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Selective determination of phenols in water by a two-trap tandem extraction system followed by liquid chromatography

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ABSTRACT

The ability of a two-trap tandem system, one containing 300 mg of graphitized carbon black (GCB) and the other filled with 50 mg of Sephadex QAE A-25 strong anion exchanger (SAX), to extract trace amounts of phenols from environmental waters and isolate them from base–neutral species was evaluated. After the water sample had passed through the GCB cartridge, the latter was connected to the SAX cartridge and base–neutral species were removed from the GCB surface by a neutral eluent. Co-eluted very weakly acidic phenols were selectively readsorbed on the SAX surface. Still maintaining the two cartridges in series, an acidified eluent was allowed to flow through the two cartridges to recover the most acidic phenols from the GCB cartridge and the least acidic phenols from the SAX cartridge. After partial removal of the solvent, the final extract was submitted to reversed-phase high-performance liquid chromatography with UV detection. Recoveries of seventeen phenols of environmental concern added to 2 l of drinking water at levels between 0.2 and 2 μg/l were higher than 90%. The effect of the presence in water of fulvic acids on the efficiency of the extraction device was assessed. In term of recovery, the two-trap tandem system was compared with other two single extraction cartridges, one containing a chemically bonded siliceous material (C_{18}) and the other SAX material. The limits of detection of the analytes considered were well below 0.1 μg/l.

INTRODUCTION

Phenols are toxic substances frequently occurring in the aquatic environment as a result of contamination from a variety of sources. Both the European Economic Community (EEC) and the US Environmental Protection Agency (EPA) include many phenol derivatives in the list of priority pollutants that should be monitored in environmental waters. This has prompted the development of various methods making use of gas chromatography [1–5] or liquid chromatography (LC) [6–10] for their separation and determination. Most of these methods, however, are not sufficiently sensitive to comply with a recent EEC Directive which sets maximum admissible individual concentrations of 0.1 μg/l for organic contaminants in drinking water. Also, many proposed analytical procedures are inadequate for monitoring traces of phenols in complex aqueous matrices, as they lack selectivity.

Liquid–liquid extraction (LLE) is usually the technique of choice for extracting phenols from water samples. In recent years, in order to eliminate some well known drawbacks of the LLE technique, liquid–solid extraction (LSE) of phenols by various reversed-phase adsorbing materials [6,10,11–13] has been included in many analytical schemes. For determining phenols in complex aqueous matrices, binding the target analytes under alkaline conditions to an anion-exchange material has been proposed [14,15] for enhancing selectivity.

Graphitized carbon black (GCB) has proved to be a valuable adsorbing material for the LSE of pesticides [16,17] in aqueous environmental samples. GCB cartridges proved to be more efficient than commonly used octadecyl (C_{18})-bonded silica

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cartridges for the LSE of polar compounds, such as phenols [10], chloroanilines [18] and certain very hydrophilic pesticides [19]. Although GCB is known to behave as a natural reversed phase, it contains on its surface chemical heterogeneities able to bind anions via electrostatic forces [20]. In previous papers [16,17,21] we have shown that this singular feature of the GCB material can be advantageously exploited for rapidly and simply isolating acidic analytes from co-extracted base–neutral compounds by differential elution. When applied to phenols, however, base–neutral/acid fractionation by stepwise desorption failed to isolate from base–neutral species phenols having pK_a values higher than 7 [21].

Recently, selective determinations of triazine herbicides [22] and chloroanilines [19] in water have been achieved by a two-trap tandem system, one containing GCB and the other filled with a strong cation exchanger. The object of this work was to evaluate the ability of a two-trap tandem system, with one cartridge filled with a GCB and the other containing a conventional anion exchanger, to achieve in a simple and rapid manner the simultaneous extraction and isolation of phenolic compounds from water samples.

EXPERIMENTAL

Reagents and chemicals

Authentic phenols were obtained from various sources. Individual standard solutions were prepared by dissolving 100 mg of each phenol in 100 ml of methanol. For recovery studies, we prepared a working composite standard solution, whose composition was as follows with concentrations of the analytes in mg/l (in parantheses): guaiacol (20); *p*-nitrophenol (15); *p*-cresol (30); *o*-chlorovanillin (10); *o*-chlorophenol (25); 2,4-dichlorophenol (15); *o*-nitrophenol (15); 2,4-dimethylphenol (40); 2,4-dichlorophenol (20); 3,5-dibromo-4-hydroxybenzonitrile (bromoxynil) (15); 4,6-dinitro-*o*-cresol (25); 3,5-diiodo-4-hydroxybenzonitrile (ioxynil) (15); 2,4,6-trichlorophenol (30); 3,4,5-trichlorosyringol (25); 2,3,4,6-tetyrachlorophenol (25); 2-*sec*.-bytyl-4,6-dinitrophenol (dinoseb) (30); and pentachlorophenol (30).

Fulvic acids were kindly donated by Dr. A. Piccolo, who prepared them as reported elsewhere [23].

For HPLC, distilled water was further purified by passing it through a Norganic cartridge (Millipore, Bedford, MA, USA). Acetonitrile and methanol of gradient grade were obtained from Riedel-de Haën, Selze, Germany. All other solvents and reagents were of analytical-reagent grade (Carlo Erba, Milan, Italy). Trifluoroacetic acid (TFA) and tetramethylammonium hydroxide pentahydrate (TMAOH) were obtained from Aldrich (Milwaukee, WI, USA).

Phenols were desorbed from the two in-line traps by passing sequentially through them two suitable eluent systems, which will be called eluents A and B; eluent A was CH_2Cl_2–CH_3OH (60:40, v/v) and eluent B was 0.25 mol/l formic acid in CH_2Cl_2–CH_3OH (90:10, v/v).

Apparatus

A 300-mg GCB extraction cartridge, commercially referred to as Carbograph 1 (Carbochimica, Rome, Italy), was prepared and pretreated as reported elsewhere [16,17]. A 50-mg amount of Sephadex QAE A-25 (particle size 40–120 μm) (Aldrich) was packed in a plastic tube (6 cm × 0.5 cm I.D.) (Supelco, Bellefonte, PA, USA). The upper polyethylene frit (Supelco) was located about 2 mm above the exchanger bed to allow it to swell on passing basified water. The connection between the two cartridges was made with a suitable plastic adapter (Supelco). The strong anion-exchange (SAX) material was converted from the Cl^- to the OH^- form by washing it with 15 ml of 0.1 mol/l sodium hydroxide in water. The excess amounts of OH^- anions and water were eliminated by washing the exchanger bed with 2 ml of methanol.

The GCB cartridge was fitted into a side-arm filtration flask and liquids were forced to pass through the cartridge by vacuum (water pump).

Procedure

Aqueous samples were fortified with known volumes of the working composite standard solution of phenols. When analysing hypochlorite-containing tap water samples, hypochlorite was reduced in advance by adding 0.4 g/l of sodium sulphite to prevent oxidation of the analytes. Water samples were then shaken for 1 min and after about 10 min were poured into a glass reservoir that was connected to the GCB cartridge. Unless they contained algae and

debris, which were eliminated by filtering the water sample through Whatman GF/C glass-fibre pads (pore size 10 μm), river water samples were extracted as collected after adding phenols to them. Water was forced to pass through the cartridge at flow-rates of 110–130 ml/min. Just after the sample had passed through the column, the cartridge was filled with 7 ml of HCl-acidified water (pH 3), which was allowed to pass through the cartridge at a flow-rate of 5–7 ml/min.

Following the passage of large volumes of water, some shrinkage of the sorbent bed may occur. In such an event, before washing with acidified water, the upper frit was pushed against the top of the sorbent bed. This expedient facilitates the subsequent removal of water from the extraction cartridge and improves the effectiveness of the eluent systems as they can permeate the sorbent bed more homogeneously.

After the acidified water had passed through the trap, most of it was removed by reducing to the minimum pressure in the flask for 30 s. The water pump was disconnected, 0.5 ml of methanol was poured into the cartridge, the pump was linked to the flask again and methanol was passed slowly through the sorbent bed to eliminate residual water. Thereafter, the GCB cartridge was connected to the SAX cartridge and 8 ml of the eluent phase A were poured into the GCB extraction cartridge (upper cartridge) and allowed to pass through the two cartridges at a flow-rate not exceeding 2.5 ml/min.

Eluent A leaving the SAX cartridge (lower cartridge) was discharged. If desired, this liquid phase could be submitted to a solvent reduction step for assaying non-acidic target compounds co-extracted with phenols from the water sample. As a consequence of the passage through the two cartridges of eluent A, phenols were distributed between the two cartridges. In particular, the most acidic phenols remaind still adsorbend on the GCB surface, while the least acidic phenols, washed away from the GCB cartridge together with non-acidic compounds, were readsorbed by the SAX cartridge. All of the phenols were eluted from the two sorbent beds by pouring 8 ml of eluent B into the GCB cartridge and allowing it to percolate through the two cartridges at a flow-rate not exceeding 2.5 ml/min. The last drops of eluent B were forced out of the two in-line traps by vacuum. The 8 ml of eluate was collected in a centrifuge tube with of *ca.* 1.4 cm I.D. The extract was basified by adding 0.52 ml of 3 mol/l TMAOH in methanol and then concentrated to about 320 μl in a water-bath at 30°C under a gentle stream of nitrogen. The methanolic solution of TMAOH, which served to basify the eluate prior to solvent reduction, was prepared weekly and stored in the dark at 4°C. When these precautions were not taken, some artifacts originated that produced chromatographic peaks, one of which overlapped that of bromoxynil. If the eluate evaporation step is terminated at volumes larger than that reported above, some methylene chloride may still be contained in the final extract. This solvent interferes with the subsequent separation and quantification by HPLC. After acidifying the solution with 170 μl of 6 mol/l HCl, the exact final extract volume was measured and 80 μl of it were injected into the HPLC system.

HPLC apparatus

A Model 5000 liquid chromatograph (Varian, Walnut Creek, CA, USA) equipped with a Rheodyne Model 7125 injector with a 100-μl loop and a Model 2550 variable-wavelength UV detector (Varian) was used with a 25 cm × 4.6 mm I.D. column filled with 5-μm LC-18 reversed-phase packing (Supelco). Phenols were chromatographed with premixed methanol–acetonitrile (10:90, v/v) containing 0.015% of TFA and water acidified with TFA (0.05%, v/v). TFA was stored in the dark at 4°C in order to avoid the formation of an artifact that produced a peak disturbing the determination of pentachlorophenol. The initial eluent composition was 34% organic modifier + 66% acidified water, which was increased linearly to 75% organic modifier after 24 min. The flow-rate was 1.5 ml/min. Phenols were detected with the UV detector set initially at 280 nm and then at 230 nm after 7.8 min.

The concentrations of the phenols in water samples were calculated by comparing the heights of the peaks obtained with the sample and with a standard solution. The latter was prepared by taking known and appropriate volumes of the working composite standard solution, by evaporating methanol and reconstituting the residue with 0.5 ml of water–methanol (60:40, v/v) acidified with HCl (pH 2).

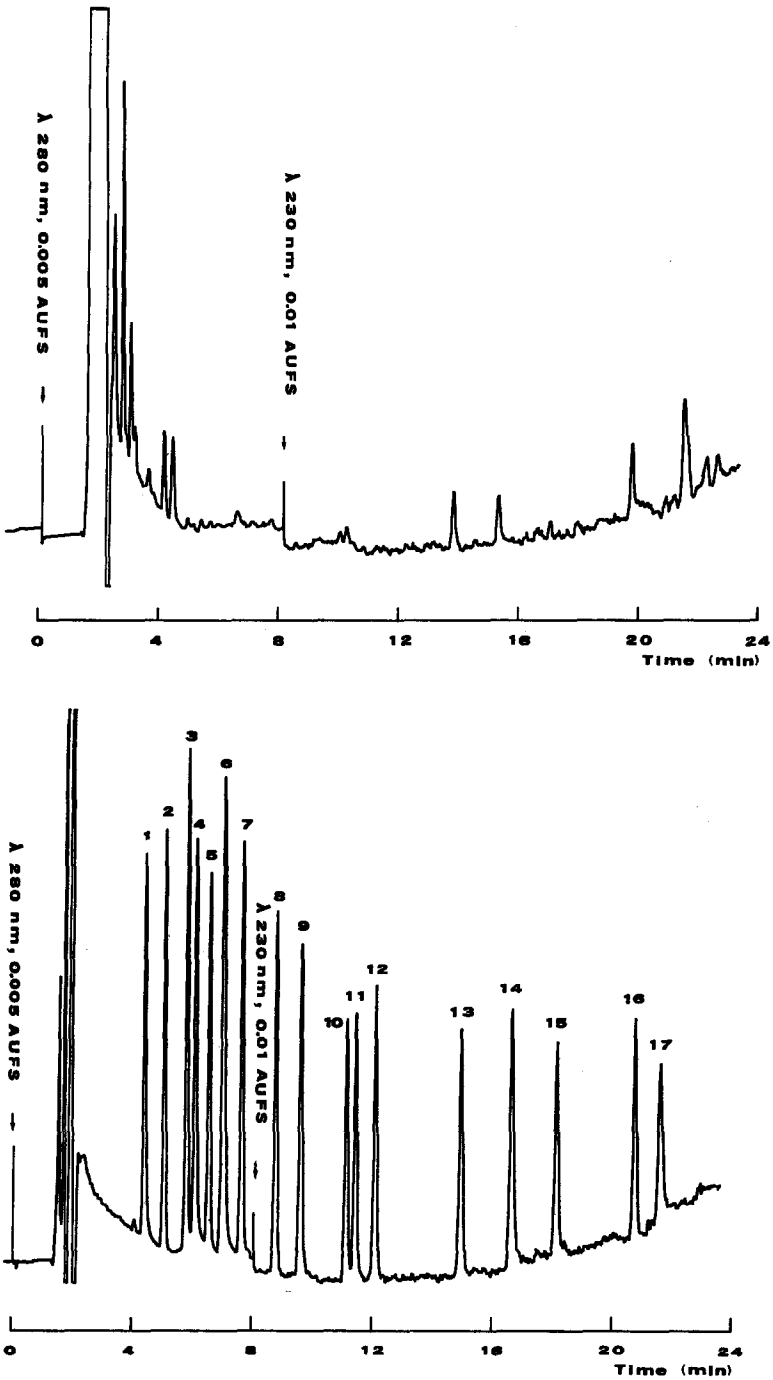

Fig. 1. Chromatograms of a typical overall blank procedure (top) and the working composite standard solution of phenols (bottom). Elution order (with amounts in ng of each phenol injected in parentheses): 1 = guaiacol (32); 2 = p-nitrophenol (24); 3 = *p*-cresol (48); 4 = *o*-chlorovanillin (16); 5 = *o*-chlorophenol (40); 6 = 2,4-dinitrophenol (24); 7 = *o*-nitrophenol (24); 8 = 2,4-dimethylphenol (64); 9 = bromoxynil (24); 10 = 2,4-dichlorophenol (32); 11 = 4,6-dinitro-*o*-cresol (40); 12 = ioxynil (24); 13 = trichlorophenol (48); 14 = trichlorosyringol (40); 15 = tetrachlorophenol (40); 16 = dinoseb (48); 17 = pentachlorophenol (48).

RESULTS AND DISCUSSION

Critical analytical variables

Originally, according to a previously reported method [21], the GCB cartridge was washed with slightly basified water (pH 8.1) after the water sample had passed through it. Under these conditions, however, about a 40% loss of 2,4-dimethylphenol was observed. In addition to benzpyrylium ions, we had experimental evidence for the existence of a few quinone groups on the GCB surface [20]. The partial loss of the 2,4-dimethylphenol may be explained by the fact that it is first oxidized by quinones under alkaline conditions and then added to them, according to a reaction mechanism reported elsewhere [24]. This problem was eliminated by replacing the alkaline wash with an acidic wash (pH 3).

Sorption of very weak acids on an anion exchanger under strictly anhydrous conditions probably takes place via salt formation or hydrogen bonding [25,26]. However, similarly to classical ion-exchange chromatography, a relatively long equilibration time is needed for the adsorption of eluates on the exchanger beads and their subsequent desorption. As a consequence, the broadening of the chromatographic band is strongly influenced by varying the flow-rate of the eluent phase. In order to avoid losses of the least acidic phenols, it is necessary that the flow-rates at which eluents B and especially A pass through the exchanger cartridge do not exceed 2.5 ml/min.

Fig. 1 shows chromatograms obtained by injecting an aliquot of the final extract relative to a blank overall procedure and an aliquot of the working composite standard solution. The extent of background interferences was calculated. When analysing 2 l of drinking water, the background for guaiacol was 25 ng/l whereas for the other phenols the background interferences were less than 4 ng/l.

Recovery studies

The ability of the GCB cartridge to retain phenols quantitatively on passing through the GCB cartridge increasing volumes (0.5, 1 and 2 l) of tap water spiked with the phenols considered at individual levels of 0.5–2 μg/l was evaluated. The recoveries obtained from three determinations for each water volume considered showed that only when 2 l of tap water were extracted was about 10% of guaiacol lost in the water effluent.

Fulvic acids (FA) represent up to 80% of the soluble organic carbon in environmental waters [27]. It has been reported [28] that the presence of relatively large amounts of humic substances in aqueous samples can make LSE cartridges less efficient in extracting target compounds. Saturation of sorptive sites by FA or formation of chemical complexes occurring between hydrophobic analytes and FA, which are scarcely retained by LSE cartridges, may be responsible for this failure.

The extent to which the efficiency of the proposed device for selectively extracting phenols was affected by the presence of fulvic acids in aqueous samples was assessed. For these experiments, two portions of a pure water sample were fortified with two different concentrations of FA, 5 and 10 mg/l, and with phenols at individual levels of 1–4 μg/l aliquots of 0.5 and 1 l of these two water samples were then analysed in triplicate. The results reported in Table I show that, among the phenols considered, the extraction efficiency of guaiacol, *p*-cresol and *o*-chlorophenol was to some extent affected by the presence of FA in the water samples. In order to account for the loss observed, both the water effluent from the GCB cartridge and eluent A which had passed through the Sephadex QAE cartridge were analysed. The amounts of the three phenols found in the water effluent completely accounted for the loss observed. Amog the phenols considered, the three phenols mentioned above have the highest mobilitis on the GCB cartridge. Therefore, it is conceivable that the incomplete adsorption of the three phenols was a result of the decrease in the number sorptive sites due to adsorption of FA rather than some kind of association occurring between phenols and FA with the formation of a complex having a low affinity for adsorption on the GCB surface. In our experience, surface water samples contain concentrations of FA lower than 10 mg/l. Anyway, when the GCB cartridge is used under field conditions, we recommend extracting volumes of water not larger than 0.5 l to monitor low-molecular mass phenols accurately.

Selectivity

Using the proposed extraction device, only acidic organic compounds can interfere with the subsequent HPLC analysis of phenols.

Linear alkyl benzenesulphonates (ALS) are wide-

TABLE I

RECOVERY OF PHENOLS FROM 0.5- AND 1.0-l ALIQUOTS OF WATER SAMPLES CONTAINING TWO DIFFERENT CONCENTRATIONS OF FULVIC ACIDS

Compound	Recovery (%)[a]			
	0.5 l		1 l	
	5 mg/l FA	10 mg/l FA	5 mg/l FA	10 mg/l FA
Guaiacol	93	86	81	69
p-Nitrophenol	98	100	101	98
p-Cresol	93	85	88	77
o-Chlorovanillin	97	97	99	87
o-Chlorophenol	98	85	93	76
2,4-Dinitrophenol	102	99	100	94
o-Nitrophenol	98	98	97	97
2,4-Dimethylphenol	97	98	98	97
Bromoxynil	99	99	100	100
2,4-Dichlorophenol	98	97	96	100
4,6-Dinitro-*o*-cresol	95	96	97	98
Ioxynil	99	100	100	101
2,4,6-Trichlorophenol	100	99	96	98
3,4,5-Trichlorosyringol	100	101	99	101
Tetrachlorophenol	100	101	99	101
Dinoseb	98	98	98	97
Pentachlorophenol	99	97	99	100

[a] Mean values obtained from triplicate measurements.

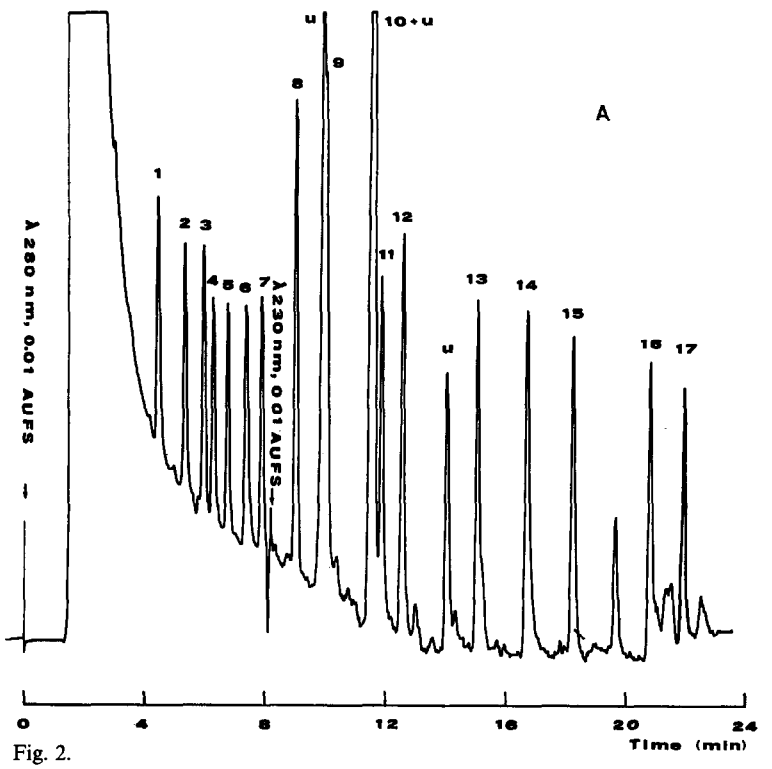

Fig. 2.

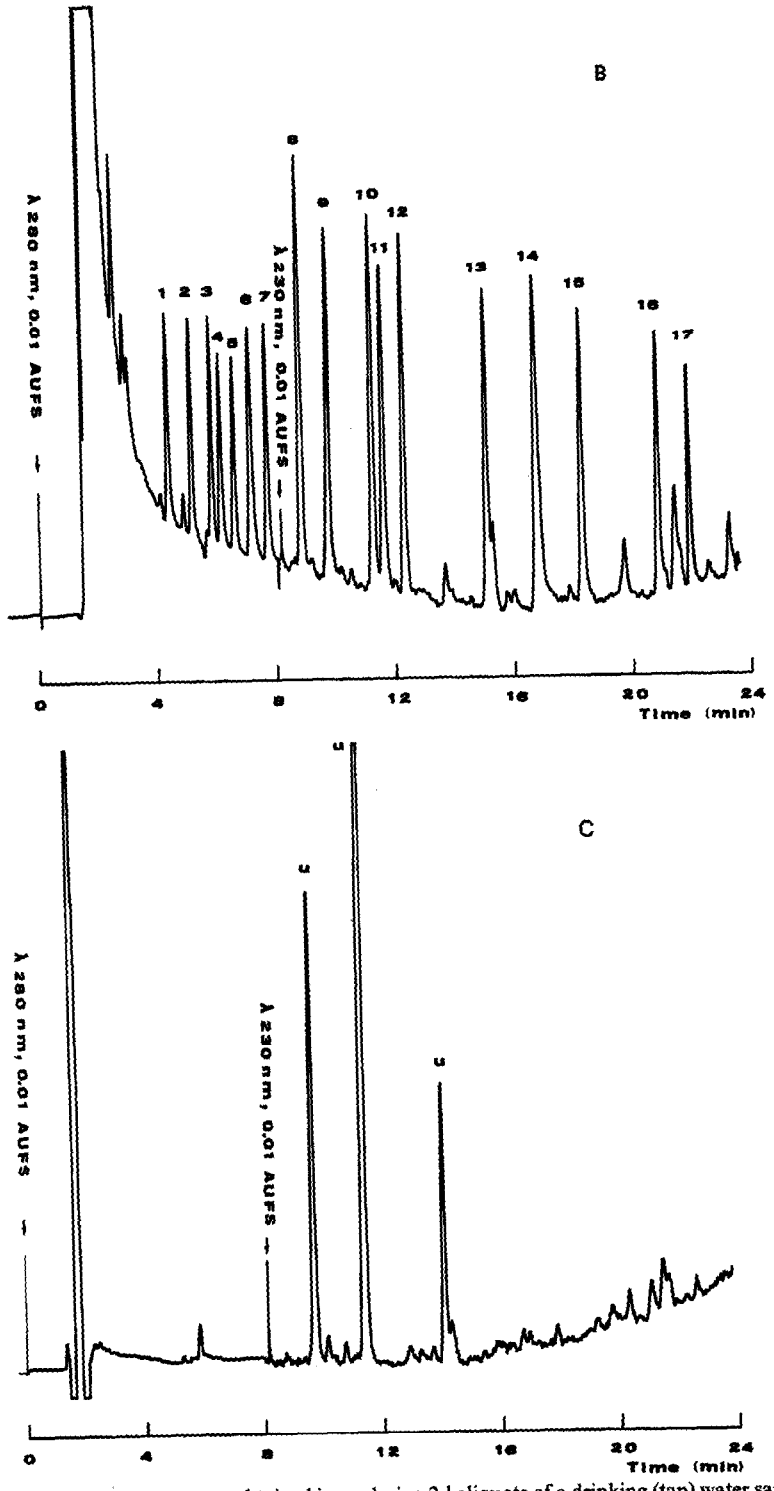

Fig. 2. Chromatograms obtained in analysing 2-l aliquots of a drinking (tap) water sample spiked with phenols at individual concentrations of 50–200 ng/l. Water was extracted with (A) the GCB cartridge alone and (B) the proposed method. The third chromatogram (C) resulted from injecting an aliquot of the base–neutral-containing extract. Peak numbers as in Fig. 1; u = unknown neutral compound.

ly used anionic surfactants and they frequently occur in aqueous environmental samples at levels ranging between 1 and 50 μg/l. LAS are co-extracted with phenols by the GCB cartridge, but the former are not eluted from it by eluent B, because formic acid contained in the mobile phase is unable to displace LAS from the positively charged sorption sites populating the GCB surface. Moreover, as reported elsewhere [21], LAS do not decrease the extraction efficiency of the GCB cartridge appreciably unless present in water at levels higher than 300 μg/l.

Among the most popular acidic pesticides, namely phenoxy acid derivatives, bentazone and dicamba, only the last compound can interfere with the determination of *o*-nitrophenol as these two compounds produced overlapping peaks.

As an example and to show the potential of this method in terms of selectivity and sensitivity, a tap water sample was spiked with phenols at individual concentrations of 50–200 ng/l and 2-l aliquots were analysed by the proposed method and by a conventional, non-selective method, such as that involving the use of a single GCB extraction cartridge for preparing the sample. In this case, the adsorbates were eluted from the GCB column by using only eluent B. Fig. 2 shows chromatograms obtained by the two procedures. As can be seen, the proposed method is less prone to positive bias than that involving the use of a single extraction cartridge.

Method comparison

The extraction efficiency of the two-stap assembly was compared with those obtained by using both a 0.5-g octadecyl-bonded silica (C_{18}) disposable cartridge (Supelco) and a 300-mg anion-exchange cartridge. The latter material was the same as that used with the GCB cartridge. The anion exchanger was converted into the OH^- form prior to use. For these experiments, 1-l aliquots of both a tap water sample and a sea-water sample were supplemented with phenols at individual concentration of 1–4 μg/l. Before extraction with the C_{18} cartridge, the pH of the water samples was adjusted to 2.5. Phenols trapped by the C_{18} cartridge were eluted with 6 ml of methanol. When using the conventional ion-exchange cartridge, the pH of the water was adjusted to 10.5 and the precipitated calcium salts were eliminated by filtration with a glass-fibre filter. After the water sample had passed through the ion-exchange cartridge, the latter was washed with 1 ml of methanol and phenols were eluted with 12 ml of eluent B, whose composition is reported under Experimental. The solvent reduction step was performed in the same way as that adopted in our method. No pH adjustment of the water samples was necessary when extracting with the GCB cartridge.

The recoveries reported in Table II show that the C_{18} cartridge failed, in both instances, to retain many of the phenols considered. Except for guaiacol, *p*-cresol and 2,4-dimethylphenol, the 300-mg anion-exchange cartridge exhibited a good capability to extract the other phenols considered from 1 l of water having a relatively low content of inorganic anions. In contrast, owing to competition of chloride ions with phenols for adsorption on the surface of the anion exchanger, poor recoveries of eight phenols were obtained when on 1 l of sea water. The extraction efficiency of the GCB cartridge was not affected by the ionic strength of the water sample. This positive feature of the GCB material has been extensively discussed elsewhere [21]. When extracting very weakly acidic phenols, it appears that the role played by GCB, when associated with a conventional high-capacity anion-exchange material, is mainly that of eliminating any inorganic ions so that the unique feature of the latter material can be fully exploited.

Recovery and precision

The recovery and the within-run precision of the proposed method with various concentrations of the seventeen phenols considered were assessed. A sample of tap water made 0.4 g/l in sodium sulphite was divided into two portions, which were spiked with the analytes at levels of 3–12 and 0.1–0.4 μg/l. Each portion was divided into six 2-l aliquots, each of which was analysed by the procedure. The results showed that the recovery of all the phenols was independent on their concentrations, demonstrating the absence of any adverse effect of irreversible adsorption by the materials composing the extraction apparatus. The relative standard deviations (R.S.D.s) at concentrations of 0.1–0.4 μg/l ranged from 2.36% for *p*-nitrophenol to 6.45% for guaiacol, and at 3–12 μg/l the R.S.D.s ranged from 0.63% for *p*-nitrophenol to 1.97% for *p*-cresol.

TABLE II

RECOVERY OF PHENOLS FROM 1 l OF TAP WATER AND SEA WATER BY THE PROPOSED METHOD COMPARED WITH THOSE FROM TWO OTHER EXTRACTION METHODS

Compound	Recovery (%)[a]					
	Anion exchanger		C_{18}		This method	
	Tap	Sea	Tap	Sea	Tap	Sea
Guaiacol	12	5	3	8	98	93
p-Nitrophenol	96	56	6	9	99	98
p-Cresol	27	7	8	10	97	92
6-Chlorovanillin	94	33	20	28	100	99
o-Chlorophenol	97	14	6	11	98	95
2,4-Dinitrophenol	98	95	7	13	96	98
o-Nitrophenol	93	47	7	9	97	97
2,4-Dimethylphenol	44	14	20	36	98	97
Bromoxynil	97	89	68	84	101	101
2,4-Dichlorophenol	94	73	20	32	99	100
4,6-Dinitro-*o*-cresol	98	93	40	56	95	95
Ioxynil	102	96	101	98	100	100
2,4,6-Trichlorophenol	98	89	88	85	98	98
3,4,5-Trichlorosyringol	99	90	93	94	96	97
Tetrachlorophenol	100	93	98	98	99	100
Dinoseb	99	94	96	97	101	101
Pentachlorophenol	101	97	99	100	97	97

[a] Mean values obtained from triplicate measurements.

Application to environmental samples

The affects that unknown compounds and FA dissolved in surface water samples can have on the recovery of the seventeen phenols considered were evaluated. Aliquots of 0.5 l of eight different river water samples (3.5–7.4 mg/l dissolved organic carbon) collected from various rivers flowing between Florence and Rome were spiked with phenols at individual levels of 1–4 μg/l and analysed. For the sake of conciseness, in Table III we reported accuracy data relating only to those three phenols (see above) whose recoveries are affected by the content of FA in water.

TABLE III

ACCURACY OF THE PROPOSED METHOD FOR THE DETERMINATION OF SELECTED PHENOLS IN RIVER WATER SAMPLES (n = 8)

Spike level: 2–3 μg/l.

Compound	Recovery (%)	Range (%)	R.S.D. (%)
Guaiacol	98.3	91–103	
p-Cresol	93.2	91– 98	
o-Chlorophenol	97.8	93–103	3.93

Limit of detection

Under the chromatographic conditions selected and extracting 2 l of drinking water, the limits of detection (signal-to-noise ratio = 3) for the seventeen phenols considered were between 2 ng/l (chlorovanillin) and 15 ng/l (pentachlorophenol).

Reusability of the LSE cartridge

The reusability of the SAX cartridge was evaluated by carrying out repeated analyses of phenols in tap water by making use of the extraction device that was renewed each time by changing only the GCB cartridge. The SAX cartridge was reactivated each time as reported under Experimental. After twenty such analyses the recovery of the phenols considered was unchanged within the precision of the method. The same experiments were conducted

with a GCB cartridge by repeatedly extracting phenols from 2-l aliquots of tap water. After each extraction, the GCB bed was restored with 3 ml of methanol and 5 ml of water. After five such extractions, the recovery of the analytes considered had not charged significantly, but the permeability of the columns was decreased to some extent, probably owing to suspendend particles in the water that plugged the upper frit. Such experiments were not performed with surface water samples.

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