo*, Joyce Djauhari

ASL Laboratories Ltd., 1988 Triumph Street, Vancouver, BC V5L-1K5, Canada

Received 5 May 1998; received in revised form 2 February 1999; accepted 3 February 1999

Abstract

Chlorophenols, chlorocatechols and chloroguaiacols were spiked at the 1 ppb level into water samples containing up to 50 ppm dissolved organic carbon, acetylated in situ and extracted onto C_{18} and activated carbon (AC) membranes by dynamic or static sorption. Recovery of the analytes from the membranes was carried out by either static (in-vial elution) or dynamic desorption (elution) using either acetone or toluene. Extracts were analysed by gas chromatography–mass spectrometric detection (GC–MSD). C_{18} membranes gave recoveries of between 70 and 102% for all compounds with the exception of phenol, when using dynamic desorption with acetone as the eluting solvent. Static desorption using C_{18} membranes gave satisfactory recoveries (65–80%) for chlorophenols. AC membranes gave quantitative recoveries for all compounds when using dynamic desorption with toluene. Static desorption using AC membranes resulted in low recoveries (<60%) for all compounds except phenol. A combination of a C_{18} membrane placed on top of the AC membrane during dynamic extraction was tested to increase retention of phenol. Dynamic desorption of both membranes in the reverse direction (AC on top of C_{18}) using 10 ml of acetone or toluene gave quantitative results for all compounds, even in the presence of 50 ppm dissolved organic carbon.

Keywords: Extraction methods; Chlorophenols; Chlorocatechols; Chloroguaiacols

1. Introduction

A large number of procedures for determining chlorophenolics in water using solid-phase extraction (SPE) have been tested [1–11]. Various types of solid phases in the cartridge format have been used, including C_{18} [1,6,7,9], polystyrene–divinylbenezene-based polymers [5,6,9] and various forms of carbon [2–4,8,10,11]. Some of these applications were based on off-line SPE followed by either liquid chromatography (LC) [1–4] or gas chromatography (GC) [8,10,11]; while others involved on-line SPE–LC [5–7] or on-line SPE–GC [9]. The analytes have been limited to chloro-substituted and nitro-substituted phenols [1–11]. To our knowledge there are no reports of using off-line or on-line SPE to determine analytes such as chlorocatechols and chloroguaiacols. These compounds are present in pulp mill effluents [12] and can be detected in receiving waters [13].

The total number of chlorophenolic compounds, including chlorocatechols and chloroguaiacols, is

*Corresponding author. Tel.: +1-604-253-4188; fax: +1-604-253-6700.

E-mail address: luis@als-labs.bc.ca (L.E. Sojo)

0021-9673/99/$ – see front matter
PII: S0021-9673(99)00186-7

over forty species, with a wide range in octanol–water partition coefficients ($1.5<\log K_{ow}<6$). This wide range in hydrophobicity poses a challenge to SPE-based procedures, which usually employ hydrophobic phases [1,6,7,9] or a combination of anion exchange and hydrophobic phases [2]. Traditionally, chlorophenolic compounds have been determined in water by capillary GC with electron capture detection (ECD) [14] or mass spectrometric detection (MSD) after derivatization and liquid–liquid extraction (LLE) [15]. GC is needed in order to resolve the large number of compounds, which are not easily resolved using LC. Although LLE effectively recovers these compounds from waters, it is time-consuming and requires large volumes of toxic solvents. These factors provide a strong incentive for replacing LLE for some form of SPE.

SPE materials such as C_{18} and activated carbon (AC) immobilized in Teflon™ membranes, presents an alternative to LLE and standard SPE cartridges. These formats permit reduction in the amount of toxic solvent used and, at the same time, they allow for the concentration of compounds with a wide range of polarities. Various forms of activated and graphitic carbons have been proven to be effective in retaining chlorophenols and other polar compounds [2–4,8,11]. SPE membranes, in general, have some clear advantages over traditional SPE cartridges, including faster sorption/desorption kinetics, elimination of channelling and the use of less solvent during the desorption or elution step [16]. AC membranes, in particular, have been used recently for extracting polar analytes from water samples using standard elution approaches [17,18]. Because most SPE membranes are pliable, they can be physically transferred into a vial containing a receiving solvent to elute previously sorbed analytes by static desorption (in-vial elution) [19–22]. This approach is quite attractive from an automation point of view. All of these applications involved the use of C_{18} membranes. To our knowledge, there are no reports of using the in-vial elution approach with AC membranes.

In this paper, we study the feasibility of using AC and C_{18} membranes for the extraction of over forty chlorophenolic species, including chlorophenols, chlorocatechols and chloroguaiacols, from water samples containing humic substances as surrogates for dissolved organic carbon (DOC). We focussed on the various extraction and elution procedures, including dynamic and static sorption (extraction) and dynamic and static desorption (elution).

2. Experimental

2.1. Materials and equipment

Chlorophenolic compounds (98% purity) were purchased from Helix Biotech., Richmond, BC, Canada, and were used as received. C_{18} and AC membranes of 47 and 22 mm diameters were a gift from 3M Company, St. Paul, MN, USA. Toluene, hexane and acetone solvents were of pesticide grade and were purchased from BDH, Vancouver, Canada. Potassium carbonate reagent was purchased from BDH. Laurentian humic acid, which was prepared at the Chemistry and Biochemistry Department of Concordia University, Montreal, Canada, was used as surrogate for dissolved organic carbon. A Millipore 47 mm vacuum filtration unit was used for extraction of water samples using either C_{18} or AC membranes. MilliQ (MQ) water was used throughout.

Gas chromatographic analysis was carried out on a HP 5890 Series II GC, equipped with a HP 7673 liquid auto-sampler, a DB-5 capillary GC column (30 m×0.25 mm I.D. and 2.5 μm film thickness) from Supelco, Oakville, Ontario, Canada. The injection port temperature was set at 270°C and operated in the splitless mode. The oven temperature was programmed as follows: initial temperature of 50°C was increased to 75°C at 20°C/min and held at 75°C for 1 min, followed by an increase to 145°C at 5°C/min and held at 145°C for 4 min, and a further increase to a final temperature of 280°C at 5°C/min. The final temperature was held for 1 min. The carrier gas was He at a total flow of 45.0 ml/min. Detection was carried out using a HP 5890 mass selective detector in the time scheduled mass selective mode using three ions per analyte.

2.2. In-situ acetylation of water samples

A 400-ml volume of Milli-Q water was spiked with a cocktail of chlorophenolic compounds, re-

sulting in a nominal concentration of 1 ppb for each compound. Water samples were acetylated in-situ by the addition of an excess (10 ml) of acetic anhydride and stirring for 15 min after buffering to pH 8 with potassium carbonate.

Calibration solutions were prepared as follows: a 50-μl aliquot of a 10 ppm stock solution of chlorophenolic compounds was spiked into 1 ml of hexane, followed by the addition of 0.5 ml of trimethylamine and 1 ml of acetic anhydride. This solution was incubated for 15 min in a sand bath at 55°C and neutralized with phosphoric acid buffer. The resulting solution containing the acetylated derivatives was back-extracted into hexane, evaporated to near dryness under a gentle nitrogen stream, diluted to 5 ml with hexane, resulting in a 0.1-ppm calibration solution.

2.3. Static desorption of analytes from membranes

Prior to the desorption experiments, 400 ml of MQ water spiked with the acetylated analytes were passed through each membrane using the Millipore vacuum filtration apparatus at about 50 ml/min. After extraction, the membranes were dried by drawing air through them for 20 min. The membranes were removed from the filtration apparatus and folded into a 7-ml glass vial using a pair of clean stainless steel pliers. A 3-ml volume of solvent was added to the vials, making sure that the membranes were fully immersed in the solvent. The vials were then capped with Teflon™-lined crimp-caps. The solvents tested included hexane, acetone, toluene and hexane containing trimethylammonium hydroxide at 1% (v/v). Samples (100 μl) were withdrawn from each vial at 0, 60 and 180 min and these were analysed by GC–MS.

2.4. Dynamic extraction procedure

A 100-ml volume of acetylated spiked water (1 ppb) was passed through either a C_{18}, AC or a combination of a C_{18} on top of an AC membrane, as described in Section 2.3. All membranes had been rinsed previously with 10 ml of acetone. Precautions were taken to avoid drying of the membranes before sample extraction took place. After extraction, the membranes were rinsed with 10 ml of MQ water and dried, as described in Section 2.3.

2.5. Static extraction procedure

A 400-ml volume of acetylated spiked water (1 ppb) was equilibrated overnight with a single C_{18} or AC membrane overnight by immersing the prewetted membranes in the sample flask. The samples were magnetically stirred. After equilibration, the membranes were withdrawn from the samples using pliers and dried as described in Section 2.3.

2.6. Dynamic desorption procedure

C_{18} membranes were eluted with 10 ml of acetone by flushing them with the solvent using the Millipore vacuum filtration unit. The combination of C_{18} and AC membranes was eluted in the back-flush mode with the AC membrane on top of the C_{18} using toluene. AC membranes were similarly eluted with 10 ml of toluene. The eluents were collected in test tubes, spiked with internal standards and transferred into a 35°C water bath where they were evaporated down to 1 ml under a gentle stream of nitrogen before GC–MS analysis.

2.7. Static desorption procedure

After sample extraction, the membranes were dried and folded into 7 ml vials followed by the addition of 3 ml of solvent, as described in Section 2.3. After a 3-h desorption period, a 1-ml aliquot of the solvent was withdrawn from each sample and transferred to a GC vial, spiked with internal standards and analysed by GC–MS.

Two types of experiments were carried out using the static desorption procedure including static extraction–static desorption (S/S) and dynamic extraction–static desorption (D/S). Similarly, two types of experiments using dynamic extraction procedure were carried out including dynamic extraction–dynamic desorption (D/D) and dynamic extraction–static desorption (D/S).

2.8. Liquid–liquid extraction

Liquid–liquid extraction was used as a means of assessing the results obtained using the various membrane extraction procedures. Briefly, 400 ml of acetylated spiked water were extracted three times with 80 ml aliquots of hexane by shaking for 10 min each time in a separatory funnel. The combined hexane extracts were dried over sodium sulfate, blown down, spiked with internal standards and reconstituted to 1 ml with hexane before analysis by GC–MS.

2.9. Effects of DOC

A 4-ml volume of MQ water containing the analytes at 1 ppb or 0.1 ppb levels was spiked with an aqueous solution of humic acids resulting in a final concentration of 50 ppm in DOC. The samples were extracted using the D/D procedure with the C_{18} on top of the AC membrane and were eluted as described in Section 2.6.

3. Results and discussion

3.1. Static desorption

Quantitative desorption (>75%) for all compounds from C_{18} membranes took place in less than 1 h when using hexane, acetone or toluene. McDonnell et al. [19] found that it took approx. 30 min for the quantitative desorption of chlorinated pesticides from C_{18} membranes into ethyl acetate. Krueger and Fields [21] found that it required more that 1 h for linear alkylbenzene sulfonates to be desorbed from the same membranes. Static desorption from AC membranes, on the other hand, gave lower recoveries for most of the test compounds, even when using toluene as the receiving solvent. Fig. 1 shows the effect of solvent type on the recovery of chlorophenols after a desorption time of 3 h. Tests conducted after 24 h resulted in similar recovery values. The recoveries from AC membranes were rather solvent-dependent, in contrast to those from C_{18} membranes. This is probably due to a combination of strong sorption onto the activated carbon surface of the membranes and slow desorption kinetics. The use of modified receiving solvents, such as hexane containing tetramethylammonium hydroxide [8], did not improve the recoveries of the analytes. Based on our results for chlorophenols, acetone and toluene were the solvents of choice for static desorption when using C_{18} and AC membranes, respectively.

3.2. Sorption and desorption of chlorophenolic compounds from AC membranes

The effect of the sorption mode on analyte recoveries can be read from Table 1. Comparison of the recoveries using the S/S and D/S procedures using toluene as a receiving solvent indicates that there is little or no difference between extraction modes. The recoveries were significantly lower (<60%) than when using LLE. As was shown above, these low recoveries are due to inefficient desorption of the analytes from the membranes.

The effect of the desorption mode can be seen by comparing the results for experiments using the D/D and D/S procedures (Table 1). There is a significant improvement in the recoveries when the dynamic desorption mode is used. In the static desorption mode, the recovery of the analytes depends on their distribution between a single solvent volume and solid phase, limiting their extraction efficiency. On the other hand, in the dynamic desorption mode, the solid phase is continuously exposed to a fresh aliquot of receiving solvent, resulting in an increase in analyte recoveries.

3.3. Sorption and desorption of chlorophenolics from C_{18} membranes

Similar to AC membranes, the sorption mode had very little effect on the recovery of analytes when using C_{18} membranes (Table 2). In general, recoveries were in between those obtained using LLE and S/S and D/S procedures with AC membranes. In general, C_{18} membranes gave higher recoveries than AC membranes when using the static desorption mode (Fig. 1). In contrast to AC membranes, the recoveries for chlorophenols ranged from 65 to 94%. Dynamic desorption also provided a significant improvement in the overall recoveries (Table 2).

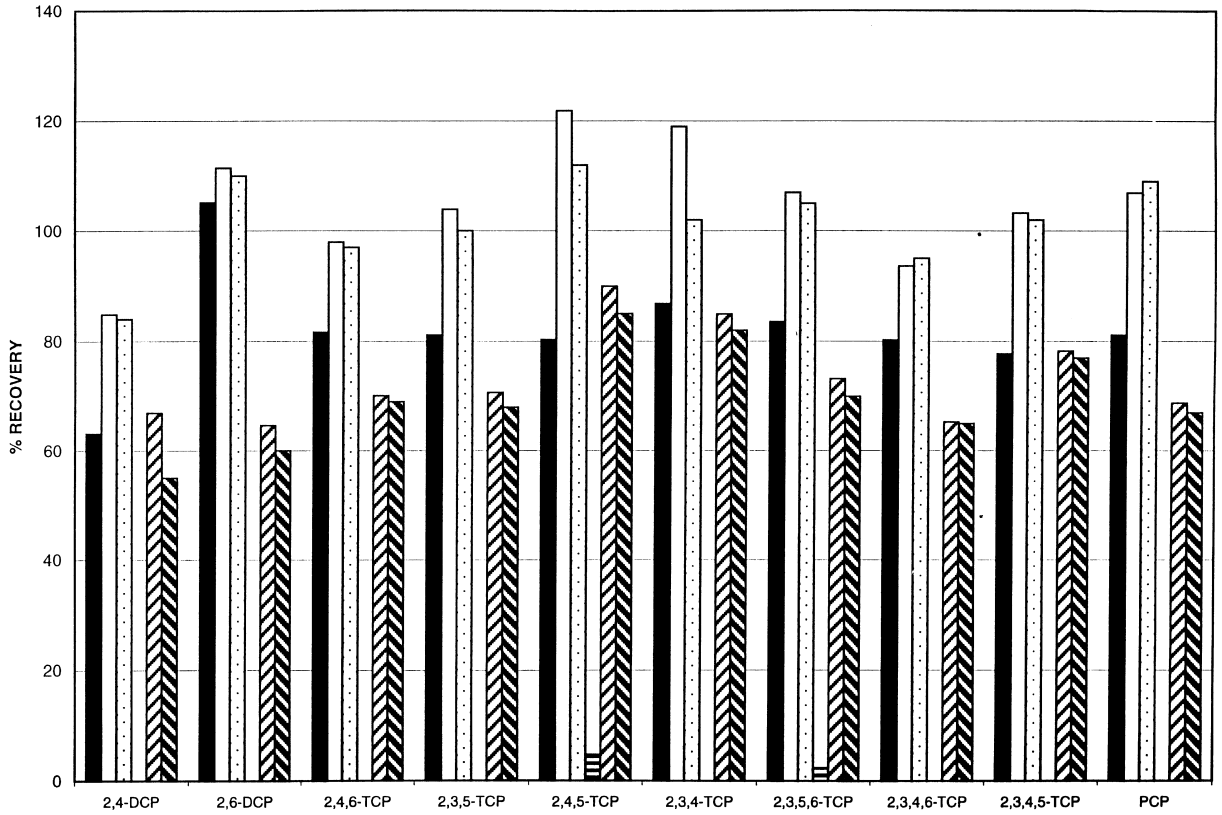

Fig. 1. Effect of receiving solvent on the recoveries of chlorophenols from C_{18} and AC membranes using the static desorption procedure. C_{18} and hexane (■), toluene (□), acetone (□). AC hexane (▨), toluene (▨) and acetone (▨).

3.4. C_{18} and AC membranes in series using dynamic extraction and desorption. Effects of dissolved organic carbon

The results so far indicate that the best recoveries were obtained using the dynamic desorption mode. C_{18} gave significantly higher recoveries than AC for most analytes, with the exception of phenol. This compound was better recovered with AC membranes. C_{18} membranes used in the dynamic desorption mode also gave better precision (Tables 1 and 2).

Because of the importance of phenol as a contaminant in waters [15], attempts were made to improve its recovery using C_{18} and AC membranes in series, as described in Section 2.4. Dynamic desorption of the analytes from the two-membrane layer was carried out with the AC membranes on top of the C_{18}. In this manner, analytes trapped on the C_{18} membrane did not interact with the AC membrane during elution, minimizing their potential loss due to retention on the AC surface. Table 3 summarizes the data obtained using this approach for spiked MQ waters and waters containing DOC. The recoveries for phenol were significantly improved using this approach. It can be seen that, in the case of spiked water samples containing no DOC, recoveries for all other compounds were quite similar to those obtained with a single C_{18} membrane. Similar results were obtained in the presence of DOC.

The presence of DOC had little effect on the recoveries of the analytes, regardless of the type of membrane used. This is in contrast to some reports that have shown that the presence of DOC can

Table 1
Recoveries of chlorophenolics from spiked water samples (1 ppb) using activated carbon (AC) membranes[a]

	D/D–AC	S/S–AC	D/S–AC	LLE
Phenol	115	124	112	124
4-Chlorophenol	71	58	47	88
2-Chlorophenol	74	61	56	89
3-Chlorophenol	71	59	51	87
2,3-Dichlorophenol	66	51	44	87
2,4- and 2,5-Dichlorophenol	64	52	45	90
2,6-Dichlorophenol	67	55	50	85
3,4-Dichlorophenol	64	47	40	97
3,5-Dichlorophenol	61	45	38	86
2,3,4-Trichlorophenol	62	38	33	90
2,3,5-Trichlorophenol	57	39	36	113
2,3,6-Trichlorophenol	64	45	43	83
2,4,5-Trichlorophenol	56	40	35	96
2,4,6-Tribromophenol	62	40	40	85
2,4,6-Trichlorophenol	60	46	43	91
3,4,5-Trichlorophenol	64	31	27	85
2,3,4,5-Tetrachlorophenol	59	26	25	101
2,3,4,6-Tetrachlorophenol	59	36	35	84
2,3,5,6-Tetrachlorophenol	68	38	39	94
Pentachlorophenol	58	28	28	90
3-Chlorocatechol	102	54	56	89
4-Chlorocatechol	105	40	50	90
3,4-Dichlorocatechol	91	42	43	89
3,5-Dichlorocatechol	75	46	48	148
3,6-Dichlorocatechol	96	40	42	146
4,5-Dichlorocatechol	80	37	36	134
3,4-Trichlorocatechol	67	27	33	118
3,4-Trichlorocatechol	76	37	42	128
Tetrachlorocatechol	60	22	30	96
4-Chloroguaiacol	74	52	48	104
5-Chloroguaiacol	73	52	44	110
6-Chloroguaiacol	77	54	55	111
3,4-Dichloroguaiacol	68	48	43	110
4,5-Dichloroguaiacol	68	40	38	106
4,6-Dichloroguaiacol	69	45	42	114
3,4,5-Trichloroguaiacol	71	37	35	108
3,4,6-Trichloroguaiacol	70	45	43	108
4,5,6-Trichloroguaiacol	74	35	34	103
Tetrachloroguaiacol	66	46	42	119

[a] Coefficient of variation ($n=3$) ranges; 11–33% for D/D–AC, 1–28% for S/S–AC, 1–18% for D/S–AC and 3–28% for LLE.

reduce the recovery of some organic compounds when using C_{18} cartridges [23]. It is quite possible that the combination of sample treatment (acetylation) prior to extraction and the fact that the membranes have a higher packing density and faster sorption/desorption kinetics than traditional cartridges [16,24] reduced the likelihood of analyte breakthrough. Recoveries of the analytes at lower spiked levels (0.1 ppb) and in the presence of 50 ppm DOC (not shown) were quite similar to those at the 1 ppb level.

Fig. 2 shows the chromatograms for two spiked water samples containing 50 ppm dissolved organic carbon extracted with C_{18} and AC membranes using

Table 2
Recoveries of chlorophenolics from spiked water samples (1 ppb) using C_{18} membranes[a]

	D/D–C_{18}	S/S–C_{18}	D/S–C_{18}
Phenol	29	40	36
4-Chlorophenol	67	50	64
2-Chlorophenol	67	53	65
3-Chlorophenol	67	52	65
2,3-Dichlorophenol	77	65	63
2,4- and 2,5-Dichlorophenol	75	76	76
2,6-Dichlorophenol	75	80	77
3,4-Dichlorophenol	77	69	69
3,5-Dichlorophenol	71	79	75
2,3,4-Trichlorophenol	76	70	64
2,3,5-Trichlorophenol	70	83	76
2,3,6-Trichlorophenol	72	94	84
2,4,5-Trichlorophenol	68	83	76
2,4,6-Tribromophenol	82	91	83
2,4,6-Trichlorophenol	71	93	82
3,4,5-Trichlorophenol	73	78	72
2,3,4,5-Tetrachlorophenol	78	70	67
2,3,4,6-Tetrachlorophenol	74	92	84
2,3,5,6-Tetrachlorophenol	72	93	84
Pentachlorophenol	76	85	81
3-Chlorocatechol	95	10	23
4-Chlorocatechol	98	10	22
3,4-Dichlorocatechol	94	15	20
3,5-Dichlorocatechol	96	26	35
3,6-Dichlorocatechol	100	23	30
4,5-Dichlorocatechol	101	16	23
3,4,5-Trichlorocatechol	82	20	25
3,4,6-Trichlorocatechol	93	32	37
Tetrachlorocatechol	61	24	31
4-Chloroguaiacol	87	32	45
5-Chloroguaiacol	86	34	42
6-Chloroguaiacol	87	33	41
3,4-Dichloroguaiacol	85	54	55
4,5-Dichloroguaiacol	89	45	47
4,6-Dichloroguaiacol	85	57	54
3,4,5-Trichloroguaiacol	87	62	60
3,4,6-Trichloroguaiacol	82	78	71
4,5,6-Trichloroguaiacol	102	55	52
Tetrachloroguaiacol	89	83	79

[a] Coefficient of variation ($n=3$) ranges: 2–12% for D/D–C_{18}, 1–13% for S/S–C_{18} and 2–14% for D/S–C_{18}

the D/D procedure. The differences in peak areas for some of the analytes is related to the recovery efficiency. Some of the peaks were higher in the lower trace (AC membrane), in particular, the first peak, which corresponds to phenol (Table 3), while other peaks have lower area counts in accordance with the data in Tables 2 and 3.

4. Conclusions

Various approaches for the extraction of chlorophenolic compounds at the 1 ppb level from in-situ acetylated water samples using C_{18} and AC solid phase membranes were tested. Dynamic extraction with C_{18} followed by either dynamic desorption

Table 3
Recoveries of chlorophenolics from spiked water samples (1 ppb) using C_{18} membranes using a two-layer membrane C_{18}/AC

	D/DC$_{18}$AC	D/DC$_{18}$AC[a]	D/DC$_{18}$	D/DC$_{18}$[a]
Phenol	189	125	29	30
4-Chlorophenol	73	81	67	73
2-Chlorophenol	72	80	67	75
3-Chlorophenol	75	82	67	74
2,3-Dichlorophenol		82	77	85
2,4- and 2,5-Dichlorophenol	72	80	75	82
2,6-Dichlorophenol	72	79	75	83
3,4-Dichlorophenol	75	83	77	83
3,5-Dichlorophenol	71	78	71	78
2,3,4-Trichlorophenol	73	80	76	84
2,3,5-Trichlorophenol	70	77	70	77
2,3,6-Trichlorophenol	71	78	72	80
2,4,5-Trichlorophenol	70	77	68	75
2,4,6-Tribromophenol	79	87	82	90
2,4,6-Trichlorophenol	70	77	71	78
3,4,5-Trichlorophenol	75	83	73	80
2,3,4,5-Tetrachlorophenol	70	77	78	85
2,3,4,6-Tetrachlorophenol	70	77	74	82
2,3,5,6-Tetrachlorophenol	71	78	72	79
Pentachlorophenol	69	76	76	84
3-Chlorocatechol	105	116	95	104
4-Chlorocatechol	105	116	98	108
3,4-Dichlorocatechol	86	95	94	104
3,5-Dichlorocatechol	80	88	96	106
3,6-Dichlorocatechol	79	87	100	110
4,5-Dichlorocatechol	86	95	101	112
3,4,5-Trichlorocatechol	76	84	82	90
3,4,6-Trichlorocatechol	75	82	93	102
Tetrachlorocatechol	63	70	61	67
4-Chloroguaiacol	82	90	87	95
5-Chloroguaiacol	76	94	86	95
6-Chloroguaiacol	93	102	87	96
3,4-Dichloroguaiacol	80	89	85	93
4,5-Dichloroguaiacol	84	95	89	98
4,6-Dichloroguaiacol	80	88	85	94
3,4,5-Trichloroguaiacol	77	85	87	96
3,4,6-Trichloroguaiacol	77	85	82	91
4,5,6-Trichloroguaiacol	83	92	102	113
Tetrachloroguaiacol	93	103	89	98

[a] Spiked water at 5-ppm DOC. Coefficient of variation ($n=3$) ranges: 2–15% for D/DC$_{18}$C, 2–16% for D/DC$_{18}$AC, 2–16% for D/DC$_{18}$ and 1–17% for D/DC$^{*}_{18}$.

proved to be the most efficient procedure for quantitative recovery of the test analytes, except phenol. AC membranes and a combination of C_{18} and AC membranes in series provided quantitative recoveries for all compounds in the presence of 50 ppm DOC. Dynamic or static extraction followed by static desorption only gave satisfactory recoveries when C_{18} membranes were used. AC membranes used in this mode gave poor recoveries.

Acknowledgements

The authors would like to thank the Science

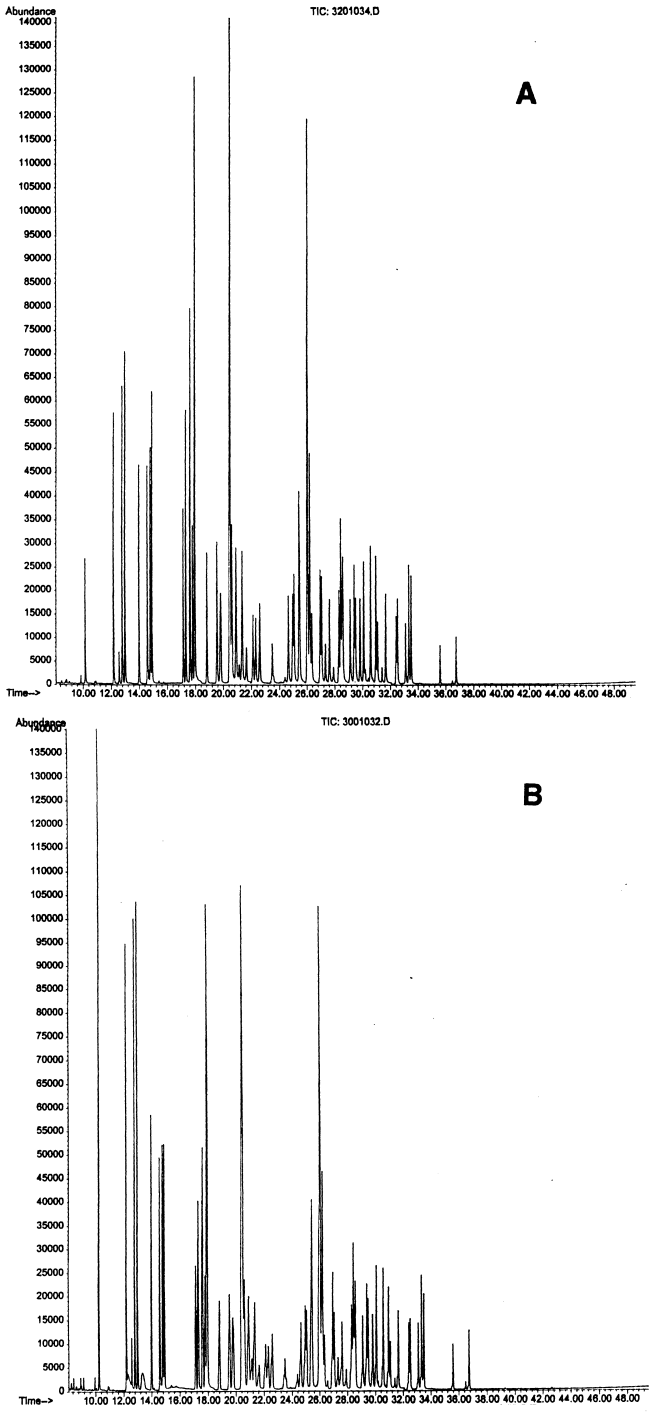

Fig. 2. Chromatograms of chlorophenolic compounds extracted from water samples containing 50 ppm dissolved organic carbon by SPE with C_{18} (A) and AC (B) using dynamic extraction (sorption) and dynamic elution (desorption). See text for details.

Council of British Columbia, Canada, for partially funding this project, Sue M. Price of 3M company for providing the C_{18} and activated carbon membranes and Jennifer Drewery of ASL for running the samples of the GC–MS and the two anonymous reviewers for their valuable comments and suggestions.

References

[1] L. Renber, K. Lindstrom, J. Chromatogr. 214 (1981) 327.
[2] A. Di Corcia, S. Marchese, R. Samperi, J. Chromatogr. 642 (1993) 175.
[3] A. Di Corcia, S. Marchese, R. Samperi, J. Chromatogr. 642 (1993) 163.
[4] A. Di Corcia, S. Marchese, R. Samperi, G. Cecchini, L. Cirili, J. Assoc. Off. Anal. Chem. Int. 77 (1993) 446.
[5] E. Brouwer, U.A.Th. Brinkman, J. Chromatogr. A 678 (1994) 521.
[6] E. Procurull, R.M. Marce, F. Borrull, Chromatographia 41 (1995) 521.
[7] M.W. Jung, D.W. Lee, K.J. Paeng, Anal. Sci. Int. 12 (1996) 981.
[8] I. Rodriguez, M.I. Turnes, M.C. Mejuto, R. Cela, J. Chromatogr. A 721 (1996) 297.
[9] A.J.H. Louter, P.A. Jones, D. Jorritsma, J.J. Vreuls, U.A.Th. Brinkman, J. High Resolut. Chromatogr. 20 (1997) 363.
[10] J. Nolte, B. Grass, F. Heinmlich, D. Klockow, Fresenius' J. Anal. Chem. 357 (1997) 763.
[11] I. Rodriguez, M.I. Turnes, M.C. Mejuto, R. Cela, J. Chromatogr. A 786 (1997) 285.
[12] M. Gregov, M. Priha, E. Talka, O. Valttila, A. Kangas, K. Kukkonen, Proc. TAPPI Environ. Confer. Charleston SC. U.S.A., April 18–20, 1988, 443.
[13] J. Paasivirta, J. Knuutinen, P. Maatela, R. Paukku, J. Soikkeli, J. Sarkka, Chemosphere 17 (1988) 137.
[14] T.M. Xie, K. Abramsson, E. Fogelqvist, B. Josefsson, Environ. Sci. Technol. 20 (1986) 457.
[15] L.E. Sojo, D. Hoover, M.C. Hamilton, B.R. Fowler, Proc. of the TAPPI Environ, Confer. Book 1 (1993) 101.
[16] P.W. Fernando, M.L. Larrive, C.F. Poole, Anal. Chem. 65 (1993) 588.
[17] B.A. Tomkins, W.H. Griest, C.E. Higgins, Anal. Chem. 68 (1996) 2533.
[18] B.A. Tomkins, W.H. Griest, D.R. Hearle, Anal. Lett. 30 (1997) 1697.
[19] T. McDonnell, J. Resenfeld, A. Rais-Firouz, J. Chromatogr. 629 (1993) 41.
[20] C.J. Krueger, J.A. Fields, Anal. Chem. 67 (1995) 3363.
[21] W.R. Reagen, A.-D. Vo, Sec. Intern. Confer., On-site Analysis Field Portable Instrumentation, The Lab comes to the Field, January 24–26, 1994, Houston (Montgomery), Texas.
[22] J.A. Fields, K. Monohan, Anal. Chem. 67 (1995) 3357.
[23] P.F. Landurm, S.R. Nihart, B.J. Eadie, W.S. Gardner, Environ. Sci. Techonol. 20 (1984) 187.
[24] H. Lingeman, S.J.F. Hoesktra-Oussoren, J. Chromatogr. A 689 (1997) 221.