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Accelerated Solvent Extraction (ASE) as a Replacement for Soxhlet Extraction: Extraction of Native PCBs from Certified reference Sediments

By:

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ACCELERATED SOLVENT EXTRACTION (ASE)
AS A REPLACEMENT FOR SOXHLET EXTRACTION:
EXTRACTION OF NATIVE PCBS FROM
CERTIFIED REFERENCE SEDIMENTS

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Abstract

An ISCO model SFX 2-10 supercritical fluid extractor was used to extract native PCBs from well characterized sediments from Hamilton Harbour and Lake Erie using dichloromethane under non supercritical conditions - the so called **Accelerated Solvent Extraction (ASE)** technique employed by EPA method 3545. A range of operational parameters were explored and the results expressed as percentage recoveries of 24 native PCB congeners relative to traditional Soxhlet extraction. Relative recoveries of the PCB congeners **as high as 136%** was achieved under the most optimum ASE conditions. There was more than a **25 fold savings** in both time and volume of solvent used. This report represents an extension of our earlier NWRI Contribution 95-183 incorporating recent advances in the art. The overall utility of this technique is similar to that reported in the earlier study, however, technical difficulties relating to the transfer and trapping of the extract are overcome in this procedure.

Accelerated Solvent Extraction (ASE) as a replacement for Soxhlet extraction: extraction of native PCBs from certified reference sediments.

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Management Perspective

Issue

Current procedures for the extraction of contaminants from sediments are labour intensive, time consuming and produce large volumes of waste solvents. Restrictions on the use, transport and disposal of hazardous solvents are expected to become more stringent. New procedures are required to address these issues.

Background

Recent advances in laboratory instrumentation have resulted in commercially available instruments utilising processes such as supercritical fluid extraction and microwave extraction. These techniques have the potential to greatly outperform traditional Soxhlet extraction of environmental samples and yet have not been fully proven to possess the versatility of Soxhlet extraction.

Status

We have tested several of the currently available techniques, most recently supercritical fluid extraction (SFE) of sediments using an ISCO SFX2-10 extractor (NWRI Contribution 95-183). This report addresses the problems reported in that contribution by using the same apparatus to perform Accelerated Solvent Extraction (ASE) of native PCBs from sediments. We extracted up to 136% of the total of 24 PCB congeners recovered by Soxhlet extraction with a more than 25-fold saving in both time and volume of solvent used. The technical difficulties experienced with the SFE procedure were overcome in this study.

Next Steps

Further validation to extend the range of analytes and sample matrices.

Accelerated Solvent Extraction (ASE) as a replacement for Soxhlet extraction: extraction of native PCBs from certified reference sediments

Traditional contaminant extraction techniques are time consuming and labour intensive. A typical Soxhlet extraction of sediment associated PCBs, for example, may take 16 to 20 hours and requires large volumes of toxic organic solvent. Consequently, new technologies have been developed that promise quicker and cheaper extractions with comparable extraction efficiencies.

Accelerated Solvent Extraction is one such technology. ASE uses a solvent that is heated above its boiling point under pressure and forced through a solid sample. An exhaustive extraction is completed in under 10 minutes using 10 to 20 mL of solvent. ASE appears to be a preferable alternative to traditional extraction methods since the extraction procedure is robust and easy to perform. Sample turnover is greatly increased resulting in higher lab productivity. Dionex Corp., a commercial manufacturer of an automated ASE extractor, suggests that the per sample cost of ASE ($14) is significantly lower than that of Soxhlet extractions ($27) (Dionex product literature, 1995). The cost to the environment is also reduced since much less toxic solvent is used and ultimately discarded or recycled.

ASE has recently been officially approved by the US EPA as a preparative method (EPA Method 3545) for extracting priority pollutants from soils, clays, and sediments. Several application notes have appeared describing the ASE extraction of spiked and native analytes from various solid environmental matrices (Ezzell et al, 1994; Woolley et al, 1995; Richer et al, 1995; Dionex Application Notes #313 to #320). Most recently, Richer et al (1996) extracted PCBs from certified sewage sludge and oyster tissue using ASE with satisfactory results.

In this report, we describe a statistically significant (n = 10) evaluation of the performance of ASE with respect to Soxhlet extraction. Native PCBs were extracted from two certified reference sediments containing approximately 2000 ng/g and 100 ng/g of total native PCBs respectively. Similar amount of sediments were extracted ten times by each method using the same solvent. Eighteen chromatographically separate PCB peaks were quantified against an external standard and the recoveries using both methods were compared.

Methods

Sediments

Two certified sediments were provided by Environmental Standards and Statistics (AEPB, National Water Research Institute, Burlington). Certified sediment EC-1 is a heavily contaminated Hamilton Harbour sediment with a certified total PCB content of 2000 ng/g with a standard deviation

of ± 50 ng/g. Certified sediment EC-6 is a less contaminated Lake Erie sediment with a certified total PCB content of 105 ng/g with a standard deviation of ± 19 ng/g. These sediments have been freeze dried and ground to less than a 200-mesh particle size.

Soxhlet extraction

Sediment samples (3 or 5g) were weighed in a tared beaker and mixed with 60 g of heat treated (450°C for >4 hours) anhydrous granular sodium sulphate (BDH). The glass frit of a glass Soxhlet extraction thimble was covered with a 1 cm bed of fine sodium sulphate and the homogenized sediment sample was added. Another 20 g of granular sodium sulphate was added to the beaker to pick up any sediment residue. This fraction was also added to the extraction thimble. A solvent rinsed 500 mL round bottom flask was filled with 300 mL of dichloromethane (DCM) (Caledon, Distilled in Glass). Several glass boiling chips and a small amount of activated copper turnings regenerated with acid and solvent rinsed were added to the flask. The solvent was allowed to flux through the sample for approximately 20 hours.

ASE

An ISCO SFX™ 2-10 extractor was used to perform ASE in this study. The instrument has two 260 mL syringe pumps, separate heat and pressure controllers, an extraction module, and a heated variable restrictor. It is designed to be used as a supercritical fluid extractor. This instrument provided satisfactory performance for ASE extractions with no modifications. The use of a supercritical fluid extractor for the purpose of ASE has been documented elsewhere (Woolley *et al*, 199x). The ASE extraction procedure is described below.

A 10 mL stainless steel extraction vessel was layered with 0.5 g of Celite 545 (Fisher Scientific, Nepean, Ontario). The dry, homogenised sediment sample (3 or 5 g) was then added directly to the vessel. The sample was retained at both ends of the vessel with Gelman A/E glass fibre filter paper (nominal pore size of 1 µm) and stainless steel frits nominal pore size of 5 µm. The loaded cell was capped at both ends with PEAK™ screw caps and the whole assembly was then positioned inside the extractor. The sample was exposed to DCM for 5 minutes at 105°C and 2000 psi (US EPA, 1995). This portion of the extraction is termed *static* since the solvent simply bathes the sediment without any actual removal of analyte. The restrictor valve was then opened and the solvent was allowed to gently elute from the sample at a rate of 3 mL per minute. This portion of the extraction is referred to as *dynamic* because there is fluid flow resulting in analyte removal. The solvent pump was interrupted after 10 mLs of DCM solvent had been collected into a 50 mL round bottom flask containing a few flakes of activated copper. At this point, the residual solvent was removed with pressurized CO_2 (Air Products, Ottawa) yielding a total of 20 mLs of eluant.

Extract concentration and clean-up

Post extraction concentration and clean up was the same for both methods. PCB congener 204 (100 ng in 100 μL of trimethylpentane) and 1 mL of hexane (Caledon, Distilled in Glass) were added immediately after the extraction was completed. The copper turnings were allowed to remain in the extract in order to enhance sulphur removal. The extract was then reduced to < 1 mL using a Buchi Rotavapor at 510 mBar. A 10 mL glass pipette column was prepared with 2 g (5 cm) heat treated 60/100 mesh Florisil (Supelco, Mississauga) deactivated with 2% w/w distilled water. The column was topped off with 1 cm of heat treated granular sodium sulphate and rinsed with 10 mLs of pentane (Baker, Resi-Analyzed). The extract was then applied to the column with two small rinses of pentane. A total of 11 mLs of pentane was eluted through the column into a 50 mL round bottom flask. One mL of hexane was added and the eluent was rotary evaporated to < 1 mL. The final volume was adjusted to 2 mLs in a volumetric flask. A portion of the cleaned up extract was placed in the glass insert of a GC vial and prepared for analysis. The remainder was archived in the dark at -20°C.

GC analysis

PCBs were analyzed using a dual column HP5890 Series II gas chromatograph equipped with an HP7673A autoinjector and dual electron capture detectors at 325°C. The analytical column was a 30 m long by 0.25 mm ID with a 0.25 um film DB5 microbore column (J&W Scientific) and was paralleled by a 30 m HP50 column of the same dimensions (Hewlett Packard) . A splitless 2 μL injection was delivered to the inlet at 230°C and purged after 1 minute. The 67 minute temperature program started at 70°C and rose to 150°C at 10°C per minute, then to 250°C at 2°C per minute, and finally to 280°C at 10°C per minute with a final hold of 5 minutes. The hydrogen carrier was kept at a constant linear velocity of 63.4 cm/sec.

Data was collected on a Hewlett Packard Vectra personal computer. Chromatograms were integrated using HPChemStation software (Hewlett Packard, Mississauga, Ontario). PCBs were quantified using an external NOI standard consisting of Arochlors 1016, 1221, 1224, 1254, and 1262 in the proportion of 1:1:1:1:1 (Comba et al, 1996). The NOI standard is a mixture of Arochlors that is designed to mimic environmental PCB concentrations. The NOI standard was checked against two other standards. A 24 PCB standard was created from stock solutions of individual congeners, the concentration of each PCB being 40 pg/μL in trimethylpentane. An additional 3 congener PCB standard was created from pristine ampuled stock solutions and diluted to 40 pg/μL in trimethylpentane. Both standards compared favourably to the NOI.

Data processing

The PCBs examined in this study were chosen on the basis of their unambiguous chromatography. An effort was also made to chose PCB peaks ranging in size and degree of chlorination. Eighteen clearly defined PCB peaks were chosen. Many of the PCB peaks are a result of co-elution of more than one PCB. As a result, the 18 peaks actually consisted of 24 PCB congeners. Peak composition is detailed in Tables 1-4. PCBs that contribute very little to the peak area are contained in brackets.

Raw data was corrected for the recovery of the PCB 204 spike before calculating the averages. Recovery of the PCB spike was generally greater than 90%. All PCB values are in ng/g. One-tailed T-tests (unequal variances) were performed to test the significance of differences between the means of treatments (QuattroPro). A difference was considered significant at the 0.05α level. Relative recovery (RR) is defined as the percentage ratio of ASE/Soxhlet values.

Results

Table 1 compares the recoveries of the PCB congeners from Hamilton Harbour (EC-1) sediments (3 g) using both Soxhlet (n=10) and ASE (n=10) methods. The total recovery of the 18 PCBs was higher using the ASE method (RR = 108.7%). The difference was not significant according to the one-tailed T-test (t_{crit}=1.77, df=13, P=0.079) at the 0.05α level. The ASE SD (14.5%) was twice that of Soxhlet (7.6%) indicating that the Hamilton Harbour PCB measurements were generally less precise when ASE was used.

Five g of Lake Erie (EC-6) sediments were analyzed using the Soxhlet method (n = 10) and the ASE method (n = 5). The results are shown in Table 2. The total recovery of the 18 PCB peaks was higher for the Soxhlet method than for the ASE method (RR = 93%). Again however, the difference does not appear to be significant (t_{crit}=1.86, df=8, P=0.213). The SDs were similar for both treatments (13.9% and 14.4% respectively).

Three grams of Lake Erie sediment (n = 5) were also extracted by the ASE method and compared with the 5 g Soxhlet data (from Table 2). Lake Erie was re-examined using 3 g of sediment in order to investigate the effect of sample size on ASE. These results are shown in Table 3. ASE extraction efficiency for the 18 selected PCBs significantly improved when a smaller 3 g sample was used (RR = 135.8%) (t_{crit}=1.78, df=12, P=6.27x10^{-6}). Individual PCB peaks from the 3 g ASE extraction were higher than those of from the Soxhlet method (RRs from 111.0% to 154.9%).

The overall PCB recovery from the 3 g Lake Erie ASE was significantly greater than the 5 g Lake Erie ASE from Table 2 (t_{crit}=2.02, df=5, P=8.70x10^{-4}). The 3 g ASE extractions (n=5) had

TABLE 1. Average concentration for PCB congener peaks in Hamilton Harbour sediments ng/g.
One standard deviation (SD), percent standard deviation (%SD), and n values are indicated.

| Congeners | Soxhlet 3g (n=10) | | | ASE 3g (n=10) | | | Relative Recovery % |
	AVG	SD	%SD	AVG	SD	%SD	
24(27)	17.9	1.9	10.3	18.3	2.7	15.0	101.8
16,32	34.1	5.6	16.4	31.6	4.1	13.0	92.7
52	132.0	10.1	7.7	125.4	15.5	12.4	95.0
44	71.2	5.7	8.1	69.3	7.8	11.2	97.3
91,55	30.3	3.2	10.4	28.2	3.3	11.6	93.0
101	119.7	8.1	6.8	120.0	15.2	12.7	100.2
99	53.9	3.5	6.5	56.6	7.3	12.9	104.9
110	116.7	9.6	8.2	120.8	13.0	10.8	103.5
135(144)	28.4	2.9	10.1	28.6	3.4	12.0	100.8
153,132,105	102.4	6.1	6.0	112.5	13.4	11.9	109.8
163,138	97.1	8.0	8.2	110.0	14.3	13.0	113.4
182,187	37.7	2.0	5.4	38.5	4.4	11.5	102.2
174	27.1	2.1	7.9	27.6	3.5	12.5	101.8
180	52.1	6.3	12.1	53.3	25.6	48.0	102.4
201	18.2	2.5	13.8	19.2	4.9	25.3	105.4
195(208)	6.5	1.4	21.2	7.5	2.2	28.9	116.0
194	8.8	2.7	31.0	10.3	4.5	44.1	117.3
206	3.9	1.4	37.2	3.9	2.1	53.0	99.6
Total	958.1	72.7	7.6	1041.6	150.6	14.5	108.7

TABLE 2. Average concentration for PCB congener peaks in Lake Erie sediments in ng/g.
One standard deviation (SD), percent standard deviation (%SD), and n values are indicated.

| Congeners | Soxhlet 5g (n=10) | | | ASE 5g (n=5) | | | Relative Recovery % |
	AVG	SD	%SD	AVG	SD	%SD	
24(27)	2.7	0.3	12.9	2.3	0.6	25.0	86.9
16,32	2.9	0.6	20.5	2.4	0.8	33.0	81.6
52	16.1	2.1	12.9	14.7	2.5	17.2	91.4
44	7.1	0.6	9.1	6.5	1.3	19.6	91.2
91,55	6.4	1.2	18.9	5.4	0.7	12.4	84.5
101	14.8	2.0	13.2	14.3	2.0	14.1	96.7
99	6.9	0.9	13.1	7.1	1.0	14.1	102.0
110	16.4	2.2	13.6	16.1	2.6	16.3	98.0
135(144)	4.2	0.7	17.0	3.9	0.4	9.9	93.1
153,132,105	16.9	4.4	26.1	16.3	2.4	14.7	96.7
163,138	15.6	2.2	14.2	15.3	2.2	14.3	97.8
182,187	7.8	1.4	17.7	6.2	0.9	13.7	80.2
174	4.9	0.7	14.3	4.4	0.7	14.8	90.6
180	9.8	1.3	13.5	8.9	1.4	16.2	91.2
201	3.4	0.6	17.7	3.2	0.9	27.9	95.7
195(208)	1.7	0.3	15.9	1.5	0.3	18.6	85.8
194	2.8	0.5	16.9	2.4	0.4	17.4	84.5
206	1.4	0.2	15.5	1.1	0.1	13.1	82.1
Total	141.8	19.7	13.9	132.1	19.0	14.4	93.2

TABLE 3. Average concentration for PCB congener peaks in Lake Erie sediments in ng/g. One standard deviation (SD), percent standard deviation (%SD), and n values are indicated. Soxhlet sample load is 5 g (from Table 2).

Congeners	Soxhlet 5g (n=10)			ASE 3g (n=5)			Relative Recovery %
	AVG	SD	%SD	AVG	SD	%SD	
24(27)	2.7	0.3	12.9	3.3	0.2	6.2	123.0
16,32	2.9	0.6	20.5	3.8	0.2	5.9	131.7
52	16.1	2.1	12.9	21.3	0.6	2.6	132.6
44	7.1	0.6	9.1	9.3	0.6	6.1	130.2
91,55	6.4	1.2	18.9	8.2	0.4	4.7	128.7
101	14.8	2.0	13.2	20.8	0.4	2.1	140.5
99	6.9	0.9	13.1	10.6	0.3	2.8	152.5
110	16.4	2.2	13.6	23.1	0.6	2.7	140.7
135(144)	4.2	0.7	17.0	5.7	0.3	5.8	136.1
153,132,105	16.9	4.4	26.1	26.2	3.2	12.4	154.9
163,138	15.6	2.2	14.2	21.7	0.5	2.1	138.5
182,187	7.8	1.4	17.7	8.7	0.2	2.3	112.4
174	4.9	0.7	14.3	6.2	0.2	2.6	127.3
180	9.8	1.3	13.5	12.5	0.5	4.0	127.5
201	3.4	0.6	17.7	3.8	0.2	4.4	113.9
195(208)	1.7	0.3	15.9	2.1	0.0	1.9	121.7
194	2.8	0.5	16.9	3.3	0.1	3.8	119.3
206	1.4	0.2	15.5	1.5	0.1	5.1	111.0
Total	141.8	19.7	13.9	192.2	5.4	2.8	135.6

Table 4. Average recovery (ng/g) of PCBs in procedural blanks using both ASE and Soxhlet methods. Standarddeviations (SD), percent standard deviations (%SD) and n values are shown. Total refers to the sum (ng/g) of the interference determined for the 18 PCB peaks only.

Congeners	Soxhlet (n=3)			ASE (n=5)		
	AVG	SD	%SD	AVG	SD	%SD
24(27)	nd			nd		
16,32	nd			nd		
52	2.2	1.6	72.0	0.9	1.1	126.0
44	0.6	0.8	141.4	nd		
91,55	nd			nd		
101	1.9	0.4	22.6	0.5	0.7	125.9
99	nd			nd		
110	1.4	0.3	23.3	nd		
135(144)	nd			0.7	0.6	90.2
153,132,105	nd			nd		
163,138	2.7	2.1	76.9	4.1	2.2	52.7
182,187	nd			nd		
174	nd			nd		
180	1.0	0.4	39.8	0.4	0.5	122.4
201	nd			nd		
195(208)	nd			nd		
194	nd			nd		
206	nd			nd		
Total	9.8	1.6	16.8	6.6	2.3	34.4

a very low SD (2.8%) as compared to Soxhlet (13.9%) or the 5 g ASE (14.4%) for Lake Erie sediment.

Procedural blanks were performed for both methods and reported in Table 4. The blanks were treated the same as the samples, except that no sediment was used. Six interference peaks were detected in the Soxhlet treatment while 5 were detected for ASE. The pattern of peaks found in the blank were similar for both methods, although not identical. The source of these interference peaks is not clear, although some sources can be ruled out. The extraction solvent can be ruled out since the more than tenfold difference in volumes used would have been reflected in similar ratios in the blank peaks. Similarly, carryover of analytes from the extraction of actual samples can be eliminated since no blank peak corresponding to PCBs 153,132,105 were observed. This peak was a major component in the real samples. The magnitude of the peaks found in the blanks were significantly less for ASE treated samples (6.6 ng/g) than for Soxhlet (9.8 ng/g) (t_{crit}=2.02, df=5, P=0.049). Standard deviations were higher for ASE blanks than for Soxhlet blanks (34.4% vs 16.8%). A simplified summary of results from Tables 1 to 4 are shown in Table 5.

Figure 1 demonstrates the effect of sample load on ASE extraction kinetics. The extraction was stopped at intervals and fractions were taken, quantified separately, then cumulatively added. The extraction performed with 1 g of EC-1 sediment (n=2, 5 fractions) is much more rapid than the 3 g extraction (n=2, 6 fractions) even though the extraction conditions were identical. For the 1 g treatment, the 90% recovery level is achieved at 5 mL of DCM extraction fluid. The 90% recovery level is achieved at 10 mL of DCM extraction fluid in the 3 g treatment. The RRs (compared to soxhlet) were 107% and 110% for the 1 g and 3 g treatments respectively.

The effect of this decrease in extract kinetics is shown in figure 2. These extractions were performed in duplicate with EC-1 sediment using only 20 mLs of extraction solvent. There appears to be a stepwise decrease in the amount of PCB (ng/g) extracted in relation to the amount of sediment used.

Table 5. Summary of results from Tables 1 to 4.

Sediment	Soxhlet Results			ASE Results			
	Weight (g)	PCB (ng/g)	SD (%)	Weight (g)	PCB (ng/g)	SD (%)	RR %
EC-1	3	958	7.6	3	1042	14.5	108.7
EC-6	5	142	13.9	5	132	14.4	93.2
EC-6				3	192	2.8	135.6
Blanks		9.8	16.8		6.6	34.4	

Figure 1. Kinetics of ASE recovery (relative to soxhlet) of 18 PCB peaks from Hamilton Harbour sediment. The recovery of PCBs from the 1 g sample is much more rapid than for the 3 g sample.
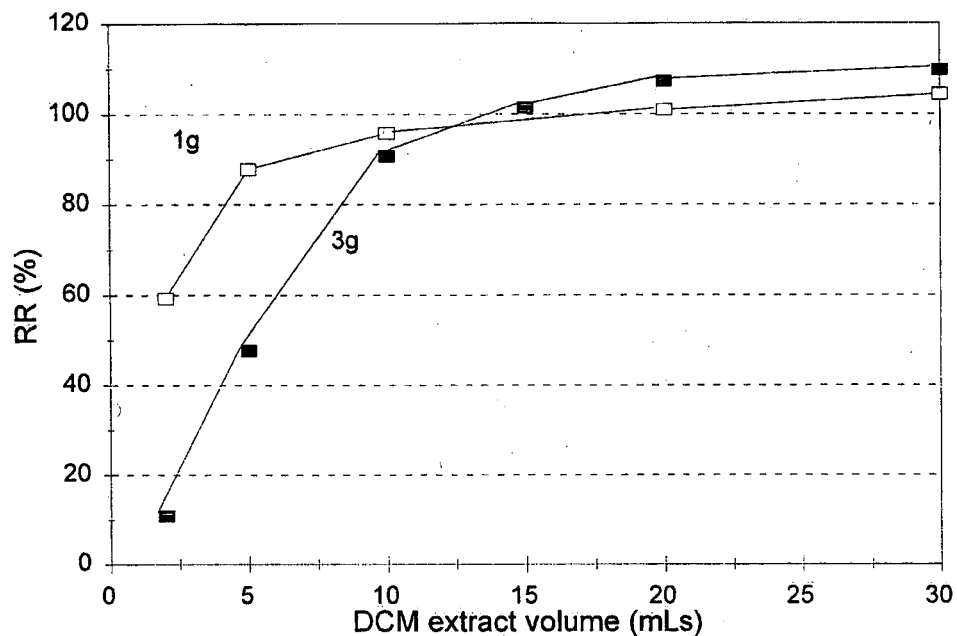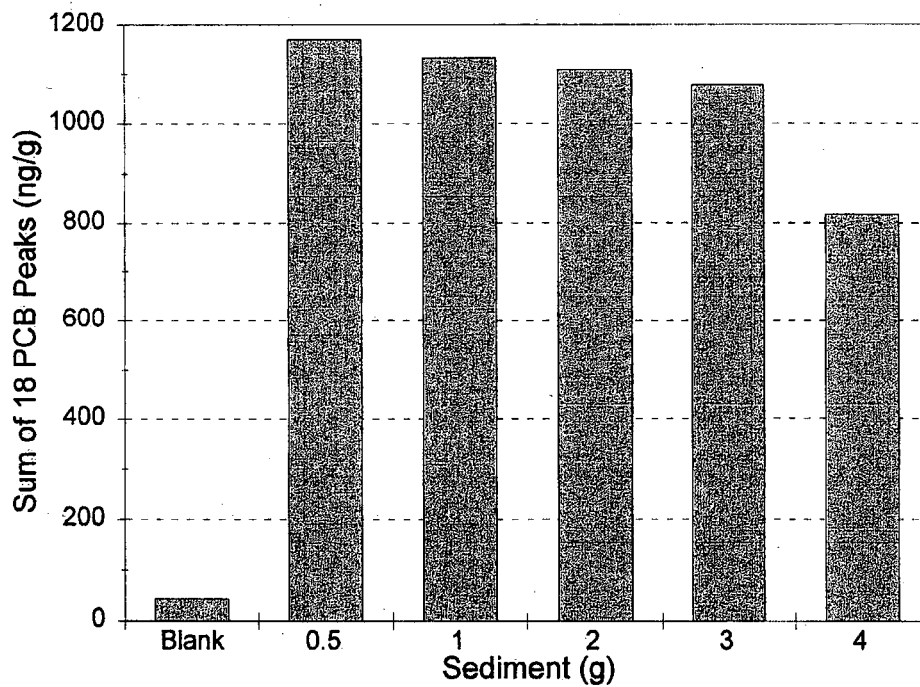

Figure 2. ASE extraction of Hamilton Harbour sediments. There appears to be a inverse relationship between PCB recovery and sediment load. The volume of the extraction solvent used was 20 mLs.

Discussion

The comparative evaluation of ASE versus Soxhlet extraction techniques was efficiently accomplished by examination of the recoveries of selected PCB congeners having a range of physicochemical properties such as vapour pressure and octanol-water partition coefficients. The data in Tables 1, 2, and 3 suggests that individual congener recovery using the ASE method does not differ greatly from the pattern of recovery for the Soxhlet method. There may be a slight bias favouring the ASE of the mid-Kow peaks since the RRs of these peaks are generally better than the lowest or highest Kow peaks. However, this point cannot be clearly resolved with this dataset.

The precision of a group of data can be estimated by looking at its SD. There appears to be no clear precision advantage when employing the ASE technique. The SD was ~14% for Hamilton Harbour ASE (n=10), Lake Erie 5 g ASE (n=5), and Lake Erie Soxhlet (n=10). The SD was less for Hamilton Harbour Soxhlet (7.6%) and Lake Erie 3 g ASE (2.8%). Some of this error is common to both methods (ie. GC error, sample weighing). However, the ASE method may be especially sensitive to small variations in the compactness of the sediment sample or solvent flow rates. There is a possibility that further investigation in these areas may reduce the error associated with ASE.

The procedural blanks (Table 4) indicate that employing the ASE method may result in cleaner blanks. False positive peaks may lead to overestimation of some PCB peaks. ASE might be expected to have cleaner blanks since extraction times are 100 times faster, there is little exposure to lab air, less glassware is used and much less solvent is used. Sources of contamination that are novel to the ASE method may result from the transfer of contaminants from sample to sample, or, contamination contributed by the sparging gas.

The kinetics of the ASE method are rapid. In these experiments, most of the PCBs are removed from the sediment within 10 to 20 mLs of extract, roughly 10 minutes. It appears that larger sediment loads may require more extraction solvent to achieve exhaustive extractions since the kinetics are slower. This hypothesis is supported by results in figure 1 that show slower rates of extraction with higher sediment loads. These observations are reinforced by the results in figure 2 that shows a decrease in the extraction efficiency when larger sample loads are used.

Slow extraction kinetics may be a result of the wide bore of the large volume extraction vessel that was used. This may cause non-linear flow of the extraction solvent and indirect removal of the analytes. However, when the narrow-bore low-volume extraction vessels are used, recovery of analytes are typically poor (data not shown). Anecdotal evidence suggests that small sample loads may extract more quickly since (1) analytes have to travel less further to leave the extraction vessel and (2) because the ratio of solvent to sediment is higher for extractions with smaller sediment loads.

In spite of the preliminary nature of this study, the results are very encouraging. The recovery of native PCBs from Hamilton Harbour sediments was not significantly different for the two methods tested. Lake Erie sediments demonstrated similar results for both methods. However, the recovery of native PCBs from Lake Erie significantly improved when the sample weight was reduced.

Conclusions

(1) The ASE extraction method performed at least as well as the traditional Soxhlet method with a more than a 25 fold reduction in both time and solvent required.

(2) Further research in the area of sample size and solvent flow rates and volumes is needed to optimize the ASE extraction method.

It is suspected that small modifications in the ASE method might further increase extraction efficiency. As observed in this study, decreasing the sample load resulted in increased Lake Erie PCB recovery by 36% with respect to Soxhlet, and, by 41% with respect to 5 g Lake Erie ASE. Other likely areas for investigation include: bulking the sediment with an inert filler such as Celite, solvent composition, extraction vessel dimensions, duration of static and dynamic extraction phases, and optimization of temperature and pressure. In view of the practical, economic, and environmental benefits associated with using the ASE extraction method, further investigation appears to be worthwhile.

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