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Design and Testing of an Aqueous Phase Liquid-liquid Extractor (APLE) for the Determination of Organochlorine Contaminants

R.C. McCrea and J.D. Fischer

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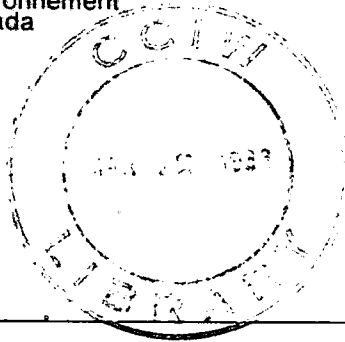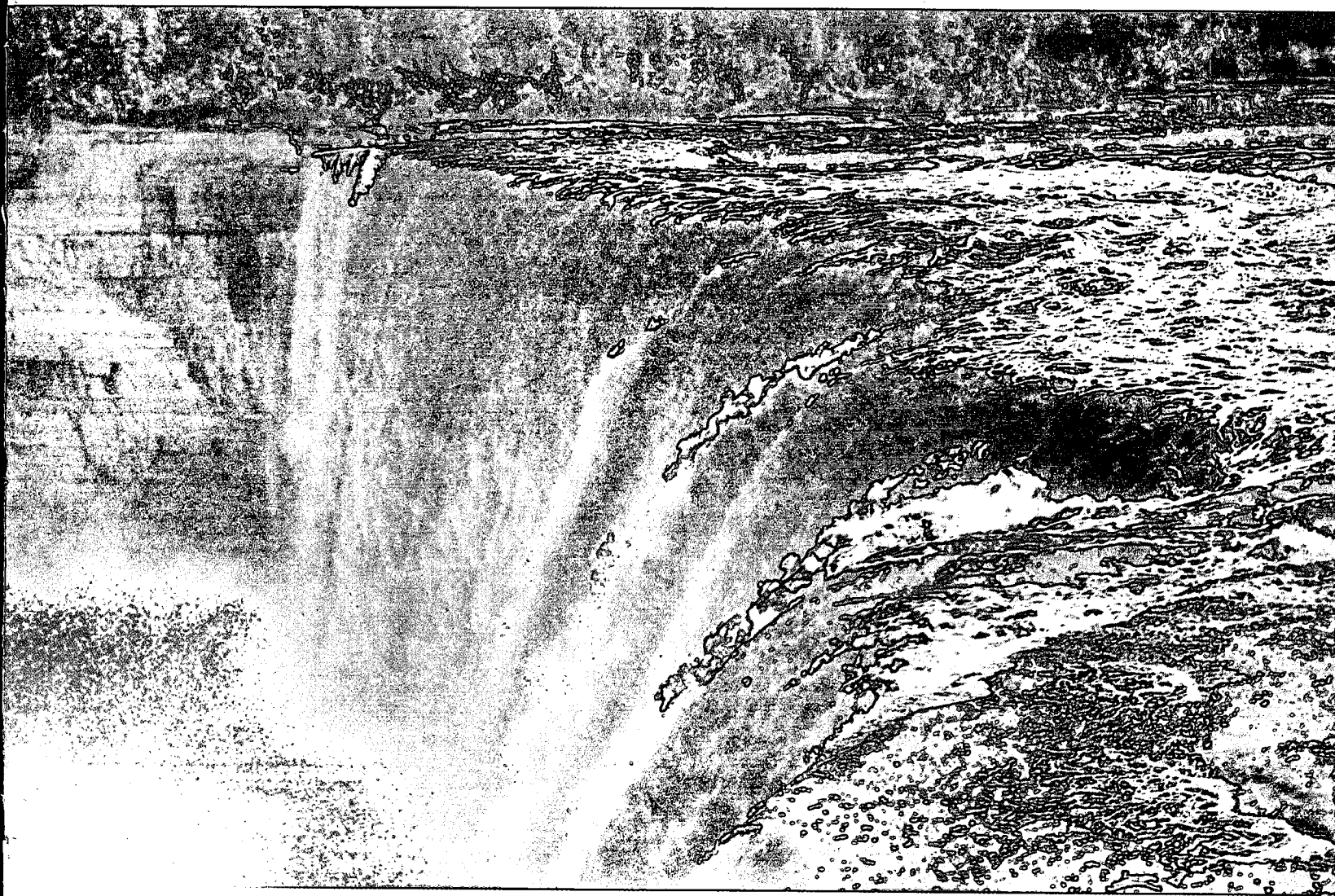
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Design and Testing of an Aqueous Phase Liquid-liquid Extractor (APLE) for the Determination of Organochlorine Contaminants

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Abstract

A field unit for the extraction of 200-L water samples was devised to provide a means of determining low-level organochlorine contaminants. This aqueous phase liquid-liquid extractor recirculates dichloromethane (d = 1.326) and is capable of concentrating and isolating contaminants in less than 2 h. Recovery tests based on two trials simulating environmental concentrations of 0.05, 0.25 and 1.0 ppt are presented for the following contaminants: HCB; α-BHC; γ-BHC; heptachlor; aldrin; heptachlor epoxide; γ-chlordane; α-chlordane; α-endosulfan; p,p'-DDE; dieldrin; endrin; o,p'-DDT; p,p'-TDE; p,p'-DDT; β-endosulfan; mirex; and p,p'-methoxychlor. Over the range of concentrations studied, the mean percent recovery was found to vary from 70% to 123%, with an overall mean recovery of 92%.

Résumé

On a conçu un appareil dans lequel il est possible d'extraire, sur le terrain, des échantillons de 200 L d'eau pour y déterminer de faibles teneurs en contaminants organo-chlorés. L'extracteur liquide-liquide pour phase aqueuse permet de recycler le solvant d'extraction, le dichloro-méthane (d = 1.326), et de concentrer et d'isoler les con-taminants en moins de 2 h. On donne le rendement d'extraction de deux essais dans des conditions simulant des concentrations environnementales de 0.05, 0.25 et 1.0 ppt des contaminants suivants : HCB; α-BHC; γ-BHC; heptachlore; aldrine; heptachlore-époxyde; γ-chlordane; α-chlordane; α-endosulfan; p,p'-DDE; dieldrine; endrine; o,p'-DDT; p,p'-TDE; p,p'-DDT; β-endosulfan; mirex; et p,p'-méthoxychlore. Dans la plage de concentrations étudiées, le rendement moyen variait de 70 % à 123 %, la moyenne globale étant de 92 %.

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INTRODUCTION

Various extraction systems based on resins (Osterroht, 1974), foam plugs (DeLappe et al., 1978) and liquid-liquid extraction (Wu and Suffet, 1977) have been designed for the investigation of trace organics in natural waters. Of these techniques, liquid-liquid extraction (LLE) is generally believed to be the best method for the isolation and concentration of organic contaminants from aqueous solutions (Wu, 1975). Several LLE systems are complicated and are employed under laboratory conditions. In contrast, a large-volume Aqueous Phase Liquid-liquid Extractor (APLE), designed by the Inland Waters Directorate, has a simple, rugged construction, and is suitable for field use.

The APLE is used in conjunction with a Westfalia continuous flow centrifuge and was designed to extract contaminants from clarified water, hereinafter referred to as the aqueous phase. This combined sampling system was devised to reduce the effective detection limits of trace organochlorine contaminants by two orders of magnitude below limits for traditional 1- to 2-L samples while providing samples of suspended sediment for subsequent analyses. Thomas and McMillan (1978) have shown that at a flow rate of 6 L/min, the Westfalia centrifuge is 90% to 95% effective in the recovery of suspended sediment from water in which 70% of the total seston is finer than 0.2 μm.

This report details the design and construction of the APLE system, as well as the recovery of 18 organochlorine contaminants based on replicate spikes at three concentrations.

CONSTRUCTION

The APLE system consists of three basic parts: a circulating pump, a spray bar and 223-L extraction drum (Fig. 1). All wetted parts are made of stainless steel, brass, ceramic or Teflon to prevent sample contamination.

A sample inlet consisting of a 1-in. National Pipe Thread (NPT) stainless steel coupling was welded at the top of the extraction drum and reduced with a fitting to accommodate a 0.75-in. NPT plug. A 0.25-in. NPT air vent was then welded adjacent to the sample inlet. Two metal straps were used to secure the extraction drum to a barrel cart, which had wheels mounted such that the cradle had an inclination of 5° from the horizontal position. A 0.75-in. drain tube, 5 cm in length, was mounted near the bottom of the extraction drum. At the lower end of the drain tube a 0.75-in. NPT tee coupling was connected to accommodate a brass drain valve and a 0.75-in. return tube.
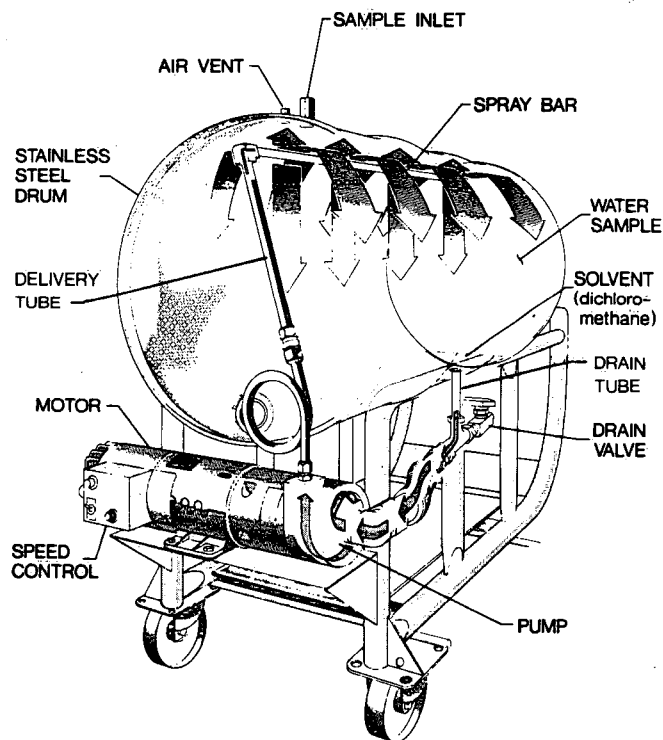

Figure 1. Cut-away illustration of the Aqueous Phase Liquid-liquid Extractor (APLE).

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Preceding the pump intake, a piece of 0.75-in. flexible stainless steel tubing was inserted into the return tube to reduce stress fatigue on the Teflon pump casing.

A magnetically driven variable speed centrifugal pump (Jupiter MPC-25-1225) was chosen, which prevented the risk of lubricant seepage into the pump head assembly. All wetted pump parts are made of Teflon, with the exception of a ceramic shaft. The pump was mounted on a bracket so that the intake was 10 cm above the drain valve to ensure proper drainage, and 3 cm below the extraction drum to facilitate the circulation of relatively small quantities of solvent for cleaning purposes.

The delivery tube assembly consists of two 0.5-in. stainless steel tubes. The lower tube was bent, forming a loop, to reduce stress fatigue and permit ease of assembly. A 0.75-in. inlet pipe having a length of 4 cm was passed through a 3-cm diameter hole drilled in the bung and welded in place. The upper delivery tube was then connected to the inlet pipe with an elbow coupling. One end of the 75-cm long spray bar (0.5-in. I.D.) was sealed with a weld. A 2-mm hole was then drilled through the weld to provide spray action on the end face of the extraction drum. Four rows of 12 holes, 2 mm in diameter, were drilled along the full length of the spray bar, 90° apart, such that they were evenly spaced. The spray bar was then mounted to the inlet pipe with a compression fitting to permit rotational adjustment (Fig. 2). This adjustment mechanism ensured that the spray could be directed in horizontal and vertical planes.
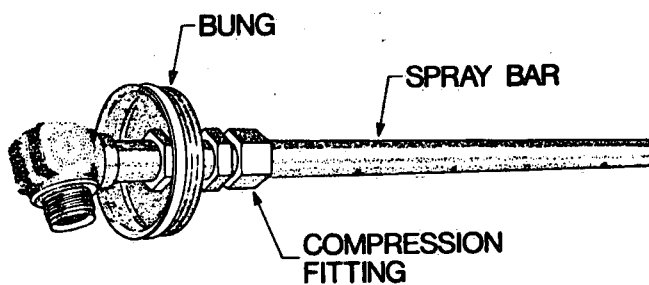

Figure 2. Illustration of the APLE spray bar and fittings.

OPERATION

Liquid-liquid extraction consists of three operations: (1) bringing the sample to be extracted into intimate contact with the solvent, (2) separating the two phases, and (3) recirculating and/or removing the solvent for analyses. It is important to provide a large interfacial area between the sample to be extracted and the solvent, in order to obtain good mass transfer between the two phases.

Thorough mixing is desired in the extraction process; vigorous mixing, however, should be avoided, as subsequent emulsion formation may create separation difficulties (Wu and Suffet, 1975).

The APLE system works much like a separatory funnel which is normally inverted and shaken, permitting the two liquid phases of different densities to contact one another. This step is usually repeated a number of times to provide sufficient solvent-water contact for complete extraction. In the case of the APLE system, the water phase tends to be more stationary. The solvent (dichloromethane, d = 1.326) is continuously pumped from the bottom of the extraction drum to the spray bar, where it is dispersed over the entire surface of the water sample by either striking the water surface directly, or immediately after deflecting off the walls of the drum. Contaminants are extracted into the solvent phase as a result of mixing in the pump head and by the solvent-enriched spray, which permeates the entire water sample and settles to the bottom of the extraction drum. The tilt of the extraction drum permits solvent to collect above the vertical drain pipe, where it is continuously drawn up and returned under pump pressure to the spray bar.

The extraction drum is typically filled to its maximum capacity of 223 L with clarified water, and 23 L is subsequently drained to obtain a standard working volume of 200 L. Eight litres of pesticide grade dichloromethane is then added to the extraction drum. This solvent is relatively safe to use in the field because it is nonexplosive and will not support combustion. The pump is then started to initiate extraction, and its speed adjusted to 70% of maximum rpm, resulting in an effective recirculating rate of approximately 12 L/min. After 90 min of extraction, the pump is turned off to permit the solvent to settle out of the sample water; 120 min later, the solvent is drained back into the original amber solvent bottles. A small volume of sample water is included during the draining process to minimize the volatilization of the solvent. Since dichloromethane is slightly soluble in water (1.5% V/V), approximately 3 of the original 8 L of solvent is not recovered. The samples are stored at 4°C to retard possible degradation of less stable organic contaminants (Environment Canada, 1979).

EXPERIMENTAL PROCEDURE

A spiking solution was prepared in methanol from a stock solution. The spiking solution contained 500 pg/μL of HCB; α-BHC; γ-BHC; heptachlor; aldrin; heptachlor epoxide; γ-chlordane; α-chlordane; α-endosulfan; p,p'-DDE; dieldrin; endrin; o,p'-DDT; p,p'-TDE; p,p'-DDT; β-endosulfan; mirex and p,p'-methoxychlor. A standard

solution of 10 ng/μL was prepared in benzene from the same stock for analytical purposes.

Approximately 200 L of tap water was passed through a 1-μm glass fibre filter and added to the extraction drum. This water was extracted three times with dichloromethane prior to each spike injection, to remove any residual trace organic contamination. During the initial testing in March 1982, the extraction drum was spiked with appropriate volumes of solution to produce six replicates of 10, 50 and 200 ng, simulating environmental concentrations of 0.05, 0.25 and 1.0 ppt, respectively. Immediately after each spike injection, the pump was operated for 30 min to ensure thorough mixing. Since the test water was saturated with dichloromethane, only 5 L of the solvent was added. The extractions were of a duration of 90 min after which the solvent was permitted to settle out for a period of 120 min.

The APLE extracts were dehydrated using an 8-cm column of anhydrous Na_2SO_4, which was previously heated overnight at 650°C. Approximately 700 mL of anhydrous extract was added to a 1-L round-bottomed flask containing 40 mL of iso-octane. The extract at 40°C was evaporated under vacuum to a volume of 50 mL. After all the dichloromethane was evaporated, the sample was further concentrated to 4 mL in iso-octane. These concentrated extracts were then fractionated into two fractions (A and B) using silica gel column chromatography. Electron capture detection analysis was later performed using two packed columns and a capillary column (Table 1).

Further testing of the APLE was performed in April 1983. This included one spike recovery at the 10-ng level, six at 50 ng and three at 200 ng. In addition, three method spikes at the 50-ng level were also prepared for analyses.

RESULTS AND DISCUSSION

In a recovery test of this type, there are three main sources of error and loss: analytical error, spiking error and incomplete recovery of the spike addition by the extractor system. Systematic errors in the analytical process are reduced by means of blanks and method spikes. These quality control measures are used to generate correction factors for each contaminant, which are then applied to the raw data, to compensate for expected losses during laboratory analyses. Random errors are not as easily identified or quantified. However, it has been found that the mean coefficient of variation for organochlorine analyses of water is approximately 20% (Thompson, 1984; Jamro, 1984). Error introduced by the spiking process was minimal, as the accuracy of the syringes used was ±1%.

During the recovery test in March 1982, the APLE was spiked at three concentrations in replicates of six. Recovery of the 18 contaminants at the 10-ng level was good for five of the six replicates (see Table A-1 in Appendix). The sixth spike, however, had an abnormally low recovery compared with the first five. Similarly, the recovery of spike 11 was low when compared with the other spikes at the 50-ng level (Table A-2). The relatively poor recoveries of these two spikes was thought to be due to the roto-evaporation process; thus they were excluded from statistical analyses.

Analyses of spikes 10 and 12 at the 50-ng level showed that the recoveries of contaminants isolated in the "A" fraction (denoted by an "A" in Table A-2) were considerably higher than contaminants in the "B" fraction. The mean recoveries of contaminants isolated in fractions "A" and "B" for these two spikes were 103% and 71%, respectively. Similarly, recovery differences of 30% were found between the "A" and "B" fractions of spikes 14, 16 and 17, which had been spiked with 200 ng of each contaminant (Table A-3). The low recoveries of organochlorine contaminants normally isolated in the "B" fraction were

Table 1. Operational Characteristics of Chromatographic Systems Used in the APLE Recovery Efficiency Analyses

Column	Operational characteristics
Silica gel	30 cm × 8 mm I.D. Pyrex, 8-cm plug of 3% H_2O deactivated silica gel + 2 cm Na_2SO_4
	25 mL hexane fraction A 30 mL benzene fraction B
Packed	A-1.5% OV-17/1.95% QF-1, 2 m × 0.2 mm I.D.
	B-4% OV-101/6% OV210, 2 m × 0.2 mm I.D.
Carrier gas	30 mL min^{-1}, 5% CH_4 in Ar (P5)
Detector	Electron capture ^{63}Ni at 300°C
Oven temperature	190°C
Capillary	30 m × 0.25 I.D., SE 30
Carrier gas	H_2 at 30 cm s^{-1}
Injection	Splitless, 30-s wait
Temperature program	80°C (2 min), to 260°C at 4°C min^{-1}, 12 - min hold
Split	30 cm^3 min^{-1}
Detector	^{63}Ni electron capture
Operating temperature	300°C
Makeup flow	30 mL min^{-1}, 5% CH_4 in Ar

Table 2. Recovery of 50-ng Method Spikes and the Determination of Correction Factors for the Second Spike Recovery Test Conducted in April 1983

Parameter	Method spike 1	Method spike 2	Method spike 3	Mean recovery	S.D.	Factor	APLE recovery
HCB	9.0	8.5	11	(9.6)	1.5	—	(14.0)
α-BHC	36	43	30	36	7.1	1.4	38
γ-BHC	33	45	31	36	7.9	1.4	34
Heptachlor	28	27	39	31	6.6	1.6	41
Aldrin	31	25	33	30	4.1	1.7	44
Heptachlor epoxide	40	45	38	41	3.4	1.2	38
γ-Chlordane	40	46	40	42	3.7	1.2	39
α-Chlordane	35	48	36	40	7.5	1.3	37
α-Endosulfan	31	41	30	34	6.2	1.5	31
p,p'-DDE	34	35	51	40	9.6	1.3	48
Dieldrin	39	44	38	41	2.9	1.2	41
Endrin	31	39	33	34	4.0	1.5	32
o,p'-DDT	14	8.8	36	(20)	14.2	—	(28)
p,p'-TDE	29	37	28	31	4.7	1.6	29
p,p'-DDT	17	7.8	14	(13)	4.6	—	(11)
β-Endosulfan	39	42	37	40	2.3	1.3	37
Mirex	36	35	47	39	6.8	1.3	42
p,p'-Methoxychlor	13	28	21	(21)	7.5	—	(19)

Notes: APLE recovery data are based on the mean of the six uncorrected 50-ng replicates from the April 1983 trial.

Data are expressed in nanograms.

Table 3. Mean Percent Recovery of 18 Organochlorine Contaminants with the APLE System Spiked with 10, 50 and 200 ng of Each Compound, Simulating Environmental Concentrations of 0.050, 0.25 and 1.0 ppt, Respectively

Parameter	Percent recovery 10 ng	Percent recovery 50 ng	Percent recovery 20 ng	10–200 ng*
HCB	66	80	64	70
α-BHC	74	84	74	88
γ-BHC	88	92	84	88
Heptachlor	97	125	85	102
Aldrin	126	149	94	123
Heptachlor epoxide	87	95	86	89
γ-Chlordane	90	94	87	90
α-Chlordane	94	91	70	85
α-Endosulfan	74	78	84	79
p,p'-DDE	112	114	81	102
Dieldrin	102	103	98	101
Endrin	85	88	89	87
o,p'-DDT	87	71	59	72
p,p'-TDE	103	98	94	98
p,p'-DDT	112	103	69	95
β-Endosulfan	86	83	91	87
Mirex	65	111	90	87
p,p'-Methoxychlor	128	124	96	116
Overall recovery	93	99	83	92
Mean Coefficient of Variation	19	27	23	—

* Mean percent recovery is based on all data collected for the 10- to 200-ng range.

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attributed to laboratory losses during or following the fractionation process. These data were also excluded from statistical analyses despite excellent recoveries of many contaminants present in the "A" fraction.

As part of the second recovery test in April 1983, three method spikes containing 50 ng of each contaminant were prepared by injecting the standard spiking solution directly into a 4-L bottle of pesticide grade dichloromethane. Recovery data for these method spikes were employed to determine the appropriate correction factor for each of the compounds investigated. Analyses showed poor recoveries for HCB; o,p'-DDT; p,p'-DDT; and p,p'-methoxychlor (Table 2). Correction factors for these compounds would have been greater than 2, and as a result were thought to be unreliable. The percent recoveries of the APLE extracts could not be calculated for the above compounds. However, recoveries of the method spikes were comparable to the uncorrected APLE recovery data at the 50-ng level (Table 2), suggesting that the APLE system effectively extracted these four compounds despite laboratory losses.

Recoveries of the other 14 organochlorine compounds were satisfactory, as indicated by the method spikes (Table 2). Data generated at the three spiking levels were corrected and are presented along with results of the first test in the Appendix.

Analyses of the 10-ng spikes, as shown in the combined data set (Table A-1), indicated that the extraction and isolation of contaminants with the APLE system were good. Of the 18 organochlorine contaminants studied, 14 had recoveries equal to or greater than 85%. Overall, the mean recovery and coefficient of variation of the 10-ng replicates were 93% and 19%, respectively (Table 3). Results of the 50-ng spike tests were somewhat more variable than at the 10-ng level. The mean recovery of contaminants at the 50-ng level was 99%, with a mean coefficient of variation of 27%. Recovery efficiencies at the 200-ng level were found to range from 64% to 98%, with a mean percent recovery and coefficient of variation of 83% and 23%, respectively.

Over the range of concentrations studied, the mean percent recovery of organochlorine contaminants was found to range from 70% to 123%, with an overall mean recovery of 92%. Analyses of secondary extracts, which are an indirect measure of the extraction efficiency, were performed immediately following the extraction of spikes 10 and 17. Results indicated that on average less than 5% of the spike remained in the APLE system following the initial extractions, corroborating the results obtained in the spike recovery tests.

The key parameter in an extraction process is the solvent-water partition coefficient. Unfortunately, values for a dichloromethane-water system are not well documented. Octanol-water partition coefficients, however, have been determined for many of the compounds; they range from log 4 to log 10. It is likely that values for dichloromethane-water would not be very different from these.

Extraction efficiency plots for a single-stage extractor, such as the APLE, have been computed for various solvent-water ratios and partition coefficients (Fig. 3). These curves indicate that the recovery of contaminants with the APLE, which has solvent-water ratios of 10:1 in the pump head and 40:1 in the drum, would be 98% or better for substances with partition coefficients of log 3 or greater.

In an experiment of this design, it is not possible to identify and quantify clearly the variability associated with extraction and subsequent analyses. Nevertheless, the total variability, as shown by the mean coefficients of variation (Table 3), was similar to that of the expected variability associated with these types of analyses (Thompson, 1984; Jamro, 1984) and to the method spike value of 20.2%. This indicated that a major component of the total variability was due to the analytical method.

The performance of the APLE system in this study clearly demonstrated that it is capable of extracting a wide range of organochlorine contaminants. Owing to the large sample volume extracted, the detection limit of these compounds is effectively reduced relative to traditional 1- and 2-L samples. It is thought that the detection of low level organochlorine contaminants will provide useful information concerning concentration and loading in the aqueous phase, as well as the distribution and partitioning of these contaminants in natural waters.
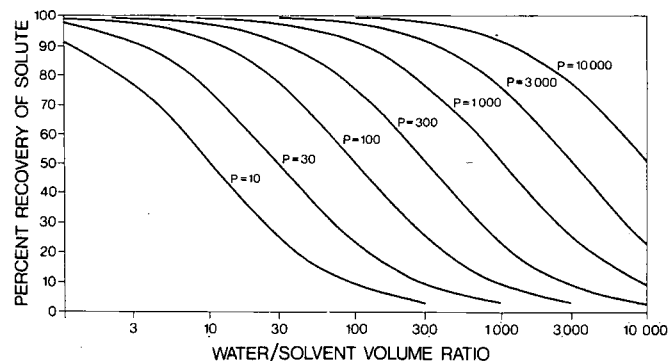

Figure 3. Extraction efficiency plots for a single-stage extractor at various solvent-water ratios and partition coefficients.

In view of the suspected loss of contaminants during the solvent concentration procedure, other techniques are now being investigated to minimize this analytical problem. A reduction in the amount of solvent added to the system may also provide an effective means for minimizing the solvent concentration problem without significantly decreasing the extraction efficiency. The isolation and concentration of other contaminants with this field extraction system should be subject to further testing. It should also be noted that several changes have since been made to the original extraction drum for ease of operation.

ACKOWLEDGMENTS

REFERENCES

DeLappe, B.W., R.W. Risebrough, A.M. Springer, T.T. Schmidt, J.C. Shropshire, E.F. Letterman and J.R. Payne. 1978. The sampling and measurement of hydrocarbons in natural waters. *Hydrocarbons and Halogenated Hydrocarbons in the Aquatic Environment*, pp. 29-68.

Environment Canada. 1979. *Analytical Methods Manual*. Inland Waters Directorate, Water Quality Branch, Ottawa.

Jamro, G.H. 1984. Personal communication, National Water Quality Laboratory, Burlington, Ontario.

Osterroht, C. 1974. Development of a method for the extraction and determination of non-polar dissolved organic substances in sea water. J. Chromatogr. 101: 289-298.

Thomas, R.L. and R.K. McMillan. 1978. Large volume water sampling for recovery of suspended solids in Great Lakes tributaries. Abstr. Int. Sedimentology Congr., p. 670.

Thompson, R.D. 1984. Personal communication (former Head, Organic Analyses Laboratory, Water Quality Branch, Ontario Region). Zenon Analytical Services, Burlington, Ontario.

Wu, C. 1975. Investigation of Liquid-Liquid Extraction Parameters and Development of a Continuous Liquid-Liquid Extraction Apparatus. Ph.D. Thesis, Drexel University, Philadelphia.

Wu, C. and I.H. Suffet. 1975. Continuous liquid-liquid extraction of organic pesticides from aqueous solutions. Water Pollution Assessment. Automatic Sampling and Measurement, ASTM STP 582, American Society for Testing and Materials, pp. 90-107.

Wu, C. and I.H. Suffet. 1977. Extraction apparatus and its application for the analysis of organophosphate pesticides in water. Anal. Chem. 49(2): 231-232.

Appendix

Appendix

Table A-1. Recovery of 18 Organochlorine Contaminants with the APLE System Spiked with 10 ng of Each Compound, Simulating an Environmental Concentration of 0.05 ppt.

Parameter	Spike							Mean	Mean percent recovery	Coefficient of variation
	1	2	3	4	5	6*	6r			
HCB	6.6	8.2	6.6	7.4	4.4	2.9	—	6.6	66	21
α-BHC	8.2	9.4	7.8	5.7	7.1	4.9	6.1	7.4	74	18
γ-BHC	9.9	9.4	8.4	7.1	8.0	6.5	10.0	8.8	88	13
Heptachlor	10.4	8.7	7.6	7.3	11.2	2.3	13.0	9.7	97	23
Aldrin	12.8	8.6	12.2	10.3	4.6	1.2	26.9	12.6	126	60
Heptachlor epoxide	8.1	10.2	7.2	6.3	8.5	6.0	12.1	8.7	87	24
γ-Chlordane	7.8	8.8	9.5	8.9	9.6	9.3	9.6	9.0	90	8
α-Chlordane	8.5	9.6	10.2	9.7	10.3	8.8	7.9	9.4	94	11
α-Endosulfan	8.5	5.9	5.6	4.4	9.9	6.2	9.9	7.4	74	32
p,p'-DDE	12.3	9.9	10.2	9.9	12.3	4.3	12.4	11.5	112	11
Dieldrin	12.3	9.0	10.2	10.2	9.3	9.5	10.1	10.2	102	11
Endrin	9.7	7.8	8.2	6.9	9.9	6.5	8.3	8.5	85	14
o,p'-DDT	8.1	8.4	7.4	7.5	12.2	7.7	—	8.7	87	23
p,p'-TDE	9.8	9.1	9.3	8.1	14.6	7.9	10.6	10.3	103	22
p,p'-DDT	11.1	10.8	11.8	10.7	11.6	10.0	—	11.2	112	4
β-Endosulfan	10.8	8.2	8.6	6.4	9.3	5.7	8.0	8.6	86	17
Mirex	4.8	6.2	5.4	5.9	5.7	3.5	11.0	6.5	65	35
p,p'-Methoxychlor	16.5	12.6	15.2	11.3	10.5	12.1	—	12.8	128	17

Notes: Spikes 1 to 6 are from the March 1982 trial; spike 6r is from the April 1983 trial. Spike 6* was excluded from the statistical analyses.

Data are expressed in nanograms.

Table A-2. Recovery of 18 Organochlorine Contaminants with the APLE System Spiked with 50 ng of Each Compound, Simulating an Environmental Concentration of 0.25 ppt

Parameter	Spike 7	8	9	10*	11*	12*	7r	8r	9r	10r	11r	12r	Mean	Mean percent recovery	Coefficient of variation
HCB	55	38	27	54A	14	53A	—	—	—	—	—	—	40	80	35
α-BHC	42	42	20	26	14	36	47	60	51	41	42	32	42	84	27
γ-BHC	53	53	29	35	19	43	46	78	49	37	38	29	46	92	33
Heptachlor	58	53	53	52A	30	60A	49	77	76	53	65	69	63	125	17
Aldrin	11	57	152	62A	32	60A	51	96	91	62	71	78	74	149	52
Heptachlor epoxide	55	53	38	36	23	41	52	59	56	40	41	32	47	95	20
γ-Chlordane	54	55	40	37A	23	43A	47	61	51	40	43	33	47	94	19
α-Chlordane	51	37	26	25	22	30	44	94	53	38	41	27	46	91	44
α-Endosulfan	40	23	14	15	15	18	51	72	52	37	38	24	39	78	45
p,p'-DDE	54	49	48	50	25	48	38	72	77	55	59	61	57	114	21
Dieldrin	60	60	42	59	24	46	52	51	52	41	73	33	52	103	23
Endrin	44	46	30	27	34	34	56	54	54	38	44	31	44	88	22
o,p'-DDT	36	42	28	28	13	35	—	—	—	—	—	—	35	71	20
p,p'-TDE	59	65	46	39	22	48	48	66	51	32	42	31	49	98	26
p,p'-DDT	55	59	41	38	24	44	—	—	—	—	—	—	52	103	18
β-Endosulfan	36	33	21	22	28	25	59	67	47	36	48	27	42	83	36
Mirex	67	53	57	59A	31	57A	35	37	73	56	58	63	55	111	23
p,p'-Methoxychlor	68	63	55	38	25	49	—	—	—	—	—	—	62	124	11

Notes: Spikes 7 to 12 are from the March 1982 trial; spikes 10*, 11* and 12*, however, were excluded from statistical analyses. Compounds present in the A fraction for the latter spikes are identified with an "A". Spikes 7r to 12r are from the April 1983 trial.

Data are expressed in nanograms.

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Table A-3. Recovery of 18 Organochlorine Contaminants with the APLE System Spiked with 200 ng of Each Compound, Simulating an Environmental Concentration of 1.0 ppt

Parameter	Spike									Mean	Mean percent recovery	Coefficient of variation
	13	14*	15	16*	17*	18	14r	16r	17r	Mean		
HCB	109	164A	175	149A	153A	100	–	–	–	128	64	32
α-BHC	91	128	173	85	74	141	159	165	164	149	74	20
γ-BHC	130	160	214	122	85	185	164	160	159	169	84	17
Heptachlor	192	212A	183	192A	170A	71	243	234	100	171	85	41
Aldrin	181	190A	179	171A	76A	144	267	252	107	188	94	33
Heptachlor epoxide	142	154	195	104	100	136	186	178	200	173	86	16
γ-Chlordane	145	157A	195	106A	102A	150	190	176	187	174	87	12
α-Chlordane	99	106	123	71	55	96	175	173	174	140	70	27
α-Endosulfan	110	120	142	81	94	184	184	179	212	169	84	21
p,p'-DDE	182	161	145	163	141	97	231	211	98	161	81	35
Dieldrin	160	172	216	124	113	215	198	176	204	195	98	11
Endrin	134	125	151	93	96	177	204	184	212	177	89	17
o,p'-DDT	91	100	121	70	64	140	–	–	–	118	59	21
p,p'-TDE	144	157	166	120	94	195	200	192	232	188	94	16
p,p'-DDT	112	117	128	133	98	174	–	–	–	138	69	23
β-Endosulfan	128	133	163	95	114	163	199	191	250	182	91	23
Mirex	217	193A	197	197A	191A	145	225	200	100	181	90	27
p,p'-Methoxychlor	172	169	216	133	107	189	–	–	–	192	96	12

Notes: Spikes 13 to 18 are from the March 1982 trial; spikes 14*, 16* and 17*, however, were excluded from statistical analyses. Compounds present in the A fraction for the latter spikes are identified with an "A". Spikes 14r and 16r and 17r are from April 1983 trial.

Data are expressed in nanograms.

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