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# Determination of organochlorine pesticides in water by membranous solid-phase extraction, and in sediment by microwave-assisted solvent extraction with gas chromatography and electron-capture and mass spectrometric detection.

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## Abstract

C<sub>18</sub>-impregnated disks for solid-phase extraction were evaluated for the extraction of organochlorine pesticides (OCPs) from water samples, and stability of the pesticides in storage. Good recoveries from different water samples were obtained. Microwave-assisted solvent extraction (MASE) was employed for the extraction of marine sediments spiked with OCPs. Factors affecting the efficiency of MASE were also investigated. Recoveries of 17 OCPs and 2 OCP surrogates were in the range 70.6–93.1% with mean deviation between 2.0 to 8.0%.

*Keywords:* Environmental analysis; Water analysis; Extraction methods; Pesticides; Organochlorine compounds

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## 1. Introduction

The contamination of the marine environment by organic pollutants is a matter of great concern. Seas, rivers and lakes are depositories of most effluent discharges, leachates and run-off from activities on land [1]. Persistence of some organic pollutants in sediment is possible owing to their low solubilities and also their tendency to associate with suspended particulate matter.

One class of organic pollutants which has rightly gained greater attention in environmental studies is the organochlorine pesticides (OCPs). They are highly persistent and toxic in nature, and one of

them, dieldrin, has been suspected to be carcinogenic [2].

In recent years, solid-phase extraction (SPE) has become an increasingly popular procedure in environmental analysis [3–5]. Recently, C<sub>18</sub>-bonded silica have been introduced as extraction disks in which they are enmeshed in a network of PTFE fibers to form a strong porous membrane. The suitability of these disks for field extraction and storage mechanism has also been investigated for some OCPs, organophosphorus insecticides and herbicides in groundwater and drinking water [2,6–12]. Comparisons between the stability of some pesticides in water and on C<sub>18</sub> disks have been made [12].

Soxhlet extraction is an established technique that has been used for the extraction of organic pollutants from marine sediment and soil samples [13,14]. The

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undesirability of this method includes long hours of exhaustive extraction, and large amounts of hazardous and toxic organic solvents. Recently, a novel microwave-assisted solvent extraction (MASE) procedure has been reported as a sample preparation technique for various solid matrices. Ganzler and co-workers [15,16] were the first to report the use of microwave energy to irradiate solid matrices such as soil, seeds, foods, and feeds in the presence of extracting solvents with high dipole moments. OCPs have been extracted from sediment samples using a domestic microwave oven with 5–6 times 30-s exposure to microwave energy [17]. Apart from OCPs, polycyclic aromatic hydrocarbons, selected base/neutral compounds and phenolic compounds have been extracted using a modified microwave digestion system with additional safety features [18]. With this method, the sample preparation time can be reduced to less than 30 min, and solvent consumption volumes to under 50 ml, in comparison to several hours and hundreds of milliliters of organic solvents for conventional techniques.

In the present work, we have evaluated both membranous SPE and MASE of 19 OCPs including 2 surrogate standards from natural water and sediments. The conditions affecting SPE were also investigated for more effective extraction of OCPs from natural waters. The chemical stability of the analytes on the disks was studied in order to demonstrate the applicability of these materials for field extraction or as storage devices during environmental screening. Additionally, conditions for the MASE recoveries of OCPs from marine sediment were optimized.

## 2. Experimental

### 2.1. Equipment

A Hewlett-Packard (Palo Alto, CA, USA) 5890A Series II gas chromatograph equipped with a  $^{63}\text{Ni}$  electron-capture detection (ECD) system and a Hewlett-Packard 3396 Series II integrator was employed for the analysis of sediment samples. A 25 m $\times$ 0.25 mm I.D. Ultra-2 capillary column (0.32  $\mu\text{m}$  film thickness) (Hewlett-Packard) was used for separations. Nitrogen was used as the carrier gas at a

flow-rate of 1.40 ml/min (at 100°C). A split/splitless injector was used in the splitless mode. The injector and detector temperatures were 270 and 360°C, respectively. For mass spectrometric (MS) analysis (water samples), a Hewlett-Packard 5890 Series I gas chromatograph coupled to a Hewlett-Packard 5988A mass spectrometer was used. The quantitation ions used for GC-MS in selective ion monitoring (SIM) detection of OCPs are given in Table 1. A Hewlett-Packard HP-5 capillary column (25 m $\times$ 0.25 mm I.D.; 0.25  $\mu\text{m}$  film thickness) was used. The carrier gas was helium at a flow-rate of 1.29 ml/min. The injector, transfer line, and ion source temperatures were 270, 280, and 200°C, respectively. The following temperature programme was used for both GC-ECD and GC-MS with a 2- $\mu\text{l}$  injection volume: 100°C (1-min hold), increase at 25°C/min to 170°C; increase at 2°C/min to 270°C and increase at 25°C/min to 290°C (3-min hold).

A standard Millipore (Bedford, MA, USA) 47-mm filtration apparatus was connected to a vacuum line, and nylon-66 and PTFE filter (0.45  $\mu\text{m}$  pore size) from Supelco (Bellefonte, PA, USA) were used for filtration of water samples.

MASE was carried out using a Model MES-1000 microwave extraction system (CEM, Matthews, NC, USA) equipped with a solvent detector. The MES-1000 is able to extract 12 samples simultaneously in PTFE-lined extraction vessels under the same conditions (temperature and pressure). An inboard pressure control system is installed for monitoring and controlling pressure conditions inside the extraction vessels.

### 2.2. Chemicals

All organic solvents used were of pesticide grade. A standard mixture of 17 OCPs (Ultra-Scientific, North Kingston, RI, USA) at concentrations of 250  $\mu\text{g}/\text{ml}$  per component dissolved in hexane-toluene (1:1, v/v) was used. Individual OCPs were of analytical grade obtained from Polyscience (Niles, IL, USA). 2,3,5,6-Tetrachloro-*m*-xylene (TCMX) and dibutyl chlorendate (Ultra-Scientific) were used as surrogate standards. Further dilution with ethyl acetate was carried out to give working standards comprising 0.5  $\mu\text{g}/\text{ml}$  of each component. Anhydrous sodium sulphate of analytical grade (Merck,

Table 1  
List of SIM acquisition groups, quantitation ions, detection limits and retention time data for the analysis of OCPs using GC–SIM–MS

OCPs	SIM acquisition groups	Quantitation ions ( <i>m/z</i> )	Detection limits ( $\mu\text{g/l}$ )	Retention time (min)
TCMX <sup>a</sup>	1	207,244	0.05	6.36
$\alpha$ -BHC	2	183,219	0.10	7.35
$\beta$ -BHC	2	183,219	0.13	8.16
$\gamma$ -BHC	2	183,219	0.10	8.31
$\delta$ -BHC	2	183,219	0.15	9.15
Heptachlor	3	100,237	0.10	10.92
Aldrin	4	263,265	0.36	12.44
Heptachlor epoxide	5	253,353	0.45	14.79
Endosulfan I	6	241,277	0.50	16.56
Dieldrin	7	79,246	0.06	18.16
<i>p,p'</i> -DDE	7	246,317,318	0.04	18.50
Endrin	7	79,263	0.17	19.50
Endosulfan II	8	165,207,235,237,250,345	0.50	20.21
<i>p,p'</i> -DDD	8	165,207,235,237,250,345	0.05	21.34
Endrin aldehyde	8	165,207,235,237,250,345	0.19	21.58
Endosulfan sulphate	9	165,229,272	0.22	23.28
<i>p,p'</i> -DDT	9	165,235	0.05	23.97
Methoxychlor	10	227	0.01	28.85
Dibutyl chlorendate <sup>a</sup>	10	99,387	0.04	31.44

<sup>a</sup> Surrogate standards.

Darstadt, Germany) was heated with 400°C for at least 4 h and pre-rinsed before use.

Extraction disks (17 mm; 3M, St. Paul, MN, USA) impregnated with C<sub>18</sub>-bonded silica Bakerbond (J.T. Baker, Philipsburg, NJ, USA) were used for extractions from water samples.

### 2.3. Methods

Water sampled from the west of Singapore island (<1 km off-shore and in the Straits of Johore) was used for recovery studies and trace analysis of OCPs. Aliquots of 10 ml of methanol per litre of water samples were added as wetting agent for the extraction disks [11]. Different natural water samples were spiked with the working pesticide standards to give a concentration of 0.5  $\mu\text{g/l}$  of each component.

The extraction disk was conditioned with, successively, 10 ml ethyl acetate, 10 ml methanol and 10 ml water [from a Milli-Q water purification system (Millipore)]. During conditioning, the vacuum was applied to draw half the volume of each solvent through the disk; the filtration was interrupted for 1 min before the vacuum was reapplied to pull through the remainder of the solvent. An aliquot of 1 l of

water sample was passed through the extraction disk under a vacuum of 68 kPa (gauge reading). After a 30-min extraction, the disk was allowed to dry for 5 min by pulling air through it. The pesticides were then eluted from the disk with 5 ml of ethyl acetate followed by 5 ml of hexane. Anhydrous sodium sulphate was used to dry the eluent. The dried eluate was then pre-concentrated down to 0.5 ml prior to GC–MS analysis.

For comparison of storage stability of OCPs, 1 l of distilled water was fortified with working standards in an amber glass bottle, and then extracted as described above. Disks were wrapped in aluminium foil and placed in 50-ml screw-capped glass vials with PTFE-lined caps. Extraction, elution, concentration and quantitation of OCPs either in stored disks or water were carried out immediately in order to normalise for differences, and achieved recoveries at time zero.

A comparison between MASE and Soxhlet extraction of marine sediment spiked with OCPs was carried out. The sediment samples were collected from the Straits of Johore (between southern peninsular Malaysia and Singapore island). The sediment samples used for optimization and recovery

studies were pre-extracted three times with dichloromethane and the third washing was injected to confirm the blank level. For MASE, 5-g samples were accurately weighed and quantitatively transferred into PTFE-lined extraction vessels of the MES-1000. After extraction, the vessels were allowed to cool before the caps were opened. After a series of filtration, washing and centrifugation, the volume of the filtrate was reduced to 1 ml prior to analysis. Sequential optimization procedure was employed to optimize the MASE parameters (extracting solvent, extraction temperature and duration of extraction). Extracting solvents evaluated for the MASE include dichloromethane, hexane–acetone (1:1, v/v) and acetone–light petroleum (boiling range 30–60°C) (1:1, v/v). Extractions were performed at 80, 100 and 120°C for 5, 10, 20 min at 50% microwave power. The optimized MASE conditions were then applied to recovery studies and genuine sediment samples. For sediments subjected to Soxhlet extraction, a 10-g sample was weighed and quantitatively transferred into a pre-extracted thimble (using 300 ml of dichloromethane) for extraction for 16 h. Only half the volume of the extracting solvent was pre-concentrated to 1 ml prior to analysis.

### 3. Results and discussion

#### 3.1. Membranous SPE of OCPs in water

SPE recoveries of the OCPs from different natural water samples at 0.5 µg/l levels as determined by GC–MS, are given in Table 2. For clean water samples, distilled water and tap water gave similar recoveries in the range 80.0–120.8% and 75.0–119.0%, respectively. In the case of spiked sea water samples, unfiltered sea water exhibited a lower recovery compared to filtered sea water. Recoveries of spiked unfiltered sea water ranged from 49.8 to 72.3% while those of spiked filtered sea water were in the range 64.8–110.1%. The low recovery obtained for the unfiltered sea water might have been due to adsorption of OCPs onto particulate matter present in the water and also the clogging of the disk by the particulate matter, resulting in a reduction of the extraction efficiency. The effect of particulate

Table 2  
Solid-phase extraction with disks of OCPs spiked in various water samples

OCPs	Recovery (%) <sup>a</sup>			
	A <sup>b</sup>	B <sup>b</sup>	C <sup>b</sup>	D <sup>b</sup>
TCMX <sup>c</sup>	83.0	82.1	67.5	82.9
α-BHC	87.0	81.9	65.0	124.1
β-BHC	83.9	81.0	61.0	97.4
γ-BHC	80.0	80.6	63.2	110.1
δ-BHC	80.0	75.7	59.8	92.1
Heptachlor	120.8	110.3	61.2	78.4
Aldrin	88.0	84.7	49.8	73.6
Heptachlor epoxide	88.4	80.5	63.6	84.8
Endosulfan I	82.6	85.5	58.5	72.5
Dieldrin	80.7	76.1	53.2	81.8
<i>p,p'</i> -DDE	95.1	89.8	57.2	86.3
Endrin	85.2	86.6	64.7	78.7
Endosulfan II	98.7	94.1	59.8	81.6
<i>p,p'</i> -DDD	82.6	75.0	55.5	84.9
Endrin aldehyde	95.3	87.5	62.6	84.2
Endosulfan sulphate	88.7	98.3	64.8	64.8
<i>p,p'</i> -DDT	89.2	79.8	57.7	107.8
Methoxychlor	111.9	119.0	64.6	89.8
Dibutyl chlorendate <sup>c</sup>	88.7	90.9	72.3	78.8

<sup>a</sup> Mean of two determinations.

<sup>b</sup> A=distilled water spiked with 0.5 µg/l of each compound; B=tap water spiked with 0.5 µg/l of each compound; C=unfiltered sea water spiked with 0.5 µg/l of each compound; D=filtered sea water spiked with 0.5 µg/l of each compound.

<sup>c</sup> Surrogate standards.

matter on the extraction efficiency of the membranous SPE is not considered to be very serious. Filtered sea water has similar recoveries when compared to spiked clean water samples. Thus, filtration prior to membranous SPE of natural water would appear to be necessary when the level of humic acid or particulate matter is too high (>10%), as adsorption and clogging may affect the extraction efficiency of SPE. The particulate level of OCPs should also be investigated separately in natural waters containing high levels of particulate matter (see below). Nevertheless, disk-based SPE is well suited for extraction of large volumes of “less dirty” water samples.

GC–SIM–MS was used for the determination of OCPs during the preliminary survey of coastal sea water. The lists of SIM acquisition groups and their respective quantitation ions for the OCPs are provided in Table 1. The entire analysis for the 17 OCPs and 2 surrogate standards could be completed within 32 min. Retention time data for individual com-

ponents OCPs are provided in Table 1. Calibration of the OCPs and surrogate compounds was carried out with standards at 1, 5, 10 and 25  $\mu\text{g/ml}$  concentrations. The regression coefficients ( $r^2$ ) for the 19 individual compounds were all better than 0.974. A total ion chromatogram of these compounds is shown in Fig. 1. At a signal-to-noise ratio of 3, the GC-MS detection limits were determined to be between 0.01–0.50  $\mu\text{g/l}$  (Table 1). Only two OCPs ( $\gamma$ -BHC and heptachlor) were detected and confirmed by GC-SIM-MS detection during the preliminary survey of 10 water samples collected from the Straits of Johore. The average concentrations of  $\gamma$ -BHC and heptachlor were 2.12  $\mu\text{g/l}$  and 8.63  $\mu\text{g/l}$ , respectively, and their relative standard deviations (R.S.D.s) were not more than 15.0%. The total ion chromatogram of the coastal sea water extract is given in Fig. 2.

### 3.2. Storage stability of OCPs

Table 3 shows the extraction recoveries of OCPs under different storage regimes. Higher average recoveries were obtained for the 17 OCPs and 2 surrogate standards stored on disks at 4°C. Despite

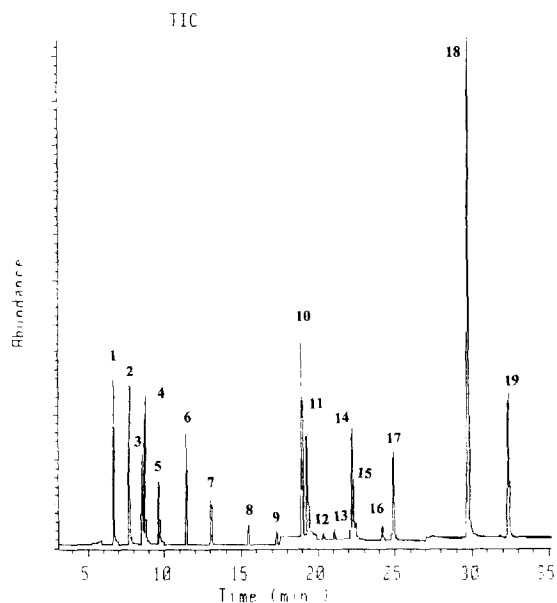


Fig. 1. Total ion chromatogram of a standard solution of organochlorine pesticides and pesticide surrogate compounds.

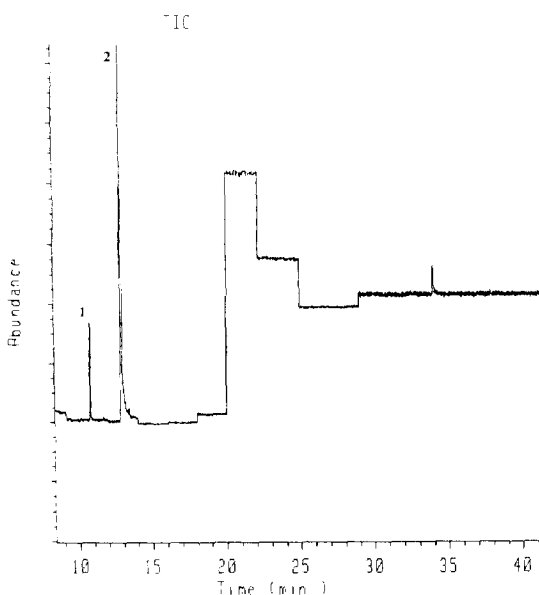


Fig. 2. Total ion chromatogram of organochlorine pesticides extracted from 1 l of coastal sea water using an SPE disk. Peak identities: 1= $\gamma$ -BHC; 2=heptachlor.

some variation in the recoveries for OCPs stored on disks in this manner for 14 days vs. 3 days, no serious degradation of analytes occurred during the two-week study. The use of disks as a form of storage device was considered feasible since generally, recoveries of most of the OCPs stored on membranous media were either equivalent or superior to those of OCPs stored in water, although these recoveries did not always differ significantly. The higher stability of some OCPs may be due to protection from hydrolysis, microbial degradation and absorption onto particulate matter when OCPs are adsorbed onto the disks. Possible rearrangement of DDT, heptachlor and endrin into their respective metabolic products might have occurred in water during the two-week study. This could have resulted in lower recoveries. However, no definite conclusion could be made from these observations especially since no heptachlor epoxide, the breakdown product of heptachlor, was detected (it is of course possible that the level of heptachlor epoxide was below the detection limit). The stabilizing ability of the  $\text{C}_{18}$  material has been observed by other researchers who stated that analytes bonded to a solid phase were generally more stable [19]. The use of SPE disks as

Table 3  
Recovery (%) of OCPs in water and on disks under different storage conditions

OCPs	Recovery (%) <sup>a</sup>							
	A1	A2	B1	B2	C1	C2	D1	D2
TCMX <sup>b</sup>	80.0	81.0	72.0	75.0	75.0	67.0	72.0	65.0
$\alpha$ -BHC	85.0	74.0	70.1	61.0	75.4	75.0	75.5	71.0
$\beta$ -BHC	85.1	67.1	59.1	50.1	79.0	70.0	78.0	61.0
$\gamma$ -BHC	69.0	61.0	70.1	51.0	69.0	65.0	70.7	56.4
$\delta$ -BHC	79.0	83.1	79.0	65.0	81.0	79.0	80.0	70.0
Heptachlor	86.7	71.2	81.4	70.0	92.0	75.4	82.0	75.7
Aldrin	73.8	48.0	65.0	40.0	78.9	69.0	70.0	60.0
Heptachlor epoxide	81.0	81.0	81.0	67.0	83.0	85.0	74.0	67.0
Endosulfan I	71.6	75.1	70.1	59.0	70.5	65.0	67.0	68.0
Dieldrin	91.0	82.0	75.0	59.0	92.0	80.0	88.0	65.0
<i>p,p'</i> -DDE	87.0	89.0	81.0	78.0	87.2	77.0	85.0	78.0
Endrin	80.0	71.0	69.0	76.0	82.1	70.0	72.0	61.8
Endosulfan II	72.0	81.0	72.3	70.0	76.0	70.0	75.0	70.0
<i>p,p'</i> -DDD	90.0	82.0	81.0	81.0	91.5	89.5	79.2	74.0
Endrin aldehyde	80.1	80.1	76.0	82.8	74.4	71.5	72.4	70.3
Endosulfan sulphate	87.2	79.2	70.6	73.0	87.4	87.8	75.0	60.3
<i>p,p'</i> -DDT	79.3	79.2	71.2	57.0	81.0	76.0	71.0	52.9
Methoxychlor	100	89.2	92.0	79.0	99.7	91.0	83.9	69.0
Dibutyl chlorendate <sup>b</sup>	83.9	82.0	82.0	61.0	85.0	83.2	80.9	77.0

<sup>a</sup> A=distilled water; B=tap water; C=unfiltered sea water; D=filtered sea water; A1=disks at room temperature and in darkness for 3 days; A2=water at room temperature and in darkness for 3 days; B1=disks at room temperature and in darkness for 14 days; B2=water at room temperature and in darkness for 14 days; C1=disks at 4°C and for 3 days; C2=water at 4°C and for 3 days; D1=disks at 4°C and for 14 days; D2=water at 4°C and for 14 days.

<sup>b</sup> Average of two determinations.

<sup>c</sup> Surrogate standards.

storage devices for OCPs thus has the potential of replacing conventional storage methodologies. The bulkiness associated with the storage and transportation of water samples can be drastically reduced if extraction disks were used instead. Thus, sampling time during field trips and sample preparation in laboratory can be greatly reduced by carrying out extractions on site and transporting the disks back to the laboratory for analysis. It was indicated above that for water samples containing high levels of particulate matter, the recoveries of OCPs were low. This does not mean that the use of SPE disks for field studies is not viable, however, since this problem is not critical when samples containing small amount of particulates are considered. Indeed, for samples heavily contaminated with particulates, the latter (particulates) should rightly be analysed separately from the water for adsorbed OCPs in order to determine a more meaningful pollution load for the samples as a whole.

### 3.3. MASE of OCPs in sediment

The optimum conditions for the MASE recoveries of OCPs from sediment could be obtained by varying the extracting solvents, extraction temperatures and duration of extraction. The average recoveries obtained with either dichloromethane (79.3%) or acetone–hexane (1:1, v/v) (78.5%) as extracting solvent were higher than that obtained by acetone–light petroleum (1:1, v/v) (70.4%). In this work, acetone–hexane (1:1, v/v) was selected because dichloromethane had to be replaced subsequently by another solvent in order to ensure compatibility with ECD. In the case of extraction temperature, there was a slight increase in recoveries from 77.8 to 78.1% when extraction temperatures were increased from 100 to 120°C. However, the increase was not significant at all. Generally, there is an increase in the average recoveries of OCPs from 65.2 to 72.3% when the duration was increased from 5 to 10 min. However,

comparable recoveries were obtained for extractions performed for 10 min, and for 20 min. No degradation of OCPs was observed throughout the experimental trials under the respective MASE conditions. Summarising, the optimum MASE conditions can be established with acetone–hexane (1:1, v/v) as the extracting solvent and with extractions performed at 100°C for 10 min.

A comparison between Soxhlet extraction and MASE technique for sediments spiked at 50 ng/g levels is presented in Table 4. When extracted by the Soxhlet method, recoveries in the range 61.3–88.7% with mean deviation 2.4–10.0% were obtained, while MASE gave comparable recoveries in the range 70.6–90.6% with mean deviations of 2.0–8.0%. MASE has several advantages over Soxhlet extraction, such as the MES-1000 allowed 12 samples to be extracted at a time with less than 30 min per sample compared to 12–24 h for Soxhlet extraction. In addition, it is cost-effective, environmental friendly and utilizes less organic solvents.

GC–ECD analysis of 5 genuine sediment samples obtained from the Straits of Johore using the op-

timum MASE conditions was subsequently carried out. However, no OCP was detected.

#### 4. Conclusions

SPE by membranous disks is well suited as a sample preparation procedure for “less dirty” natural waters for the extraction of OCPs. Large volumes of water samples can be extracted by disks which can also be used as storage devices. Samples need not be transported back to the laboratory in bulky water bottles. The stabilities of OCPs stored on disks have been studied and no serious analyte degradation was observed over a period of two weeks. We have also demonstrated that MASE as a sample preparation technique for sediments merits serious consideration. MASE recoveries were comparable to those that might be obtained by Soxhlet extraction. The advantages of the former technique are cost-effectiveness, and environmental friendliness in terms of organic solvent usage and wastage. Together, the novel sample preparation techniques discussed in this work make routine analysis of environmental samples much more convenient and efficient.

Table 4  
Comparison between Soxhlet extraction and MASE of marine sediments spiked with OCPs at 50 ng/g levels of each compound

OCPs	Recovery (%) [M.D.(%)] <sup>a</sup>	
	Soxhlet extraction	MASE
TCMX <sup>b</sup>	72.0 (4.5)	76.4 (3.0)
$\alpha$ -BHC	75.0 (8.0)	76.4 (4.9)
$\beta$ -BHC	79.0 (6.3)	82.0 (6.1)
$\gamma$ -BHC	85.0 (9.4)	82.2 (2.4)
$\delta$ -BHC	80.0 (10.0)	85.0 (4.0)
Heptachlor	62.1 (6.1)	75.5 (6.6)
Aldrin	88.1 (4.5)	88.0 (2.5)
Heptachlor epoxide	82.0 (3.7)	81.9 (3.0)
Endosulfan I	61.3 (4.0)	82.8 (4.2)
Dieldrin	62.3 (3.9)	83.3 (4.0)
<i>p,p'</i> -DDE	84.9 (5.1)	85.6 (5.0)
Endrin	79.0 (7.2)	90.0 (8.0)
Endosulfan II	88.7 (5.0)	93.1 (4.3)
<i>p,p'</i> -DDD	67.0 (2.4)	78.7 (3.7)
Endrin aldehyde	82.0 (7.4)	90.6 (3.0)
Endosulfan sulphate	72.7 (5.0)	89.3 (4.1)
<i>p,p'</i> -DDT	79.2 (5.0)	89.3 (4.1)
Methoxychlor	82.0 (6.1)	90.6 (2.0)
Dibutyl chlorendate <sup>b</sup>	72.7 (4.0)	70.6 (3.0)

<sup>a</sup> Mean deviation for  $n=4$ .

<sup>b</sup> Surrogate standards.

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