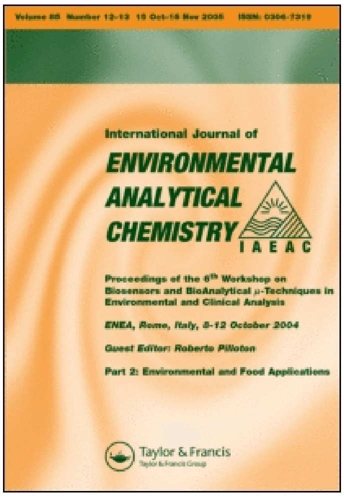

International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

Microextraction of Chlorinated Benzenes and Selected Organochlorines from Environmental Water

C. Samson[a]; V. N. Mallet[a]; J. Doull[b]; G. Brun[b]
[a] Chemistry and Biochemistry Department, Universite de Moncton, Moncton, N.B., Canada [b] Environmental Quality Laboratory, Environmental Sciences Centre, Environment Canada, Universite de Moncton, Moncton, N.B., Canada

To cite this Article Samson, C. , Mallet, V. N. , Doull, J. and Brun, G.(1996) 'Microextraction of Chlorinated Benzenes and Selected Organochlorines from Environmental Water', International Journal of Environmental Analytical Chemistry, 64: 2, 111 — 121

To link to this Article: DOI: 10.1080/03067319608028340

URL: http://dx.doi.org/10.1080/03067319608028340

Intern. J. Environ. Anal. Chem., Vol. 64, pp. 111–121
Reprints available directly from the publisher
Photocopying permitted by license only

MICROEXTRACTION OF CHLORINATED BENZENES AND SELECTED ORGANOCHLORINES FROM ENVIRONMENTAL WATER

C. SAMSON[1], V. N. MALLET[1]*, J. DOULL[2], and G. BRUN[2]

[1]*Chemistry and Biochemistry Department, Universite de Moncton, Moncton, N.B., Canada, E1A 3E9 and* [2]*Environmental Quality Laboratory, Environmental Sciences Centre, Environment Canada, Universite de Moncton, Moncton, N.B., Canada, E1A 3E9*

(Received, 9 November 1995; in final form, 12 February 1996)

This study describes the extraction of CBs and OCs from water by conventional means using only a few millilitres of solvent. The parameters investigated were volume of n-hexane versus time of extraction. The results clearly show several alternatives to the analyst, i.e., smaller volume-longer time or larger volume-shorter time. Typically, good recoveries could be achieved with as little as 5 mL of n-hexane in a single step 10-minute extraction of a one-litre water sample, but more consistent results were obtained with environmental water with two successive extractions using 2 mL of solvent and a few drops of acetone to reduce emulsions. For instance, average recoveries of 83.29% and 87.75% were obtained for CBs and OCs, respectively, in a multi-residue analysis of environmental water comprising 22 components at the 0.01–0.25 ppb level. Thus, the microextraction approach looks very promising as an alternative to the lengthy and often more costly traditional liquid-liquid extraction approach for the analysis of CBs and OCs from environmental water. In fact recoveries are better for volatile analytes such as low molecular weight chlorobenzenes because the evaporation step is eliminated. Also, precision of the data for individual congeners is better.

KEY WORDS: Chlorinated benzenes, organochlorines, solvent extraction, water.

INTRODUCTION

Organochlorines (OCs) and chlorinated benzenes (CBs) are still commonly used chemicals worldwide[1] and as a consequence they eventually find their way into the ecosystem. In the Maritime Provinces of Canada small amounts of chlorinated benzenes were detected in Herring Gull and double-crested Cormorant, as early as 1980[2] and there is evidence that their presence in the environment is common. That is also the case for organochlorines which are even more widespread[3]. Thus, the need to monitor these chemicals in various substrates, including water, and to improve on existing methods for more complete and reliable data, is on-going. The traditional liquid-liquid extraction approach for water is lengthy, cumbersome, time consuming and requires large amounts of costly solvents. Also, during evaporation of these solvents, loss of chlorinated benzenes may be important[4]; this in turn may affect recoveries and

---

* To whom correspondence should be addressed.

reproducibility of the data. Mathews[5] showed that stringent controls on the evaporative conditions could yield 90 and 100% recoveries for these chemicals but this adds to the complexity of the process.

There are presently many attempts being made to offer an alternative to the traditional liquid-liquid extraction approach for contaminants in water through the technique of solid phase extraction. For instance, Vidal *et al.*[6], have extracted 16 organophosphorous pesticides (OPs) from water using XAD resins and have compared their results with those obtained with method 608 of the Environmental Protection Agency (EPA). Others[7] use special extraction disks to remove pesticides from water. These techniques are not without drawbacks, one of which is cost, and they cannot be considered universal.

Liquid-liquid microextraction of water offers an alternative to both the traditional liquid-liquid extraction approach and solid phase extractions. Emphasis can be put on reducing the volume of solvent and sample and on miniaturizing the equipment to handle smaller volumes. Some attempts to achieve both were made in the past[8] but with little success because of poor recoveries and lack of reproducibility due in part to less than optimal experimental conditions.

Our work has been focused on the choice of solvent and on their performance. From a previous study[9] with organophosphorous pesticides it was learned that n-hexane was a solvent of choice because its lack of solubility in water makes it easier for phase separation and thus, to recover a small aliquot (1 mL) of extraction solvent from a larger (1 L) sample of water. The method yielded greater than 90% recoveries for some organophosphorous pesticides at the 10 ug/L level. In a subsequent study[10] with polychlorinated biphenyls, DDT and some related compounds, the method was refined and adapted to a 500 mL water sample which was extracted twice for five min. with 1 mL portions of n-hexane. This allowed the determination of PCBs and DDTs (18 components) with high recoveries at the 40 ng/L level.

In this study, it was intended to evaluate the potential of the microextraction approach (small volume of solvent) to extract OCs and CBs from water. Since a small volume requires no evaporation it was expected that loss of volatile components would be curtailed. Another objective was to study in more detail the relationship between extraction time and volume of solvent and the possible impact of additives to improve recoveries such that the most appropriate conditions for optimum recoveries could be determined. A third objective was to compare the results from selected experimental conditions with those obtained by the more traditional liquid-liquid extraction approach. The ultimate goal of these experiments was to reduce the total amount of solvents used in extraction procedures and to considerably shorten analysis time for overall cost reduction, without too much loss in overall recoveries.

EXPERIMENTAL

Chemicals

Analytical standards of chlorinated benzenes and organochlorines were obtained from Supelco. The chlorinated benzenes were: 1,3-dichlorobenzene (DCB), 1,4-dichlorobenzene (DCB), 1,2-dichlorobenzene (DCB), 1,3,5-trichlorobenzene (TCB), 1,2,4-trichlorobenzene (TCB), 1,2,3-trichlorobenzene (TCB), 1,2,3,5-tetrachlorobenzene (TTCB), 1,2,4,5-tetrachlorobenzene (TTCB), pentachlobenzene (PTCB), hexachlorobenzene (HCB). The organochlorines were: a-BHC, Lindane, Heptachlor, Aldrin, Heptachlor epoxide, g-Chlordane, a-Endosulfan, a-Chlordane, p,p'-DDE, Endrin, and b-Endosulfan. A mixed stock solution (A93) containing each chemical of concentration between 0.5–12.5 ng/uL was prepared in isopropanol (Burdick and Jackson). Water samples were fortified with an aliquot of the stock solution to obtain the desired sample concentration. Working standard solutions (0.01–0.25 ng/uL) used for reproducibility and other quantitative studies were prepared by dilution of the stock solution in the solvent of extraction, usually n-hexane.

All solvents were pesticide grade or equivalent: n-hexane, acetone and methanol (Burdick and Jackson) and acetonitrile (Caledon). Unless otherwise stated sodium chloride consisted of Sifto pickling salt available commercially.

The water used for recovery studies was doubly distilled and deionized (Millipore-RO). The water used to represent an environmental matrix came from an open spring for which quality data are available (Trites St., Riverview, N.B. Canada) through Environment Canada in Moncton, N.B. Canada, E1A 3E9.

Instrumentation

The gas chromatography was a Varian Vista 6000 equipped with a dual fused-silica capillary column system with two Ni63 electron capture detectors. One column (J & W Scientific) contained DB-5 (0.25 um) and was 30 m × 0.25 mm (i.d.); the other (Supelco) contained DB-608 (0.5 um) and was 30 m × 0.32 mm (i.d.). A 1 m × 0.53 mm (i.d.) pre-column of deactivated silica was used. A T-shaped glass joint at the junction of the pre-column and the dual column system assured the correct splitting of the sample. Instrument settings were: initial temp., 50°C, increased to 90°C at 25°C/min., increased to 210°C at 4°C/min., increased to 240°C at 15°C/min., increased to 250°C at 10°C/min. and kept there for 10 min. The injection port was set at 230°C and the detectors at 350°C. The mobile phase was high purity Helium with a linear velocity of 1 mm/min. The make-up gas was nitrogen at a linear velocity of 75 mL/min.

Method

This is the standard method developed in this study. Experimental conditions that differ are indicated in the Table legends.

(a) Fortification of water samples. — Typically, 100 uL of the stock solution (A93) was added to 40 mL of purified or environmental water in a beaker and the solution was stirred, with a magnetic stirrer, for three min. at sufficient speed to produce a vortex to assure proper contact of the phases. Any other addition (such as salt) was made at this point with further mixing. The solution was then transferred to a l-litre volumetric flask to which water was added to the mark.

(b) Extraction of water. — Typically, 10 g of NaCl followed by 2 mL of n-hexane was added to a 1-litre volumetric flask containing one litre of purified or environmental water and the solution was stirred for 10 min. (see above). After equilibration (about 3 min.) the extract was recovered with a Pasteur pipette. If necessary, the extraction was repeated with another aliquot of n-hexane. The extract (or extracts) was transferred to a 5-mL volumetric flask and diluted to the mark with n-hexane. The addition of a few drops of acetone during the equilibration step accelerates the separation of the phases and helps break down emulsions. The hexane extract obtained from this method was not dried before injection and no adverse effect on the clean-up or the chromatography was ever observed.

(c) Quantitation. — A 2-uL aliquot was injected splitless into the chromatograph via the automated injection system. Quantization was achieved by comparing peak areas with those of an external standard of comparable concentration.

(d) Method used for comparison (see ref. 11).

RESULTS AND DISCUSSION

Previous experiments[10] established that n-hexane is a good solvent for the extraction of PCBs from water. The method used was similar to that developed for OPs in a previous study[9], that is, a 500-mL water sample was extracted with successive portions of 2 mL of n-hexane. In practice, however, it is more common to use a 1 L water sample. Also, while investigating the possibility of using n-hexane for OCs and CBs, it was felt that a thorough investigation of the relationship between volume of solvent and time of extraction was necessary. In order to achieve that it was felt that a good quality control program was justified.

Quality control

Initially the gas chromatograph was optimized for qualitative and quantitative performance within our quality control program. Reproducibility of retention times on both columns was tested by injecting six replicates of the standard solutions at the concentrations used for quantification. At a concentration range of 0.01–0.25 ng/uL, the values of the coefficients of variation for retention times were typically between 0.01% and 0.05% indicating excellent reproducibility. A typical chromatogram showing all the components of standard A93 is shown in Figure 1. For comparison purposes Figure 1 also shows the chromatogram from an environmental water extract. As can be observed the chromatogram does not contain too many extraneous peaks, which was to be expected since the water source is considered to be clean of OCs and CBs.

With the same standard injected six times coefficients of variation for peak areas varied between 1.68% and 6.98%, with a few exceptions. Values for double, triple and four times the concentration of the standards were all within acceptable limits of variation. The data suggested that a reasonable reproducibility could be expected under the current experimental conditions. A typical linearity curve with aldrin shown in Figure 2 is linear between 0.005 and 0.04 ng/uL, which is within the expected limits of concentration of the extracted samples.

Recovery experiments

Basically the experimental conducted in this study consisted in the extraction of water samples fortified with OCs and CBs under various experimental conditions. In the initial experiments the relationship between small volumes (1–10 mL) of n-hexane as a function of time (1–30 min.) was studied in order to determine the minimum amounts of solvent and time required to achieve acceptable recoveries. The volume of the sample remained the same at one litre and the concentrations of the components of the sample (0.01–0.25 ug/uL) were chosen to represent environmental values. However, this type

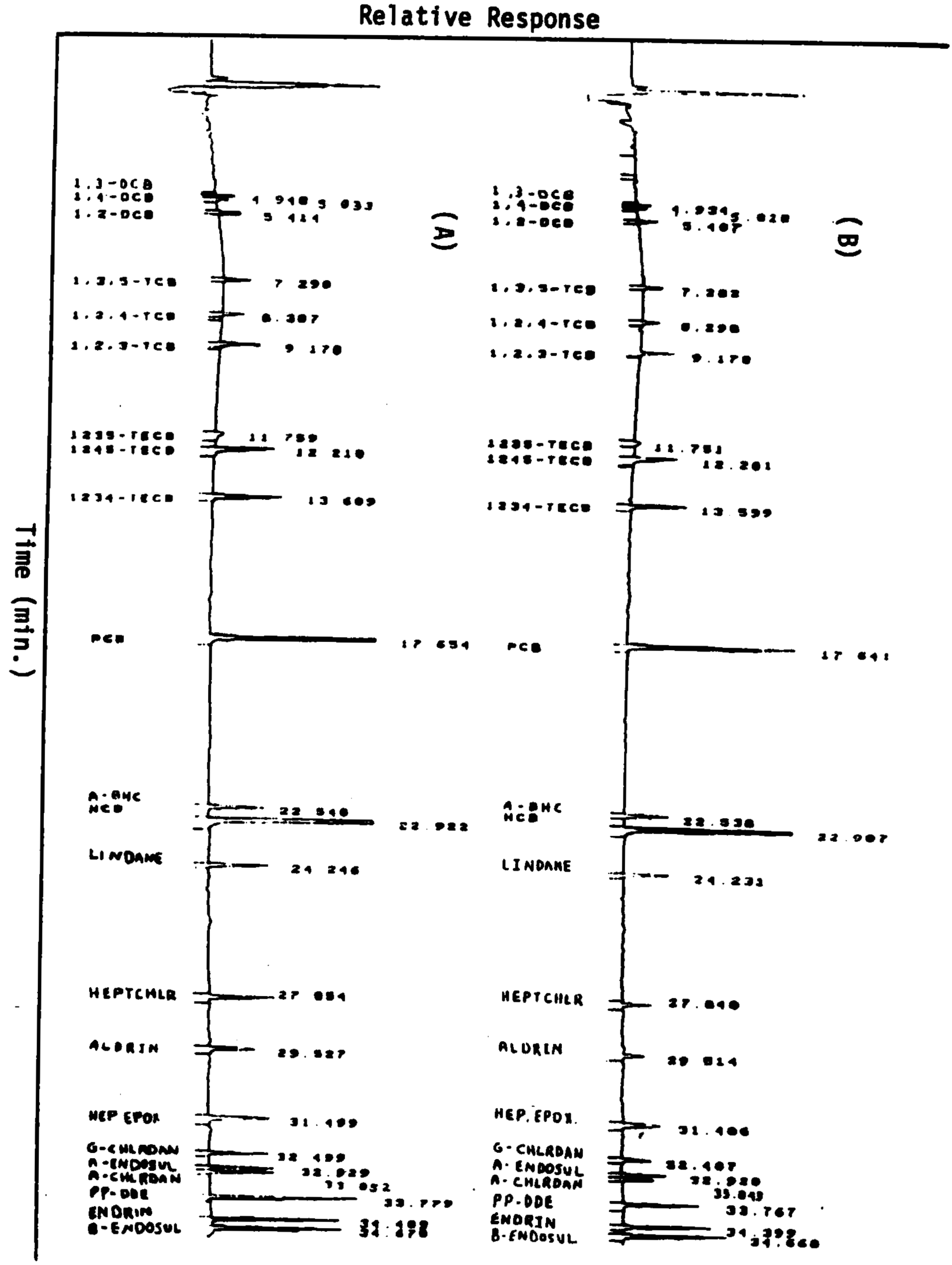

Figure 1 A. Chromatogram of Analytical Standard A93 (see Table 1 for concentrations; B. Chromatogram of Environmental Water Extract (see Table 6, method 1).

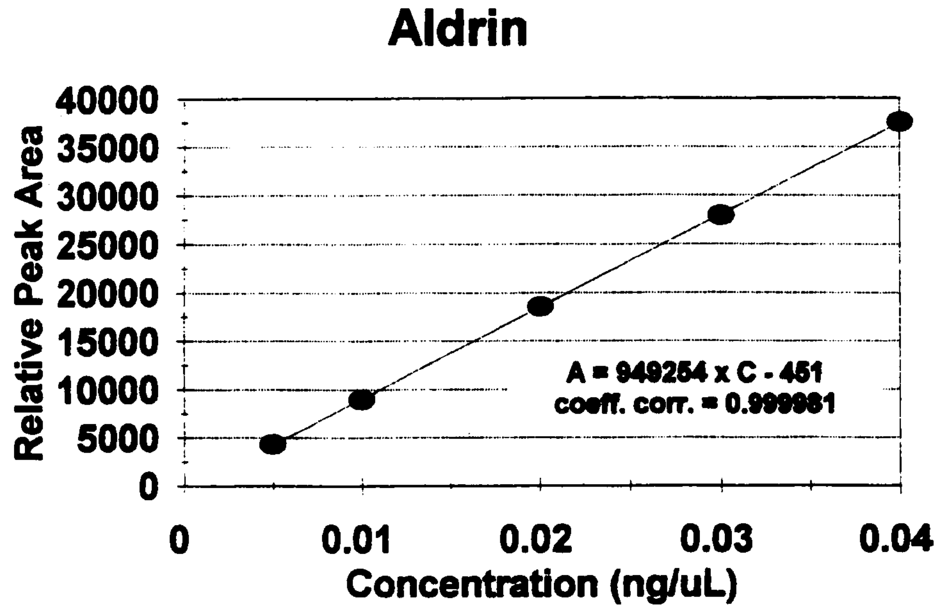

Figure 2 Typical Calibration Curve: Aldrin.

of experiment generates a lot of data. In order to reduce the total number of experiments and data and still obtain reliable indicators of performance the first series of experiments were conducted with duplicate samples which were extracted and chromatographed on the dual column system. Thus, the data from each injection represented the average response from the two columns and the registered data for each set of conditions was the average from duplicate extractions. Typical data are shown in Table 1. More replicate analysis could have been carried out such that comparison of recoveries between various chemicals would have been possible. But that was not the intent. Instead, the objective was to determine in a minumum number of experiments the most promising conditions for optimum recoveries. Comparing group averages from duplicate analysis, as shown in Table 1, provides this opportunity. It is not without fault, however, since weak recoveries for whatever reasons will affect the group average substancially. This is why the data can only be taken as indicative at this point.

Typical "group average" data from duplicate analyses as a function of time of extraction and volume of solvent are presented in Table 2. They definitely show that extraction with one mL of n-hexane does not give acceptable recoveries (greater than 80%) even after 30 min. With two mL of solvent there are signs of increased recoveries particularly with 10 min. or more, of extraction time. This becomes evident with four or more mL of solvent. Thus, as expected, both an increase in extraction time and volume of solvent play an important role in improving the recoveries. The data also show that excellent recoveries could be expected for both groups of chemicals using 10 mL of solvent for 10 min.

In the present study, however, the main objective was to use a minimum amount of solvent and a minimum amount of time. Also, it was intended to compare our method with that used by Environment Canada (Moncton, N.B.), which requires a final volume of 5 mL. Thus, in order to avoid having to evaporate the solvent after extraction it

Table 1 Typical recovery studies for CBs and OCs in water.

Chemical	*Conc. (ng/uL)*	*% Recovery*		*Average (x)*
		(I)	(II)	
1,3-DCB	0.15	77.75	78.07	77.91
1,2-DCB	0.25	75.46	72.63	74.05
1,3,5-TCB	0.03	77.15	93.61	85.38
1,2,4-TCB	0.03	84.16	84.87	84.52
1,2,3-TCB	0.03	83.37	85.94	84.66
1,2,3,5-TTCB	0.03	86.91	90.22	88.57
1,2,4,5-TTCB	0.03	85.52	84.66	85.09
1,2,3,4-TTCB	0.03	88.42	92.78	90.60
PTCB	0.06	87.09	85.95	86.52
HCB	0.06	80.87	76.50	78.69
Group average		**82.67**	**84.52**	**83.60**
a-BHC	0.01	82.17	86.68	84.43
Lindane	0.01	75.83	77.54	76.69
Heptachlor	0.01	76.25	68.59	72.42
Aldrin	0.01	66.68	59.42	63.05
Hepta. epox.	0.01	30.09	28.91	29.50
g-Chlordane	0.01	78.82	75.24	77.03
a-Endosulfan	0.01	89.82	86.84	88.33
a-Chlordane	0.01	65.71	62.35	64.03
p,p'-DDE	0.01	81.39	76.12	78.76
Endrin	0.02	91.76	93.16	92.46
b-Endosulfan	0.02	89.10	90.87	89.99
Group average		**75.24**	**73.25**	**74.24**

Legend: extract 1 L water with 2 mL n-hexane for 30 min.

Table 2 Summary of average % recoveries for CBs and OCs in water volume of n-hexane (ml).

Group of chemicals	*Time (min)*	*1*	*2*	*4*	*6*	*10*
CBs	1	22.31	25.58	44.24	50.67	55.07
OCs	1	9.41	39.41	35.88	35.54	51.77
CBs	5	44.82	52.72	69.77	80.59	78.98
OCs	5	30.58	39.41	62.48	55.35	79.11
CBs	10	48.29	74.47	**85.14**	**86.46**	**89.19**
OCs	10	33.26	60.09	72.48	62.81	**87.47**
CBs	20	54.52	82.34	**89.50**	**90.66**	**85.56**
OCs	20	39.42	71.89	83.34	70.93	**85.27**
CBs	30	67.33	83.60	**90.16**	**87.92**	**86.93**
OCs	30	54.37	74.24	**85.20**	73.34	73.09

Legend: extract 1 L water with n-hexane; see Table 1 for concentrations.

was decided to use a maximum of 4 mL and make up the final volume to 5 mL. In fact Table 2 shows that both families of chemicals show promising recoveries with less than 5 mL of solvent and an extraction time of 10 min. This does not mean that other conditions of time and volume could not be adapted to suit particular needs in different laboratories.

Thus, using either 2 or 4 mL of n-hexane and an extraction time of 10 min., several conditions were tested, including: the addition of salts, namely sodium chloride or sodium sulfate; the addition of polar solvents, namely acetonitrile, methanol or acetone; various pH values (4.0, 5.6 and 9.0); another solvent, toluene and even the size and shape of the container. The latter two sets of conditions did not improve recoveries for one-litre water samples. The addition of salt or polar solvent, however, will affect the dielectric constant of water and therefore may have an impact on the extractability of chemicals from water. As shown in Table 3, the addition of 10 g/L (1%) of NaCl improves the average recoveries noticeably (Note: data in Table 3 do not match with the data in Table 2 since they represent a different set of experiments and different instrumental calibration over the span of one year). Larger amounts (100 g/L) do not seem to contribute significantly. The addition of acetonitrile or acetone seems to improve recoveries, particularly for OCs. In fact the addition of only a few drops of acetone (in presence of NaCl) improves the recoveries quite substantially. In our opinion a little bit of acetone helps break down emulsions which makes it easier to recover most of the solvent of extraction and improves reproducibility. The addition of methanol seems to have very little effect.

Successive extractions by themselves seem to be only slightly beneficial as shown in Table 4. Addition of NaCl alone does not seem to have much impact but the addition of acetone or acetonitrile seems positive. A combination of both (for example acetone and NaCl) seems to have a larger synergist effect on the recoveries. The addition of only a few drop of acetone, however, seems to have a similar effect and this is the preferred

Table 3 Effect of additives on the recovery (%) of CBs and OCs from water.

	CBs	*(n = 6) Average*		*OCs*
Sample	*%R*	*C.V.*	*%R*	*C.V.*
Reference*	69.13	6.50%	55.75	11.73%
Replicate	69.75	7.64%	58.95	9.64%
+10 g NaCl	79.49	6.97%	73.78	8.68%
Replicate	70.39	8.80%	69.72	7.32%
+100 g NaCl	71.60	3.54%	69.85	5.24%
+10 mL ACN	79.76	5.30%	74.04	7.59%
Replicate	68.03	5.51%	59.35	8.98%
Replicate +10 g NaCl	70.29	6.00%	69.01	5.11%
+10 mL MeOH	72.48	3.56%	61.62	10.26%
+10 mL ACET	74.90	5.58%	67.82	7.89%
Replicate	72.04	7.92%	62.10	9.32%
Replicate +10 g NaCl	81.33	8.09%	75.41	9.73%
+5 Drops ACET +10 g NaCl	82.33	5.99%	80.33	7.98%

Legend: 1-L water; 4 mL n-hexane; 10 min. extraction; final vol. 5-mL.

* Reference refers to basic sample of water to which salt or solvent or both were subsequently added.

Table 4 Effect of successive extractions on the % recoveries of CBs and OCs from water.

	OCs	*(n = 6) Average*	*CBs*	
Sample	*%R*	*C.V.*	*%R*	*C.V.*
Reference*	74.87	5.06%	61.73	8.31%
Replicate	75.26	6.59%	69.91	8.22%
+10 g NaCl	75.86	9.06%	73.73	9.86%
+10 mL ACN	81.28	3.77%	70.22	7.23%
+10 mL ACET	82.05	4.73%	71.58	6.39%
+10 mL ACN + 10 g NaCl	85.25	7.54%	83.63	10.77%
+10 mL ACET + 10 g NaCl	83.95	8.03%	80.68	7.86%
+ 5 drops ACET + 10 g NaCl	88.30	3.65%	91.94	8.21%
Environment Canada	89.74	6.21%	98.38	5.93%

Legend: Basic conditions: 1 L of water; 2 × 2 mL of n-hexane; successive extractions of 10 min; final volume 5 mL.

Environment Canada method: successive 2-min. extractions with 2 × 100 mL of n-hexane, final volume 5 mL.

approach. These latter results are very comparable to those obtained using the conventional approach.

Finally, some of the most promising conditions were tested on environmental water. The results, in terms of group averages for % recoveries and presented in Table 5, show very good recoveries for all of the conditions tested. In fact the data obtained by the microextraction approach are quite comparable to those obtained with the traditional approach. In addition the average coefficients of variation are reasonably low which is indicative of good reproducibility. It should be mentioned that turbidity caused by suspended matter in water could affect the extraction process with some types of environmental samples. However, this parameter was not studied in this work.

The detailed recovery data for three methods are presented in Table 6. Comparing method 1 (a few drops of acetone) and method 2 (10 mL of acetone) reveals very similar results indicating that the presence of acetone more or less ensures complete separation of the phases by reducing emulsions rather than take part in the extraction process. The addition of acetone is important since without it the average % recoveries may be 10% less. Comparing method 3 with method 1 reveals that recoveries of the more volatile CBs are less with the conventional method probably because they were lost during the evaporation process, as anticipated. For the other chemicals studied method 3 yields close to 100% recoveries in several cases and very comparable values to method 1 in other cases. In terms of reproducibility as indicated by coefficients of variation both method 1 and method 3 appear comparable. Both methods yield low recoveries for heptachlor and aldrin.

Table 5 Recovery of CBs and OCs from environmental water (1 L).

Comparison between different sets of conditions				
Condition	*CBs*		*OCs*	
	Average			
	%R	C.V.	%R	C.V.
1	79.01	3.96%	74.77	6.10%
2	83.30	3.99%	83.73	7.45%
3	84.77	2.77%	84.98	4.76%
4	79.68	6.16%	88.68	5.36%
5	83.29	5.97%	87.75	5.70%
6	82.38	4.86%	83.54	5.17%

Legend: 1) 10 mL acetone + 10 g NaCl, extrac. 4 mL of hexane for 10 min.; 2) 10 mL acetone + 10 g NaCl, 2 × 2 mL of hexane for 10 min.; 3) 10 mL acetonitrile + 10 g NaCl, 2 × 2 mL of hexane for 10 min.; 4) Environment Canada Method; 5) five drops of acetone + 10 g NaCl, ext. 2 × 2 mL hexane for 10 min.; 6) five drops of acetone + 10 g NaCl, ext. 4 mL hexane for 10 min.

Table 6 Detailed recovery data from most promising methods.

	Method 1		*Method 2*		*Method 3*	
Chemical	*% Recovery*	*C.V.*	*% Recovery*	*C.V.*	*% Recovery*	*C.V.*
1,3-DCB	77.76	4.31	78.18	2.23	68.96	9.12
1,4-DCB	76.18	5.05	74.82	4.60	69.30	9.06
1,2-DCB	80.23	4.29	79.75	2.06	63.79	9.75
1,3,5-TCB	81.24	6.07	81.66	2.44	79.59	5.54
1,2,4-TCB	85.39	5.50	88.09	2.45	84.09	7.27
1,2,3-TCB	87.92	3.76	89.03	1.89	85.95	6.16
1,2,3,5-TTCB	86.00	7.00	85.28	3.23	86.18	2.68
1,2,4,5-TTCB	86.51	5.61	89.44	9.15	93.25	8.52
1,2,3,4-TTCB	89.12	5.35	90.37	2.36	93.92	3.13
PTCB	87.01	9.02	83.76	4.42	82.21	3.56
HCB	78.86	9.72	75.96	9.12	69.21	2.92
Group average	83.29	5.97	83.30	4.00	79.68	6.16
a-BHC	99.24	3.07	95.55	2.32	102.04	2.76
Lindane	95.68	4.34	91.89	3.72	104.02	1.83
Heptachlor	70.37	10.79	65.43	10.96	71.03	20.00
Aldrin	67.48	10.09	57.95	10.77	62.74	3.16
Hepta. epox.	99.47	5.01	89.73	5.78	119.29	6.54
g-Chlordane	84.42	6.58	74.98	11.22	61.66	2.85
a-Endosulfan	97.43	4.75	95.56	7.46	107.91	4.03
a-Chlordane	83.60	5.96	81.52	7.19	81.20	3.88
p,p'-DDE	83.09	5.93	80.28	9.86	74.66	4.38
Endrin	90.02	3.46	92.18	8.28	90.75	5.97
b-Endosulfan	94.46	2.76	96.02	4.35	100.21	3.55
Group average	87.75	5.70	83.74	7.45	88.68	5.36

Method 1: one litre of environmental water extracted with 2 × 2 mL of n-hexane for 2 × 10 min. + 10 g of NaCl + 5 drops of acetone.

Method 2: one litre of environmental water extracted with 2 × 2 mL of n-hexane for 2 × 10 min. + 10 g of NaCl + 10 mL of acetone.

Method 3: one litre of environmental water extracted with 2 × 100 mL of n-hexane for 2 × 2 min.

CONCLUSION

In terms of the set objectives, this study has proven to be very revealing. The potential of using a smaller volume of organic solvent with respect to time of extraction to recover OCs and CBs from environmental water, has been demonstrated for the first time. In fact the study clearly shows the ability of this approach to yield high recoveries when using as little as 4 mL of solvent in a single step 10-minute extraction. However, less solvent may be used with a prolonged extraction time, or more solvent may be used with a shortened extraction time, to yield approximately the same results. Thus, there are several choices to the analyst depending upon which parameter, solvent or time, is most critical to the operation.

The implications of these results are important; less solvent used in the extraction process means less or no solvent to evaporate. In this case the evaporation step was eliminated altogether. This has proven to be important for volatile compounds such as the low molecular weight chlorobenzenes which have a tendency, as shown in this study, to yield lower recoveries when extracted with a large volume of organic solvent which must subsequently be evaporated to reduce volume of the final extract for analysis. Less solvent used also implies less solvent to recycle which in both instances relate directly to the cost per analysis. Also, precision of the data for individual congeners is improved because of less manipulation of the sample. The study also supports the claim made in previous studies[9,10], that sodium chloride and a little bit of acetone are important additives to improve the recoveries. Overall, the study shows that the microextraction approach is a viable alternative to the conventional liquid-liquid extraction approach using large amounts of solvent for the extraction of CBs and OCs from water.

Acknowledgement

The authors are indebted to the National Science and Engineering Research Council of Canada and Université de Moncton for financial support. Thanks are also due to Environment Canada for use of their facilities.

References

1. Y.-F. Li and E. C. Vordner, *Sci. Total Environ.*, **160/161**, 201-232 (1995).
2. R. A. F. Matheson, E. A. Hamilton, A. Trites and D. Whitehead, *Chlorinated benzenes in Herring gull and double-crested Cormorant eggs from three locations in the Maritime Provinces* (Surveillance Report EPS-5-AR-80-1, Environment Canada, Environmental Protection Service, Halifax, Nova Scotia, Canada, March 1980).
3. S. Tanabe, H. Iwata and R. Tatsukawa, *Sci. Total Environ.*, **154**, 163-178 (1995).
4. M. D. Erickson, M. T. Giguere and D. A. Whitaker, *Anal. Lett.*, **14(A11)**, 841-857 (1981).
5. E. W. Mathews, *Anal. Chem.*, **57**, 1494-1496 (1985).
6. L. H. Vidal, W. R. Trevelin, M. D. Landgraf and M. O. O. Rezende, *Intern. J. Environ. Anal. Chem.*, **56**, 23-31 (1994).
7. S. Bentsson, T. Berglof and S. Granat, *Pest. Sc.*, **41**, 55-60 (1994).
8. D. A. J. Murray, *J. Chromatogr.*, **177**, 135-140 (1979).
9. D. Bourgeois, J. Gaudet, P. Deveau and V. N. Mallet, *Bull. Environ. Contamin. Toxicol.*, **50**, 433-440 (1993).
10. D. Bourgeois, P. Deveau and V. N. Mallet, *Intern. J. Environ. Anal. Chem.*, **59**, 15-24 (1995).
11. Methods Manual of Environment Canada, Environment Canada, Moncton, N.B., Canada, E1A 3E9 (1990).