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Optimisation of the microwave-assisted extraction in open cell of polycyclic aromatic hydrocarbons from soils and sediments

Study of moisture effect

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Abstract

Sample preparation remains a long and critical step for organic contaminant analyses in the environment. Classical extraction methods, like Soxhlet extraction, are time and solvent consuming. Some new techniques have been developed in the last few years (supercritical fluid extraction, microwave-assisted extraction, pressurized liquid extraction). Among them, microwave-assisted extraction at atmospheric pressure has appeared an interesting alternative method to Soxhlet extraction. In this present study the extraction of polycyclic aromatic hydrocarbons (PAHs) from solid environmental matrices (soils and sediments) by a microwave-assisted method has been studied. In a first part the experimental conditions have been optimised and different parameters adjusted using the SRM 1941a as a test matrix: time and power of irradiation, nature of solvent, percentage of water. Among all the parameters, the quantity of water is of primary importance to maximize the recoveries. With the optimised conditions applied to the SRM 1941a, the results obtained by the microwave-assisted extraction with 30% of water are in a good agreement with those obtained by Soxhlet. When compared to the certified values, the global recovery obtained by microwave-assisted extraction with 30% of water is 90%. The relative standard deviations obtained for the microwave-assisted extraction with 30% of water are in the same range as those given by the Soxhlet extraction and in good agreement with the certified values (relative standard deviations below 15%). The optimal conditions (30% of water, 30 ml of dichloromethane, 30 W, 10 min) have then been applied to the extraction of PAHs from various matrices (certified and natural ones; soils, sediments, sewage sludge) and the concentrations of PAHs obtained by microwave-assisted extraction are compared to those obtained by Soxhlet extraction. For all the samples tested in this study the recoveries for the microwave-assisted extraction using 30% of water are very good (more than 85%) in comparison to the Soxhlet extractions. The reproducibility of such an optimised procedure is also very correct (under 15% for Σ PAHs for all the matrices). The method developed has appeared to be not matrix-dependent. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Microwave-assisted extraction; Extraction methods; Soil; Sediments; Environmental analysis; Polynuclear aromatic hydrocarbons

1. Introduction

In organic contaminant analysis, sample prepara-

tion is a critical step in the global analytical cycle. It is generally time-consuming and often at the origin of most of the quantification errors. Among the various steps of a sample preparation procedure, the extraction is one of the most important. The quality of the final result in terms of precision and repro-

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ducibility is dependent on it. Classical liquid–solid extraction techniques such as Soxhlet extraction are laborious and solvent consuming techniques and there is a real need to replace them. Among the new techniques that have appeared these last few years microwave-assisted extraction seems to be a good alternative. Most of the studies have dealt with closed vessels [1–9] and few studies have been reported on the extraction of organic contaminants with open-vessel microwave (MW) apparatus [10–13]. If in the case of volatile compounds microwave systems using closed vessels appear to be necessary, in the case of the extraction of semi- or non-volatile hydrophobic contaminants such as polycyclic aromatic hydrocarbons (PAHs) or polychlorinated biphenyls (PCBs) the open system for which the microwaves are focused on the sample, presents evident advantages: homogeneity of the electromagnetic field, ease of use, safety of handling, possibility to extract important amounts of material (more than 30 g if needed for example in the case of low contaminated sandy sediments). Moreover the combination of atmospheric pressure and low temperature (normal b.p. of the extracting solvent) insures the absence of reactivity and safer experiments which is not always strictly the case with the closed vessels [14].

This paper discusses the use of open-vessel microwave-assisted extraction to recover PAHs from sediments and soils. Solvent type, power and time of irradiation are studied to determine the optimum conditions for extraction of PAHs from a Standard Reference Material, SRM1941a [marine sediment from the National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA]. The results are interpreted in terms of precision and reproducibility. The influence of the water content of the matrix to be extracted can also be a factor that influences the recovery of the analytes [11]. In the present work the influence of the moisture of the sediment on the recovery of PAHs has been studied using SRM1941a.

2. Experimental

2.1. Standards, solvents and reagents

The studied PAHs range from three-ring aromatic

compounds (phenanthrene, anthracene) to six-ring aromatic compounds (benzo[ghi]perylene) (Fig. 1). The used abbreviations for the PAHs are given in Fig. 1. The Standard Reference Material, SRM 2260, aromatic hydrocarbons in toluene (nominal concentration 60 µg/ml), a standard compound solution of 24 aromatic hydrocarbons (23 are certified) was provided by the NIST and used for calibration.

The compounds used as internal standards were perdeuterated PAHs. [²H₁₀]Phenanthrene, [²H₁₂]benzo[a]pyrene and [²H₁₂]benzo[ghi]perylene were bought from Cambridge Isotope Labs. (CIL, Andover, MD, USA), [²H₁₀]fluoranthene, [²H₁₀]pyrene and [²H₁₂]chrysene from MSD isotopes (Division of Merck Frost Canada, Montreal, Canada).

Pestnorm grade dichloromethane was purchased from Prolabo (Fontenay-sous-Bois, France). HPLC-grade isooctane (99.5% GC purity), HPLC-grade hexane (95% GC purity), toluene (multisolvant, 99.8% GC purity), acetone (multisolvant, 99.7% GC purity) and pentane (99% GC purity) (Scharlau, Barcelona, Spain) were purchased from ICS (St. Médard en Jalles, France). Pentane was distilled. The copper (40 mesh, 99.5% purity) (Aldrich, Saint Quentin Fallavier, France) was activated with hydrochloric acid (7 M), then washed with water, acetone and dichloromethane. The alumina (150 basic, type T, 0.063–0.2 mm) and the silica (silica gel, 0.063–0.2 mm) (Merck, Darmstadt, Germany) were washed with dichloromethane, deactivated at 150°C overnight and then stored at 150°C.

2.2. Standard reference materials and natural matrices

Different samples have been used in this work (Table 1): some of them belong to the Standard Reference Materials that are available for optimisation and validation of analytical procedures [distributed by the NIST or by the Community Bureau of Reference of the European Commission (BCR, Brussels, Belgium)] [15–19]; the others are natural soil or sediment samples.

2.3. Microwave-assisted extraction procedure

2.3.1. Apparatus

MW-assisted extractions in open cells are per-

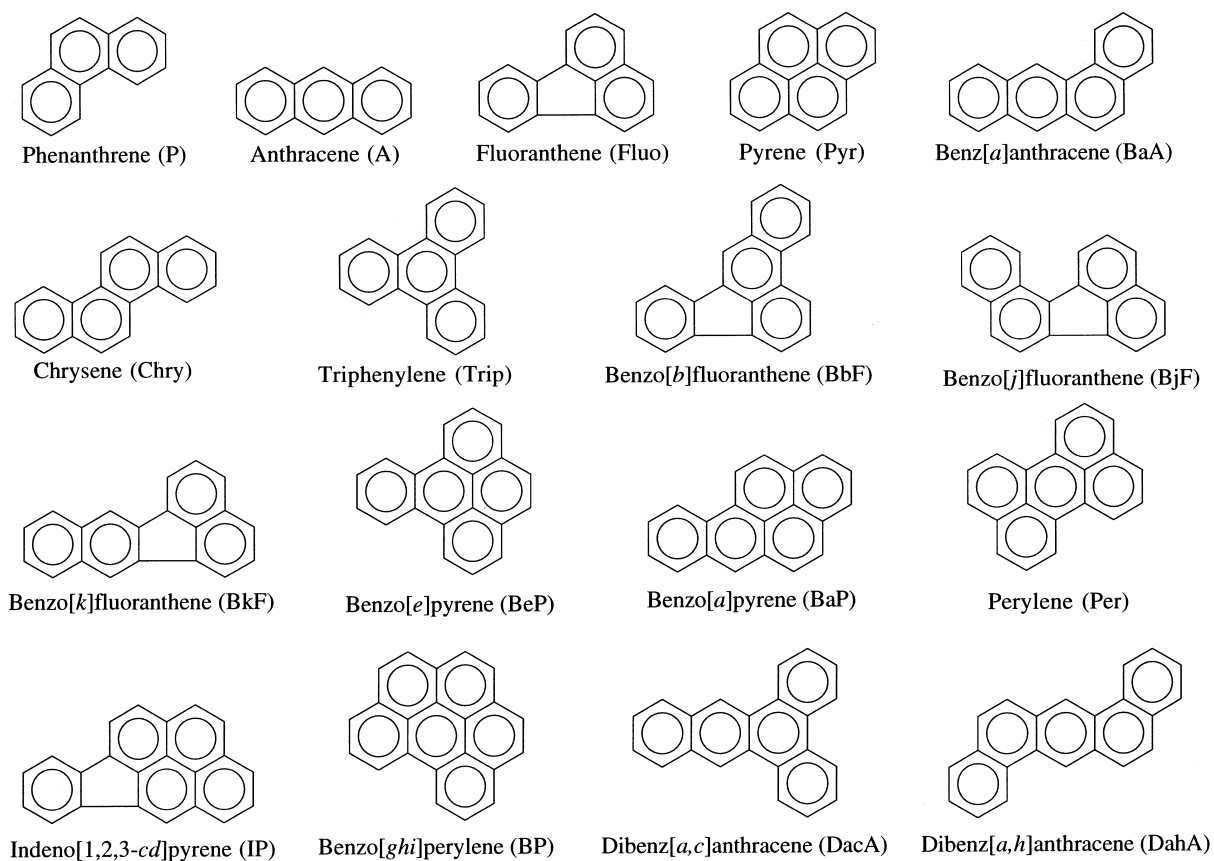


Fig. 1. Structure of the studied PAHs (the used abbreviations are given in parentheses).

formed at a frequency of 2450 MHz using a Soxwave apparatus (Prolabo) with a programmable heating power (from 30 W to 300 W). The use of focused microwaves allows homogeneous and reproducible treatment of samples. The Soxwave proceeds at atmospheric pressure. Its schematic diagram is presented in Fig. 2.

2.3.2. Procedure

The MW extractions are performed using conditions as close as possible to those of Soxhlet extractions using 0.1 to 1 g of freeze-dried sediments and soils (Table 1). A solution containing the perdeuterated PAHs used for the quantification is added to the matrix prior to the extraction. Thirty ml of solvent are added. The power varies from 30 to 210 W and time varies from 5 to 30 min. Blank experiments are performed and used for correcting the concentration if needed. The extract is filtered

and is reduced to a small volume (a few millilitres) using a rotary evaporator. The organic extract is desulphurized on activated copper. Then it is purified as described below.

2.4. Soxhlet extraction procedure

Soxhlet extractions with dichloromethane as extracting solvent (2×250 ml) are performed using the same amounts of freeze-dried samples as the MW extraction (Table 1) spiked with perdeuterated internal standards for quantification. The extraction lasts for 48 h. Blank experiments are performed and used for correcting the concentrations if needed. The extract is reduced to a small volume (a few millilitres) using a rotary evaporator. The organic extract is desulphurized on activated copper. Then it is purified as described below.

Table 1
Studied matrices with their type and the extracted amount of each of them

Sample name	Type	Extracted amount (g of dry sediment)	Ref.
SRM 1941a	Reference material Marine sediment	1	[15]
SRM 1944	Candidate reference material Marine sediment	0.1	[16]
CRM 088	Reference material Sewage sludge	0.5	[17]
CRM 524	Reference material Soil	0.5	[18]
CRM 535	Reference material River sediment	0.5	[19]
Arcachon	Natural sample Marine sediment collected in a yachting harbour Freeze-dried and sieved at 300 μm	1	[10]
S2	Natural sample Industrial soil Freeze-dried and ground	1	
S3	Natural sample Industrial soil Freeze-dried and ground	0.1	

2.5. Purification

The extract is purified on a micro-column containing alumina by eluting with dichloromethane. This purified extract is then fractionated on a micro-column containing silica in order to collect separately saturated and aromatic compounds eluted with respectively pentane and a mixture of pentane–dichloromethane (65:35, v/v) [20]. The purified aromatic fraction is reduced to few μl in isoctane and analysed by gas chromatography coupled to mass spectrometry (GC–MS).

2.6. Gas chromatography–mass spectrometry conditions

The analyses are performed on an HP 5980 series II gas chromatograph (Hewlett-Packard, Palo Alto, CA, USA) equipped with a splitless injector (purge delay 1 min, purge flow 60 ml/min). The injector is

maintained at 270°C. The temperature program is: 50°C (2 min) to 290°C (20 min) at 5°C/min. The carrier gas is helium at a constant flow-rate of 1 ml/min. The capillary column used is a PTE-5 (Supelco, Bellefonte, PA, USA): 60 m \times 0.25 mm I.D., 0.25 μm film thickness. The gas chromatograph is coupled to an HP 5972 mass selective detector (electron impact: 70 eV, voltage: 2000 V) operated under the single ion monitoring (SIM) mode using the molecular ion of each compound at 1.23 scan/s. The interface temperature is maintained at 290°C.

The PAHs are quantified relatively to perdeuterated PAHs. The response factors of the different compounds are measured by injecting a solution of Standard Reference Material, SRM 2260, containing 23 PAHs at certified concentrations and spiked with perdeuterated compounds used as internal standards in the case of the extraction of the samples.

Some coelutions have been noted between structural isomers (Fig. 3): (i) chrysene is not totally

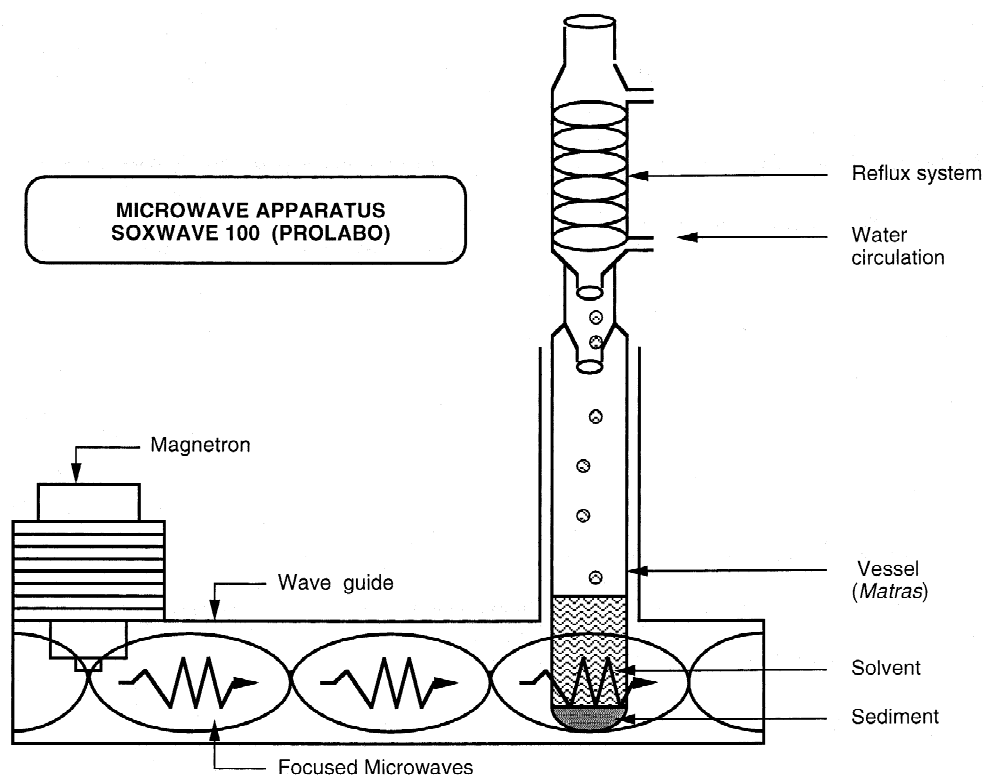


Fig. 2. Schematic of the used apparatus.

separated from triphenylene; (ii) benzo[*b*]fluoranthene coelutes with benzo[*j*]fluoranthene and is not sufficiently separated from benzo[*k*]fluoranthene; (iii) dibenz[*a,h*]anthracene coelutes with dibenz[*a,c*]anthracene. The concentrations given for the compounds suffering from these coelutions, take them into account: $\text{Chry}^* = \text{Chry} + \text{Trip}$, $\text{BF} = \text{BbF} + \text{BjF} + \text{BkF}$, $\text{DaA} = \text{DahA} + \text{DacA}$.

3. Results and discussion

3.1. Optimisation of MW conditions on SRM 1941a

The sediment SRM 1941a (NIST) has been chosen in order to test the influence of the different factors (power and time of irradiation, type of solvent and amount of water). At first the parameter which is considered to evaluate the influence of the factors is the sum of the concentrations of the

quantified PAHs (ΣPAHs) expressed in ng/g of sediment.

3.1.1. Influence of the type of solvent

Different solvents or mixtures of solvents classically encountered for the extraction of PAHs have been tested. When considering microwave-assisted extraction, due to the principle of microwave heating the choice of the solvent depends on its ability to absorb microwaves, defined by its dielectric constant ϵ . Apolar solvents such as aliphatic hydrocarbons do not meet this requirement and despite the fact they are known to be good solvents for aromatic compounds they cannot be used alone. On the other hand solvents like dichloromethane and acetone are also good solvents for aromatic compounds and they are quite polar. So dichloromethane, a mixture of dichloromethane–toluene (50:50, v/v), mixtures of acetone–hexane [50:50, v/v and 60:40, v/v (which is the azeotropic composition obtained in the case of

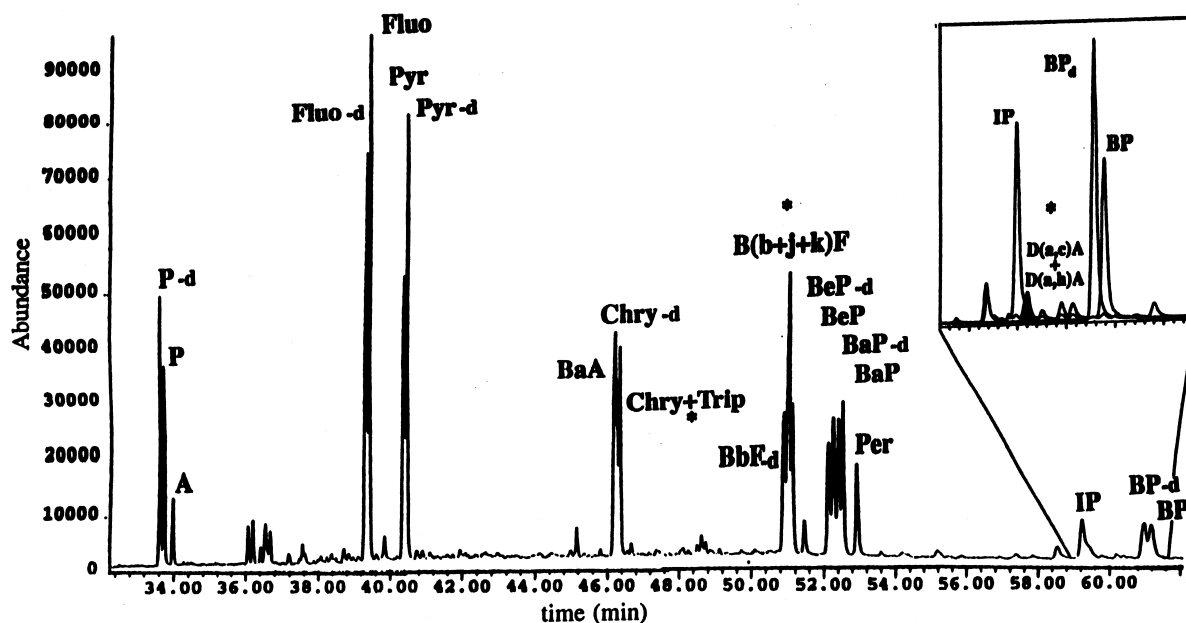


Fig. 3. Gas chromatogram (acquired in the selected ion monitoring mode) of an aromatic fraction of a MW sediment extract. Note the various coelutions (*).

the Soxhlet extraction)] and acetone have been tested. The microwave-assisted extraction conditions are the following: irradiation power=30 W; time of irradiation=10 min.

The results expressed as Σ PAHs (mean and relative standard deviation obtained for three replicates) are shown in Fig. 4A. When considering the relative standard deviations (RSDs) (up to 7%) there is no significant difference between the recoveries obtained with the various solvents. All of them give recoveries comprised between 5700 and 6700 ng/g for Σ PAHs. In terms of recovery and RSD, the solvents that have been tested are equivalent. This was not the conclusion of a previous study [11] which has shown that the dichloromethane was the best solvent to use but in this previous work the tests have been undertaken only once and no replicates have been performed. So it was not possible to take into account the heterogeneity of the matrix which was greater than the variations observed previously [11].

For the study the dichloromethane is chosen since it is easy to remove during reconcentration steps due to its low boiling point ($T=39.8^{\circ}\text{C}$) thus its use

prevents from analyte losses due to too drastic evaporation.

3.1.2. Influence of the time and the power of microwave irradiation

The power of microwave irradiation has been tested (with the time of irradiation fixed at 10 min) between 30 W (the minimum delivered by the system) and 210 W (the maximum power below which there is no bumping phenomenon) and the time of irradiation has been tested between 5 min and 30 min (with the irradiation power fixed at 30 W). The extractions have been performed with 30 ml of dichloromethane. The results are shown in Fig. 4B and C (Σ PAHs expressed as a mean with standard deviation for three replicates). When considering the reproducibility of the experiments (see the RSDs) it appears that neither the time nor the power of irradiation significantly influences the recovery. Based on practical considerations the time and the power of irradiation can be chosen at respectively 10 min and 30 W. When considering that the time needed to reach the boiling point is about 2 min (with dichloromethane at low power), a time of

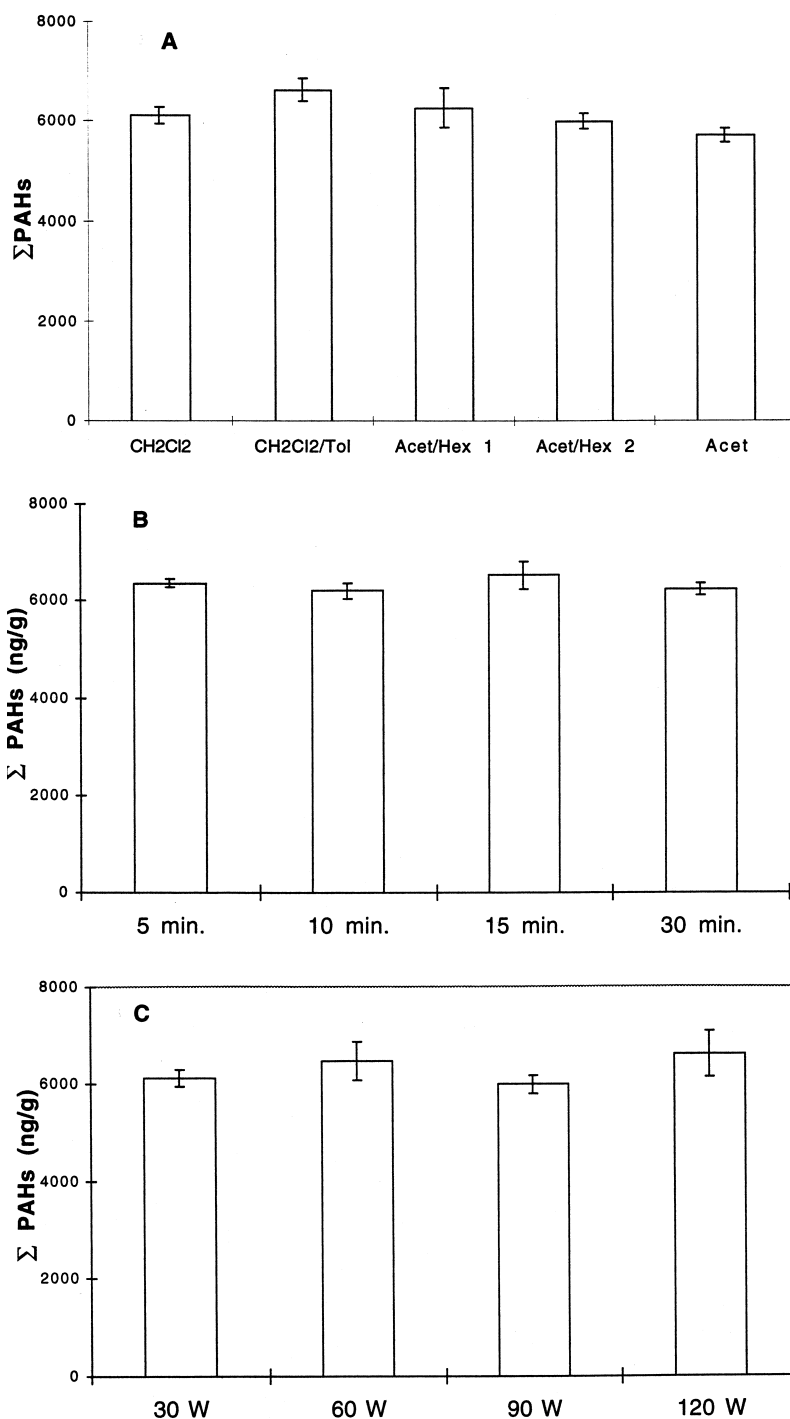


Fig. 4. Evolution of the total amount of PAHs (Σ PAHs in ng/g) that is extracted by MW-assisted extraction depending on various parameters: (A) type of solvent; (B) time of irradiation; (C) power of irradiation.

extraction of 5 min can be in some cases too short especially with matrices difficult to extract. In this respect an extraction time of 10 min insures a safety margin. Moreover, an extraction time of 10 min is short especially when compared to the time needed for the Soxhlet extraction (several hours) and allows the treatment of several samples per day. A power of irradiation fixed at 30 W allows savings of energy and insures safer implementation of the extraction.

3.1.3. Influence of the water content of the sediment

In most of the cases, extractions are performed on dry matrices. Indeed a lot of sediment and soil matrices are stored dried (freeze-dried or just dried) and then processed of course in a dry state. The drying allows a good and convenient storage of the samples for it prevents from evolutions. It allows also to obtain more homogeneous matrices. Moreover, most of the reported environmental studies provide data as content per dry mass. In the case of the extraction itself, it improves the efficiency of the conventional extraction processes such as the Soxhlet extraction [21]. Indeed, these conventional methods use very often hydrophobic extracting solvents which are non miscible with water and the removal of water from the matrix prevents from the formation of emulsions.

Nevertheless, the surface tensions of a solvent in the pores of a dry matrix can be sufficient to prevent the complete diffusion of the liquid in the micro-cavities of the matrix. It can be useful in such a case to humidify the matrix. The water demonstrates a swelling effect [22]. Moreover in the environment, especially in the aquatic (marine or continental) one, the natural samples that are collected are wet and show various amounts of water, from 20% for sandy samples to more than 40% for muddy ones.

In the case of the present work the way of heating (microwaves) used for the extraction is specifically well adapted to the water. The water present in the matrix can allow important local heatings and in this way this could favour the expansion of the pores and liberate the molecules in the solvent. This could accelerate the extraction phenomenon. Various authors [2,7,23–25] have tested the influence of the water on the extraction of organic contaminants from sediments and soils by microwaves under pressure

(closed vessels) and have attributed the increase of the recoveries to interactions between water and microwaves.

In the case of the present work, it has appeared interesting to study the influence of the water on the recovery of PAHs using MW-assisted extraction at atmospheric pressure. The influence of the water content of the matrix on the extraction efficiency has been tested from 0% to 100% (w/w) of water. In this case, the internal perdeuterated standards for quantification have been added just at the end of the extraction in the vessel (matras) in order to test with accuracy the recovery whatever the amount of water. In this way we have tried to minimize potential artefacts due to the poor miscibility of the solvent, the solutions of standards and the water. The extractions have been performed for 10 min with 30 ml of dichloromethane under 30 W. The results are shown in Fig. 5 (Σ PAHs expressed as a mean with relative standard deviation for three replicates). The total amount of extracted PAHs increases with increasing quantity of water (from 0% to 30% of water). With greater amount of water the recovery decreases slightly and when the quantity of water is important (100%, i.e., 1 ml) the recovery is very low (Σ PAHs=3290±860 ng/g, i.e., recovery=43%). When the amount of water becomes too important there are certainly problems of miscibility with the organic solvent used to extract. The water may act as a barrier and hinder the transfer of the analytes from the matrix to the solvent.

The higher water content limit appears to be 30% (i.e., 300 μ l). It allows a 10% increase of the global recovery. This quantity of water is important enough to be added accurately and low enough to allow the use of the extract without any drying step. It is also roughly the amount of water found classically for the natural sediments (around 20% for sands and 40% for muds). So this allows the use of natural sediments without any drying or freeze drying step prior to the extraction. This could be very useful for the treatment of samples directly in the natural field during for example an oceanographic cruise. This could represents an important time-saving.

The optimal conditions (30% of water, 30 ml of dichloromethane, 30 W, 10 min) have been applied to the extraction of PAHs from SRM 1941a (the perdeuterated internal standards for quantification

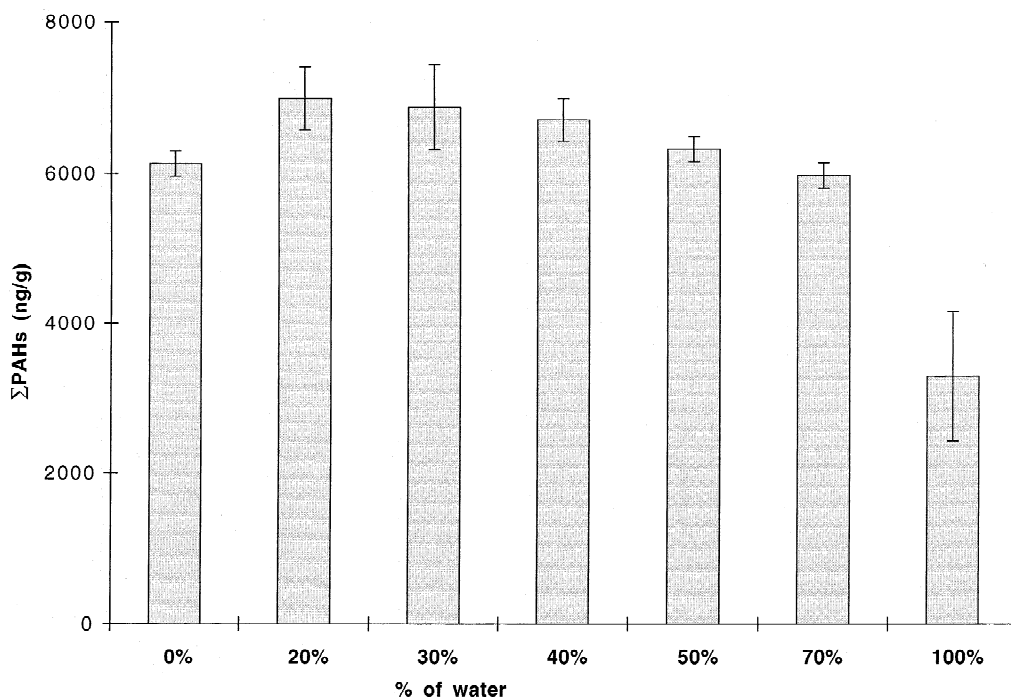


Fig. 5. Evolution of the total amount of PAHs (Σ PAHs in ng/g) that is extracted by MW-assisted extraction depending on the sediment water content (in mass percentage).

have been added prior to the extraction). The detailed results for each PAH, comparing the MW extraction without and with water, Soxhlet extraction and certified values, are summarised in Table 2 and presented in Fig. 6 for three replicates for the MW and Soxhlet extractions.

The results obtained by the MW extraction with 30% of water are in a good agreement with those obtained by Soxhlet (Fig. 6). When compared to the certified values, the global recovery obtained by MW extraction with 30% of water is 90%. The individual recoveries are for the less important around 80% (phenanthrene, anthracene, pyrene, fluoranthene, benzo[*a*]pyrene and perylene) and the others are around 100%.

The RSDs obtained for the MW extraction with 30% of water are in the same range as those given by the Soxhlet extraction and in good agreement with the certified values. They are below 8% in all the cases except for the indeno[1,2,3-*cd*]pyrene (IP) which shows a relative standard deviation of 16.2%.

This relative standard deviation could seem important but even for the certification it was quite strong, i.e., 14.4%. This relatively high values can be related to the structure of the compound which is heavy and can suffer from some GC interactions with the dibenzanthracenes (Fig. 3). If we compare with a structural isomer, the benzo[*ghi*]perylene (BP), this latter compound shows a lower RSD (6.2% for the MW with 30% of water). The difference of precision can be explained by the fact that to quantify IP and BP the perdeuterated homologue of BP, BP- d_{12} , is used. The use of BP- d_{12} can correct problems very well for BP but not so efficiently for IP.

3.2. Application to other matrices

The optimal conditions (30% of water, 30 ml of dichloromethane, 30 W, 10 min) have then been applied to the extraction of PAHs from various matrices presenting different characteristics in terms

Table 2

Individual concentrations (in ng/g) of the studied PAHs: (i) certified values; (ii) concentrations obtained by Soxhlet extraction; (iii) concentrations obtained by MW-assisted extraction; (iv) concentrations obtained by MW-assisted extraction with 30% of water^a

Compounds	Certified values		SOX		MW		MW – 30% water	
	Concentration (ng/g)	RSD (%)	Concentration (ng/g)	RSD (%)	Concentration (ng/g)	RSD (%)	Concentration (ng/g)	RSD (%)
P	489±23	4.7	489±18	3.7	347±5	1.4	381±18	4.7
A	184±14	7.6	171±16	9.4	121±2	1.7	131±7	5.3
Fluo	981±78	7.9	845±29	3.4	727±21	2.9	814±43	5.3
Pyr	811±24	2.9	701±56	8.0	602±33	5.5	658±42	6.4
BaA	427±25	5.8	368±3	0.8	324±16	4.9	359±12	3.3
Chry ^b	577±24 ^b	4.1	569±15	2.6	526±25	4.8	544±14	2.6
BF	1442±110 ^b	7.6	1337±90	6.7	1229±36	2.9	1300±101	7.8
BeP	553±59	10.7	527±20	3.8	512±5	1.0	533±36	6.8
BaP	628±52	8.3	488±22	4.5	461±7	1.5	500±29	5.8
Per	452±58	12.8	357±18	5.0	305±2	0.7	343±12	3.5
IP	501±72	14.4	561±21	3.7	476±24	5.0	554±90	16.2
BP	525±67	12.8	494±19	3.8	458±21	4.6	497±31	6.2
DaA	117±13 ^b	11.1	111±5	4.5	105±12	11.4	135±7	5.2

^a The concentrations are expressed as the mean of three replicates with the relative standard deviations for the MW and Soxhlet extractions.

^b Non-certified concentration.

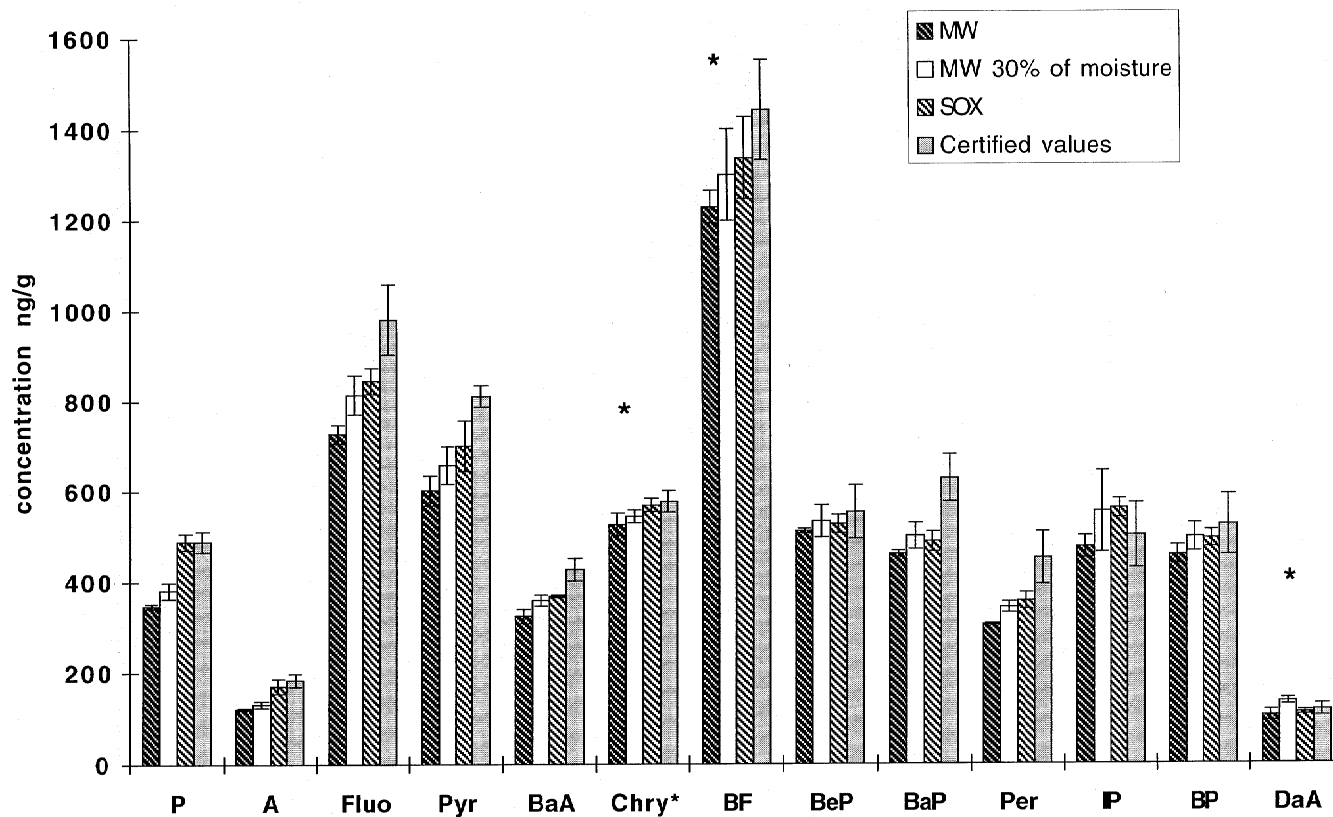


Fig. 6. Concentrations (in ng/g) of the studied PAHs in SRM 1941a obtained (i) by MW-assisted extraction; (ii) by MW-assisted extraction with 30% of water; (iii) by Soxhlet extraction; (iv) from certified values. Abbreviations: MW=Microwave, SOX=Soxhlet. * Non-certified concentrations.

of carbon (organic and inorganic) content, location of sampling, grain-size, type (i.e., soil, marine sediment, river sediment, sewage sludge), degree and origin of the contamination... The results are shown in Fig. 7. They are expressed as recoveries (in percentage) of Σ PAHs extracted by MW and MW with 30% of water in comparison to Σ PAHs extracted by Soxhlet. Three Soxhlet extractions have been performed for each matrix and the mean for each matrix has been considered as the reference value. Three MW extractions (without and with water) for each matrix have been performed and the

total amount of PAHs obtained (Σ PAHs) has been compared to the reference value. The recovery ranges from 76% to 99% for the MW-assisted extraction and from 85% to 106% for the MW-assisted extraction with 30% of water. If the recoveries are already good (near 80% to almost 100%) for the MW-assisted extraction on dry matrices, the use of water allows in most of the cases a significant increase (from 12% to more than 20%) of the extracted amount of PAHs as already observed for the SRM 1941a. The presence of water never hinders the efficiency of the extraction. If in some

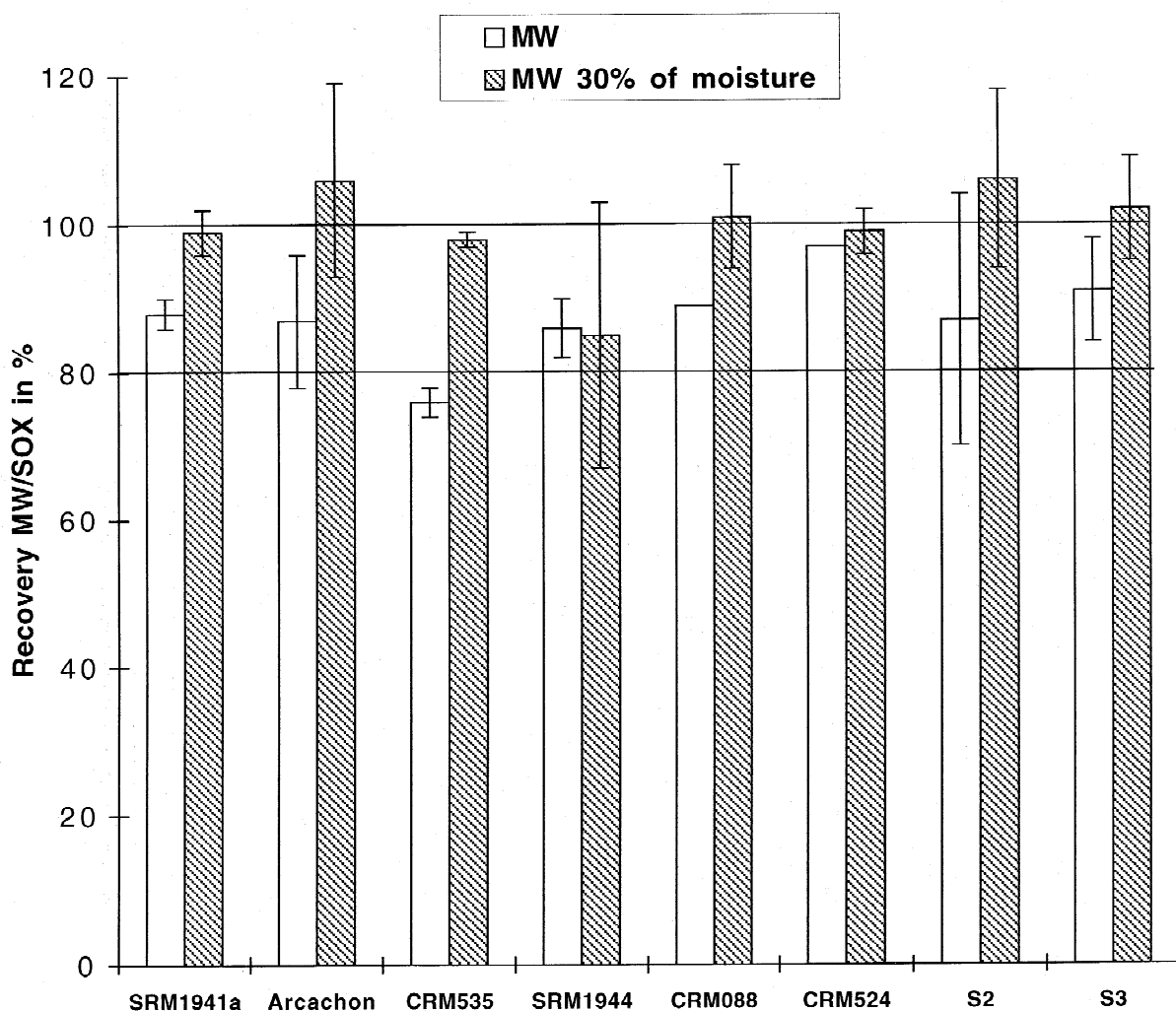


Fig. 7. Recoveries expressed as Σ PAHs for the MW-assisted extraction and the MW-assisted extraction with 30% of water given in comparison with the values obtained with the Soxhlet extraction. Abbreviations: MW=Microwave.

cases (SRM 1944, CRM 524) the addition of water does not lead to any increase of the recovery, it does not either generate lower recoveries. Thus the addition of water is of primary importance to maximize the recoveries.

For all the samples tested in this study the recoveries for the MW-assisted extraction using 30% of water are very good (more than 85%) in comparison to the Soxhlet extractions. The reproducibility of such an optimised procedure is also very correct (under 15% for Σ PAHs for all the matrices). Moreover all the studied solid samples demonstrate similar general behaviour under microwave irradiation for recovery of total PAHs. The method developed appears to be not matrix-dependent.

4. Conclusions

The MW-assisted extraction conditions have been studied and optimised. The study of various parameters (time, power, nature of solvent and moisture) has demonstrated that the time and the power of irradiation are not important parameters and that the classical solvents used for the Soxhlet extraction can be used for MW-assisted extraction. The amount of water on the contrary is an important parameter to take into account. The addition of water to the dry matrices allows an important increase of the recoveries of PAHs from solid matrices such as soils or sediments.

This study has allowed one to optimise the extraction conditions: 30 ml of dichloromethane, 10 min and 30 W for the MW irradiation, 30% of water. Under these conditions, for various types of matrices (soils and sediments), the recoveries are very good; in every cases they are greater than 85% and in most of the cases near 100%. The reproducibility is also satisfactory (relative standard deviations less than 15%). These optimised conditions have been applied to different matrices and have allowed to point out that MW-assisted extraction (with the optimised parameters) is not matrix-dependent. The role of the water is not totally clear but its presence can only have a positive effect on the recoveries.

The experimental results of this study allow to conclude that MW-assisted extraction at atmospheric pressure is a good alternative to Soxhlet extraction

for the analysis of PAHs in sediments and soils. Its main advantages in comparison to the Soxhlet extraction are: the reduction of the extraction time (10 min) and the reduction of the solvent volume (30 ml). Moreover this method is easy to manage and the optimised conditions using 30% of water seem to be convenient for all the solid matrices (soils or sediments). It is independent from the type of solid sample.

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References

- [1] V. Lopez-Avila, R. Young, W.F. Beckert, *Anal. Chem.* 66 (1994) 1097.
- [2] F.I. Onuska, K.A. Terry, *Chromatographia* 36 (1993) 191.
- [3] J.R.J. Paré, J.M.R. Bélanger, *Trends Anal. Chem.* 13 (1994) 176.
- [4] K. Ganzler, A. Salgo, K. Valco, *J. Chromatogr.* 371 (1986) 299.
- [5] I.J. Barnabas, J.R. Dean, I.A. Fowles, S.P. Owen, *Analyst* 120 (1995) 189.
- [6] M.P. Llompart, R.A. Lorenzo, R. Cela, K. Li, J.M.R. Bélanger, J.R.J. Paré, *J. Chromatogr. A* 774 (1997) 243.
- [7] V. Lopez-Avila, R. Young, J. Benedicto, P. Ho, R. Kim, *Anal. Chem.* 67 (1995) 2096.
- [8] K. Li, J.M. R. Bélanger, M.P. Llompart, R.D. Turpin, R. Singhvi, J.R.J. Paré, *Spectroscopy* 13 (1997) 1.
- [9] US Environmental Protection Agency (EPA) Method 3546 (SW846), US Government Printing Office, Washington, DC, USA.
- [10] M. Letellier, H. Budzinski, P. Garrigues, S. Wise, *Spectroscopy* 13 (1997) 71.
- [11] H. Budzinski, P. Baumard, A. Papineau, S. Wise, P. Garrigues, *PAC J.* 9 (1996) 225.
- [12] L.E. Garcia-Ayuso, M. Sanchez, A. Fernandez de Alba, M.D. Luque de Castro, *Anal. Chem.* 70 (1998) 2426.
- [13] S. Dupeyron, P.M. Dudermeil, D. Couturier, *Analisis* 25 (1997) 286.
- [14] E. Hwang, K.R. Vanderveen, presented at the 45th Pittsburgh Conference in Analytical Chemistry and Applied Spectroscopy, New Orleans, 5–10 March, 1995.
- [15] Certificate of Analysis for Standard Reference Material SRM 1941a, Organics in Marine Sediment, National Institute of Standards and Technology, Gaithersburg, MD, 1994.

- [16] M.M. Schantz, J.J. Nichols, S.A. Wise, *Anal. Chem.* 69 (1997) 4210.
- [17] Certificate of Analysis for Certified Reference Material CRM 088, Community Bureau of Reference of the European Commission, Brussels, 1994.
- [18] Certificate of Analysis for Certified Reference Material CRM 524, Community Bureau of Reference of the European Commission, Brussels, 1996.
- [19] Certificate of Analysis for Certified Reference Material CRM 535, Community Bureau of Reference of the European Commission, Brussels, 1997.
- [20] F. Behar, C. Leblond, C. Saint-Paul, *Rev. Inst. Fr. Pét.* 44 (1989) 387.
- [21] J.D. Haddock, P.F. Landrum, J.P. Glesy, *Anal. Chem.* 55 (1983) 1197.
- [22] T.M. Fahmy, M.E. Paulaitis, D.M. Johnson, M.E.P. McNally, *Anal. Chem.* 65 (1993) 1462.
- [23] V. Lopez-Avila, R. Young, W.F. Beckert, *Anal. Chem.* 66 (1994) 1097.
- [24] V. Lopez-Avila, R. Young, N. Teplitsky, *J. AOAC Int.* 79 (1996) 142.
- [25] R. McMillin, L.C. Miner, L. Hurst, *Spectroscopy* 13 (1997) 41.