



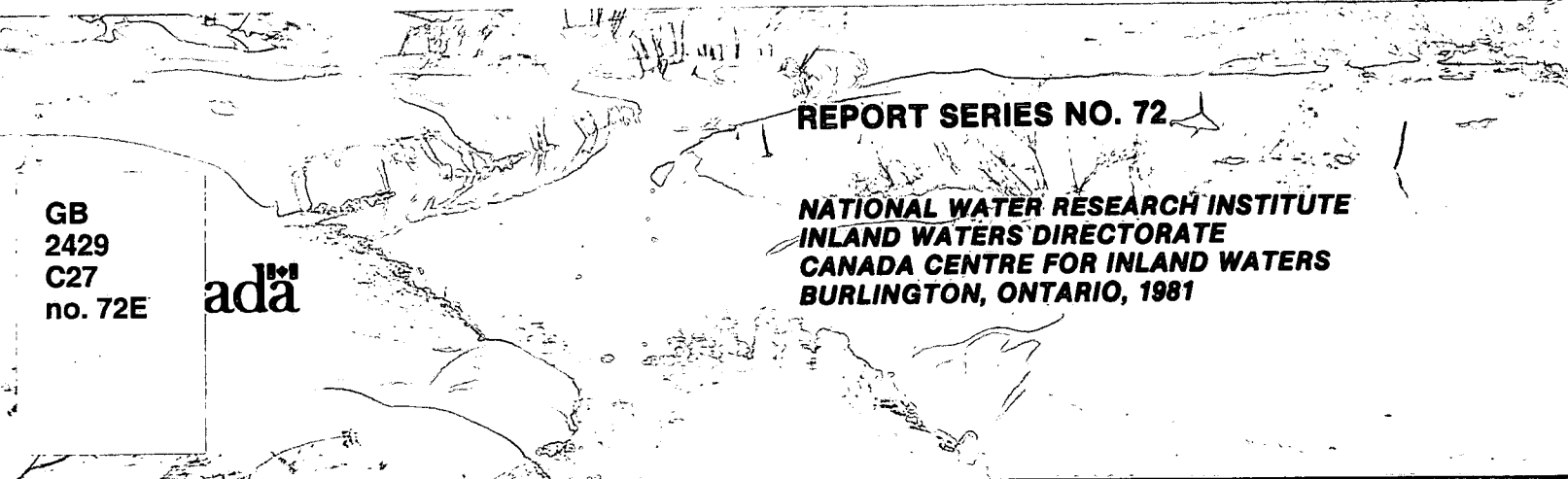
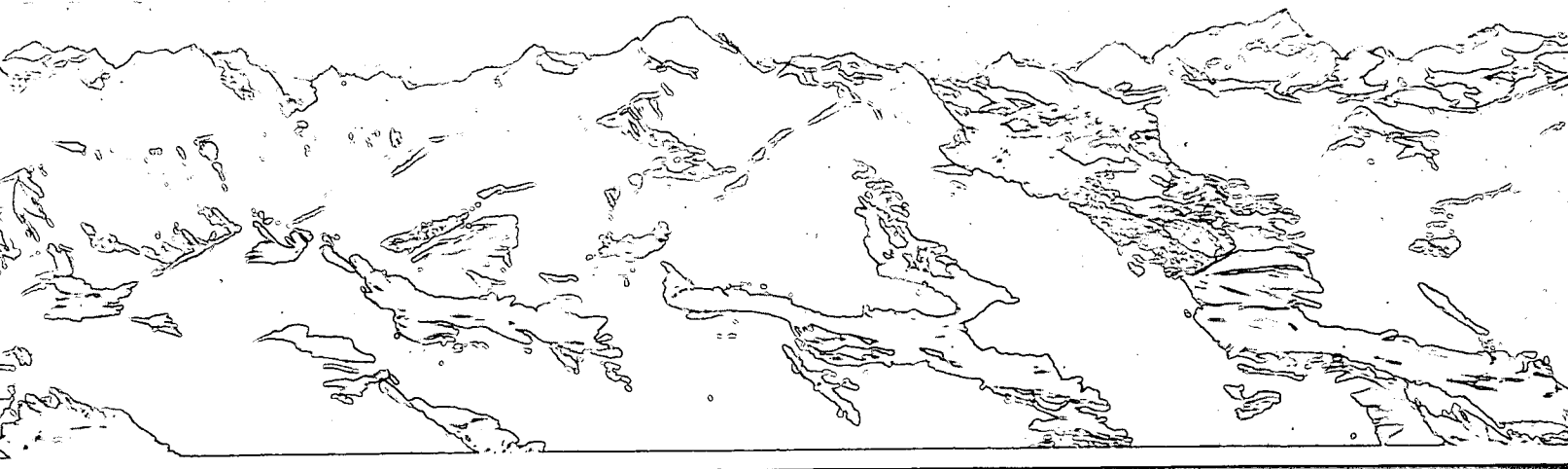
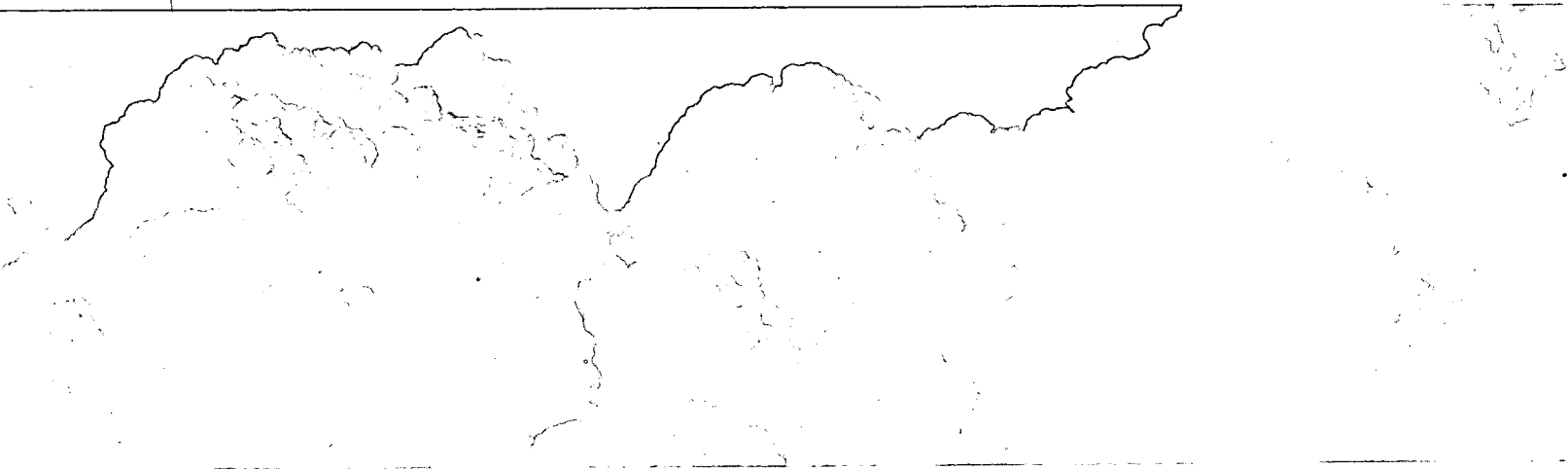
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# National Interlaboratory Quality Control Study No. 27

## PCBs In Naturally Contaminated Dry Sediments

H.B. Lee and A.S.Y. Chau



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NATIONAL WATER RESEARCH INSTITUTE  
INLAND WATERS DIRECTORATE  
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## **Abstract**

This report describes an interlaboratory study on the analysis of polychlorinated biphenyls (PCBs) in naturally contaminated dry sediment reference materials and in isooctane solutions. The quality of data submitted by the participants varied widely. Among the more comparable laboratories, the average interlaboratory coefficient of variation (C.V.) of the sediment samples is 29.0%. This result is significantly worse than the interlaboratory C.V. (16.2%) on sediment samples spiked with PCBs, as reported in interlaboratory quality control study No. 25. The less reliable PCB results, as found on real sediment samples, are likely due to problems in recovery, interference and quantitation of PCB mixtures.

## **Résumé**

Le présent rapport décrit une étude interlaboratoires du dosage des biphényles polychlorés (BPC) dans des échantillons de référence constitués de sédiments naturels pollués asséchés et dans des solutions d'isooctane. Les résultats ont varié fortement d'un laboratoire à l'autre. Parmi les laboratoires le plus comparables, le coefficient de variation (C.V.) moyen est de 29,0 %, ce qui est beaucoup plus grand que celui (16,2 %) de l'Étude interlaboratoires nationale de contrôle de la qualité n° 25, qui portait sur l'analyse de sédiments contenant des ajouts de BPC. Les résultats moins fiables de la présente étude découlent probablement des interférences et des difficultés de récupération et de dosage de mélanges de BPC.

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## PCBs in Naturally Contaminated Dry Sediments

H.B. Lee and A.S.Y. Chau

### INTRODUCTION

Two types of sediment samples are currently used in interlaboratory quality control programs involving PCBs: naturally contaminated samples and spiked samples. Natural samples are believed to be better for quality control purposes because they reflect the analytical performance of the laboratories in "real life" situations. The difficulties of including natural samples in quality control studies are:

- (1) the true pollutant level is not known, so that the accuracy of the analysis cannot be ascertained,
- (2) in many cases, the samples are not well characterized in terms of homogeneity and stability, and
- (3) the samples are not in sufficient quantities for long-term quality control applications.

Recently, Chau and Lee completed a report on the preparation of large quantities of homogenized dry sediments naturally contaminated with PCBs (Chau and Lee, 1980). The homogeneity of nearly 5000 subsamples, each 25 g to 30 g of dry sediments, was confirmed by extensive (over 100 replicate determinations) analyses of the PCB content.

This report summarizes the results of the first quality control study using subsamples of these well-characterized natural sediment samples. Only with the results from this study can the quality of the PCB data generated by the participating laboratories be evaluated in real situations.

### EXPERIMENTAL DESIGN

The samples distributed in this study are listed in Table 1. Two freeze-dried natural sediment samples (internally coded as EC-1 and EC-2) were sent in blind duplicate. In this case, samples 1 and 4, which are subsamples of EC-1, are identical. Also identical are samples 2 and 3, which are subsamples of EC-2. In addition, two ampoules, each containing a PCB mixture suitable for direct GLC analysis, were also sent. Ampoule A was generated from a pooled extract (after all the usual cleanup procedures) collected during the in-house homogeneity test of the EC-1 subsamples. The mixture was in a 1:3

dilution, i.e., 1 g of sediment extracted diluted to 3 mL. Ampoule B was a 1:1:1 mixture of Aroclors 1242, 1254 and 1260, with a total concentration of 600 pg/ $\mu$ L. This experimental design was not disclosed to avoid bias. As well, no information regarding the Aroclor composition of the sediment samples and the ampoules was given to the participants, who were instructed to choose their own standards for quantitation.

Table 1. Samples Distributed for Interlaboratory Study No. 27 (PCBs in naturally contaminated dry sediments)

Sample number	Internal quality control code	Design level	Type of sample
1	EC-1	—	SRM (dry sediment)
2	EC-2	—	SRM (dry sediment)
3	EC-2	—	SRM (dry sediment)
4	EC-1	—	SRM (dry sediment)
A	—	1/3 EC-1	Sediment extract in iso-octane after cleanup
B	—	600 pg/ $\mu$ L	Mixture of Aroclors in iso-octane*

\*Ampoule B is a 1:1:1 mixture of Aroclors 1242, 1254 and 1260, with a total PCB concentration of 600 pg/ $\mu$ L.

The design of this study permits the following evaluation:

- (1) comparability of PCB standards on an inter-laboratory basis,
- (2) interlaboratory precision on sediment PCB analysis, and
- (3) recovery of PCBs from natural sediment samples.

Questionnaires and test samples were sent to 25 laboratories in April 1980 and most of the data were received by August 1980.

### SAMPLE PREPARATION AND CHARACTERIZATION

The bulk sediment used to prepare EC-1 sediment reference material was collected from a landfill site in

Hamilton Bay, Ontario. The sediment used to prepare the EC-2 reference material was a mixture of the Hamilton Bay sediment and a Lake Ontario sediment.

After collection, the bulk sediment was frozen at  $-20^{\circ}\text{C}$  for one week. It was then thawed at room temperature to remove excess water before this partially dry sediment was freeze-dried. The dry sediment was then crushed, screened and blended in a large blender before it was subsampled into small bottles. [For a detailed procedure, see Chau and Lee (1980)].

The homogeneity of the bulk sediment sample before subsampling was established by replicate ( $n = 5$ ) PCB analyses of six representative samples from the blender. The experimental results were then analyzed by a two-way analysis of variance technique (ANOVA). The homogeneity of the sediment subsamples was confirmed by PCB analysis on one random sample out of every 50 sediment subsamples, and the results were treated statistically. A total of 97 samples were analyzed in the latter case.

Both EC-1 and EC-2 were analyzed routinely to monitor any possible degradation during storage. No observable decomposition was found for either sediment reference material for a period of one year.

Qualitatively, both EC-1 and EC-2 were heavy clay sediments rich in humic substances. In-house analyses indicated that EC-1 was composed of PCB components closely resembling a 2:2:1 mixture of Aroclors 1248,

1254 and 1260. The sample EC-1 also contained detectable amounts of hexachlorobenzene and  $p,p'$ -DDE. On the other hand, EC-2 was a more complicated mixture of these Aroclors together with chlorinated benzenes,  $p,p'$ -DDE and Mirex.

## DATA EVALUATION AND DISCUSSION

Table 2 summarizes the raw data submitted by the participants. Table 3 provides the results of the direct GLC analysis of a mixture of PCB standards (ampoule B) in isooctane. Even under these ideal analytical conditions, the results from this sample have a wide range, from 240 to 1440  $\text{pg}/\mu\text{L}$ . After the rejection of outliers, the mean result is 572.3  $\text{pg}/\mu\text{L}$ , representing an average recovery of 95.4% compared with the design level of 600  $\text{pg}/\mu\text{L}$ . The interlaboratory results together with two previous studies (Lee and Chau, 1981; Aspila, 1980) confirmed the design levels of the standard solutions prepared by this laboratory, indicating that the standards among the participating laboratories are mostly comparable. The interlaboratory coefficient of variation (C.V.) on ampoule B is 12% and is similar to the value found for the same type of sample in a previous study (Lee and Chau, 1981). To obtain comparable results, it would be beneficial for the participants (laboratories Nos. 43, 69 and 104) whose reported results on ampoule B were over 30% different from either the design level or the interlaboratory mean to re-check their in-house PCB standards to confirm their labelled concentrations.

Table 2. Results of Interlaboratory Study No. 27

Laboratory number	Sample 1 ( $\mu\text{g}/\text{g}$ )	Sample 2 ( $\mu\text{g}/\text{g}$ )	Sample 3 ( $\mu\text{g}/\text{g}$ )	Sample 4 ( $\mu\text{g}/\text{g}$ )	Ampoule A ( $\text{pg}/\mu\text{L}$ )	Ampoule B ( $\text{pg}/\mu\text{L}$ )
1	0.652* 1.956†	0.535* 1.605†	0.468* 1.404†	0.803* 2.409†	303* 909†	203* 609†
2	0.25* 1.55‡	0.33* 0.86‡	0.45* 0.71‡	0.33* 1.49‡	740* —	590* —
4	2.4	1.4	1.6	3.0	1035	720
8	1.93	3.35	3.26	1.99	864	534
10	1.4	1.5	1.3	1.7	780	530
14	1.63	1.07	1.06	1.71	667	514
17	2.19	1.43	1.46	2.16	712	619
31	3.408	3.784	1.470	3.158	706	494
43	0.04	0.22	0.02	0.10	290	240
44	2.28	0.647	0.674	2.47	768	473
53	0.560	1.443	1.407	1.736	762	605
69	1.85	1.45	2.01	1.59	1700	1440
71	0.96	0.94	0.76	1.28	990	620
104	0.337	0.146	0.150	0.326	301	300
105	3.01	1.25	1.17	2.09	553	559

\*Results originally submitted.

†Revised results after a calculation error was found.

‡Revised results after a second set of sediment samples was reanalyzed.

**Table 3. Percent Recovery of PCBs in Isooctane (ampoule B) by Direct GLC Analysis**

Laboratory number	PCB content reported (pg/ $\mu$ L)	Percent recovery
1	609	101.5
2	590	98.3
4	720	120.0
8	534	89.0
10	530	88.3
14	514	85.7
17	619	103.2
31	494	82.3
43	240 R	40.0 R
44	473	78.8
53	605	100.9
69	1440 R	240.0 R
71	620	103.3
104	300 R	50.0 R
105	559	93.2
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Design level (pg/ $\mu$ L)	600	
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Range (pg/ $\mu$ L)	240-1440	40.0-240.0
Mean* (pg/ $\mu$ L) (n = 12)	572.3	95.4
S.D. (pg/ $\mu$ L)	68.6	11.4
C.V. (%)	12.0	12.0

\*Values calculated after rejection of outliers (R).

Table 4 summarizes the ranking of the sediment results. For each sediment sample, rank 1 is given to the laboratory that provided the highest result, rank 2 to the next highest, and so on. The total rank of each laboratory on the four sediment samples is given in the last column of Table 4. The permissible score limits for 15 laboratories and four materials are 9 and 55 (Youden and Steiner, 1975). For laboratories Nos. 31, 43 and 104 whose scores are out of this limit, it is concluded that in each laboratory,

a pronounced systematic error is present. Laboratory No. 31, which has a score of 7, shows the consequence of getting high results consistently. On the other hand, laboratories Nos. 43 and 104 with scores of 59 and 57, respectively, are getting low results consistently. On this basis, most of the sediment results submitted by these three laboratories are rejected in the statistical analysis.

Tables 5 and 6 summarize the results on the duplicate analyses of sediment reference materials EC-1 and EC-2, respectively. A quick inspection of the sediment data reported by the participants shows that the results vary widely. For example, results on EC-1 have a range from 0.04 to 3.408  $\mu$ g/g and those for EC-2, a range from 0.02 to 3.784  $\mu$ g/g. After the rejection of outliers, there is a range of approximately three to three and one-half fold for those sediment results. The interlaboratory mean (after rejection) for EC-1 is 2.054  $\mu$ g/g (n = 25) with an interlaboratory C.V. of 29.8%, while the interlaboratory mean for EC-2 is 1.244  $\mu$ g/g (n = 23) with an interlaboratory C.V. of 28.2%. It is noted that the interlaboratory precision on natural PCB sediment samples is worse than that for spiked PCB sediment samples, as found in interlaboratory quality control study No. 25 (Lee and Chau, 1981). The poorer interlaboratory precision on the natural samples is probably due to the following reasons:

- (1) The naturally contaminated sediment samples distributed in this study have more complicated and unknown PCB compositions. By comparing the results on ampoules A and B that involve quantitation only (see Table 7 and later discussions), it is demonstrated that the results are less precise on the natural samples than on the PCB standards.

**Table 4. Ranking of Sediment Results**

Laboratory number	Sample 1	Sample 2	Sample 3	Sample 4	Total rank
1	6	3	7	4	20
2	10	12	12	12	46
4	3	8	3	2	16
8	7	2	1	7	17
10	11	4	8	10	33
14	9	10	10	9	38
17	5	7	5	5	22
31	1	1	4	1	7
43	15	14	15	15	59
44	4	13	13	3	33
53	13	6	6	8	33
69	8	5	2	11	26
71	12	11	11	13	47
104	14	15	14	14	57
105	2	9	9	6	26

Table 5. Results from Duplicate Analysis of EC-1 (sediment samples 1 and 4)

Laboratory Number	Results reported ( $\mu\text{g/g}$ )	Intralaboratory mean $\pm$ S.D. ( $\mu\text{g/g}$ )	Intralaboratory C.V. (%)
1	1.956, 2.409	2.183 $\pm$ 0.320	14.7
2	1.55, 1.49	1.52 $\pm$ 0.042	2.8
4	2.4, 3.0	2.7 $\pm$ 0.424	15.7
8	1.93, 1.99	1.96 $\pm$ 0.042	2.2
10	1.4, 1.7	1.55 $\pm$ 0.212	13.7
14	1.63, 1.71	1.67 $\pm$ 0.057	3.4
17	2.19, 2.16	2.175 $\pm$ 0.021	1.0
31	3.408, 3.158	3.283 $\pm$ 0.177	5.4
43	0.04 R, 0.10 R	0.07 $\pm$ 0.042	60.6
44	2.28, 2.47	2.375 $\pm$ 0.134	5.7
53	0.56 R, 1.736	1.148 $\pm$ 0.832	72.4
69	1.85, 1.59	1.72 $\pm$ 0.084	10.7
71	0.96, 1.28	1.12 $\pm$ 0.226	20.2
104	0.337 R, 0.326 R	0.332 $\pm$ 0.008	2.4
105	3.01, 2.09	2.55 $\pm$ 0.051	25.5
Range ( $\mu\text{g/g}$ )		0.04-3.408	
Interlaboratory mean* ( $\mu\text{g/g}$ ) (n = 25)		2.054	
Interlaboratory S.D. ( $\mu\text{g/g}$ )		0.611	
Interlaboratory C.V. (%)		29.8	

\*Values calculated after rejection of outliers (R).

Table 6. Results from Duplicate Analysis of EC-2 (sediment samples 2 and 3)

Laboratory number	Results reported ( $\mu\text{g/g}$ )	Intralaboratory mean $\pm$ S.D. ( $\mu\text{g/g}$ )	Intralaboratory C.V. (%)
1	1.605, 1.404	1.505 $\pm$ 0.142	9.5
2	0.86, 0.71	0.785 $\pm$ 0.106	13.5
4	1.4, 1.6	1.5 $\pm$ 0.141	9.4
8	3.35 R, 3.26 R	3.305 $\pm$ 0.064	1.9
10	1.5, 1.3	1.4 $\pm$ 0.141	10.1
14	1.07, 1.06	1.065 $\pm$ 0.007	0.7
17	1.43, 1.46	1.445 $\pm$ 0.021	1.5
31	3.784 R, 1.470	2.627 $\pm$ 1.636	62.3
43	0.22 R, 0.02 R	0.12 $\pm$ 0.141	118.0
44	0.647, 0.674	0.661 $\pm$ 0.019	2.9
53	1.443, 1.407	1.425 $\pm$ 0.026	1.8
69	1.45, 2.01	1.73 $\pm$ 0.396	22.9
71	0.94, 0.76	0.85 $\pm$ 0.127	15.0
104	0.146 R, 0.150 R	0.148 $\pm$ 0.003	1.9
105	1.25, 1.17	1.21 $\pm$ 0.057	4.7
Range ( $\mu\text{g/g}$ )		0.02-3.784	
Interlaboratory mean* ( $\mu\text{g/g}$ ) (n = 23)		1.244	
Interlaboratory S.D. ( $\mu\text{g/g}$ )		0.351	
Interlaboratory C.V. (%)		28.2	

\*Values calculated after rejection of outliers (R).

- (2) The recovery of PCBs from natural sediment samples varies widely from highs of over 150% to lows of ca. 35% (see Table 7 and later discussions).
- (3) Interferences by sulphur and sulphur compounds, chlorinated benzenes and a few organochlorinated pesticides in the sediments affect the PCB results to various degrees.

Sulphur and/or organosulphur interference in both EC-1 and EC-2 caused problems for some participants. In several cases, sulphur was incompletely removed from the sample extract, and this may have affected the quantitative results in two ways. For those who recognized the interference problem and chose to disregard the PCB component peaks under the off-scale sulphur peak, the results would be biased low. For those who treated the



interference peak as a PCB component, the results would be biased high. Apart from sulphur, only small amounts of hexachlorobenzene and p,p'-DDE are present in the PCB fraction of EC-1 and they would not significantly affect the results even if they had been misidentified as PCB components. However, in-house analyses of EC-2 indicated that this sediment reference material contains a number of chlorinated benzenes which appear in the early part of the chromatogram. Their identities were confirmed by GLC on both packed and capillary columns of widely different polarities (e.g. SP-2100 and Carbowax 20M). In at least one case (laboratory No. 8), all or some of these early interference peaks were treated as PCB components and the results on EC-2 were biased high. (In its report, this laboratory had indicated that problems were experienced with the quantitation of EC-2 and that the initial part of the chromatogram did not correspond to its in-house PCB standards.)

Intralaboratory precision on sediment PCB analyses was also calculated on the basis of the results on the duplicate analysis of EC-1 and EC-2. Because of the limited number of replicates, these numbers should only be used semiquantitatively. As expected, the intralaboratory precision is in general much better than interlaboratory precision. In a few cases (laboratories Nos. 31, 53 and 105) in which one of the results from duplicate analysis of either EC-1 or EC-2 was substantially different from the other, the intralaboratory precision is rather low. The results from duplicate analysis of EC-1 and EC-2 are plotted in Figures 1 and 2, respectively. The laboratories whose results lie on or very close to the diagonal lines represent excellent to very good intralaboratory precision. The laboratories with results farther away from the lines indicate the presence of random errors or imprecision on the duplicate analyses. The farther away the results from the lines, the less the in-house precision.

Table 7 lists the calculated percent recoveries on sediment reference material EC-1. Also included are the mean value and interlaboratory precision on ampoule A. As mentioned previously, the interlaboratory C.V. on ampoule A (17.4%) is significantly larger than that on ampoule B (12.0%). This indicates that the participating laboratories are less precise in quantitating natural samples than PCB standards. Since ampoule A is a 1:3 diluted extract of EC-1, the ratio of the results on the intralaboratory mean of EC-1 and ampoule A has a design value of 3. The percent recovery of EC-1 can then be obtained by dividing this ratio by 3 in each case. Since the percent recovery is corrected for any potential intralaboratory bias on the quantitation step, this number is the most rigorous basis of a laboratory's true recovery on the real sample. With the exception of one outlying laboratory,

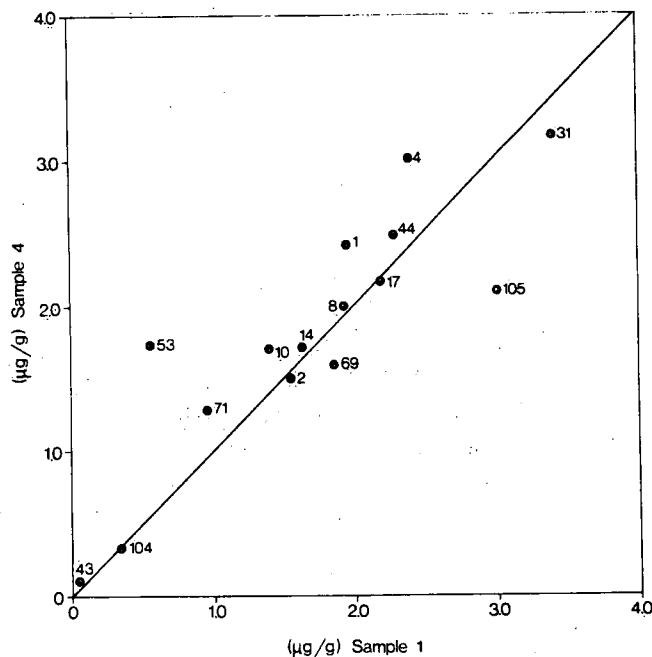


Figure 1. Paired sample plot of duplicate analysis of EC-1.

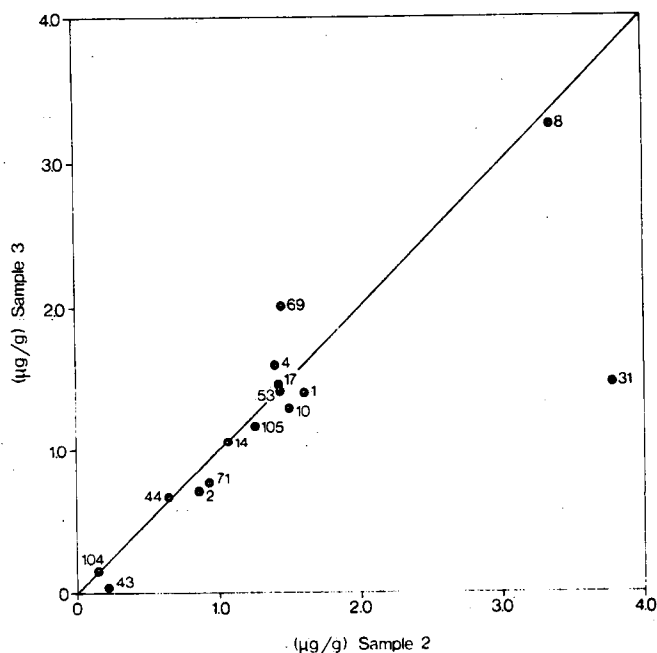


Figure 2. Paired sample plot of duplicate analysis of EC-2.

the mean recovery of EC-1 is 80.9% with an extremely high interlaboratory C.V. of 47.4%. In two cases (laboratories Nos. 31 and 105), recoveries were as high as +150%, while in four other cases (laboratories Nos. 53, 69, 71 and 104) recoveries were 50% or lower.

Table 7. Percent Recovery of Sediment Sample EC-1

Laboratory number	Intralaboratory mean of EC-1 (ppm)	Ampoule A (ppm)	Ratio	Percent recovery
1	2.183	0.909	2.40	80.0
2	1.52	0.740	2.05	68.3
4	2.7	1.035	2.61	87.0
8	1.96	0.864	2.27	75.7
10	1.55	0.780	1.99	66.3
14	1.67	0.667	2.50	83.3
17	2.175	0.712	3.05	101.7
31	3.283	0.706	4.65	155.0
43	0.07	0.290 R	0.24	8.0 R
44	2.375	0.768	3.09	103.0
53	1.148	0.762	1.51	50.3
69	1.72	1.700 R	1.01	33.7
71	1.12	0.990	1.13	37.7
104	0.332	0.301 R	1.10	36.7
105	2.55	0.553	4.61	153.7
Design level			3.00	
Range		0.290-1.700		8.0-155.0
Mean* (n = 12)		0.791 (n = 12)		80.9 (n = 14)
S.D.		0.127		38.4
C.V. (%)		17.4		47.4

\* Values calculated after rejection of outliers (R).

Table 8. Analytical Methodology (extraction and cleanup)

Laboratory number	Amount of water	Extraction	Solvent system	Cleanup
1	30%	Ultrasonic	100 mL, acetone/hexane (three times)	c,h
2	37%	Ultrasonic	100 mL, 1:1 acetone/hexane (three times)	a,b,d
4	Unspecified	Shaker	Acetonitrile	a,b,d
8	Unspecified	Homogenizer	300 mL, acetonitrile/water	a,d
10	Nil	Soxhlet	130 mL, 9:1 acetone/hexane	a,d,i,j
14	33%	Shaker	300 mL, acetone/hexane	b,d
17	2 mL	Ultrasonic	100 mL, 1:1 acetone/hexane (three times)	a,b,d
31	Nil	Shaker	20 mL, hexane	d,g
43	10%	Soxhlet	150 mL, 1:1 acetone/hexane	b,d
44	Nil	Ultrasonic	10 mL, acetone (once)	g
53	29%	Soxhlet	150 mL, 1:1 acetone/hexane (once)	b,d
69	Nil	Stirrer	50 mL, acetonitrile (twice)	a,b,d
71	25%	Shaker	10 mL/g, 1:1 acetone/hexane	a,c,d,e
104	Nil	Ultrasonic	130 mL, 1:1 acetone/hexane	a,d
105	25%	Blender ultrasonic bath	100 mL, 1:1 methanol/dichloromethane (twice)	a,d,f

Cleanup methods: a - Liquid and liquid partitioning  
 b - Mercury  
 c - Activated copper  
 d - Florisil  
 e - Charcoal column  
 f - Silver nitrate on alumina  
 g - Gel permeation chromatography  
 h - Silica gel  
 i - Chromium trioxide/acetic acid  
 j - Potassium hydroxide/ethanol

Tables 8 and 9 list the analytical methodologies of the participants. As in study No. 25, the methods used by the participants all differ greatly. Because of the number

of variables involved in extraction, cleanup and quantitation, no attempt has been made to correlate the results and the analytical methodologies.

Table 9. Analytical Methodology (instrumentation and quantitation)

Laboratory number	Gas chromatograph	GLC column and temperature	Quantitation method	Sample injection	Standard
1	Tracor 220	3% OV-101 and 1.5% OV-17 + 1.95% QF-1, 200° C	Webb-McCall	Automatic	a
2	Tracor 222	3% OV-101, 195° C	Webb-McCall	Manual	b
4	HP 5840	5% SP-2100, 145-230° C	Total area of minimum 5 peaks	Manual	b
8	HP 5739	4% SE-30 + 6% OV-210, 200° C	Webb-McCall	Manual	c
10	HP 5700	3% SP-2100, 190° C	Webb-McCall	Automatic	b
14	Tracor MT 222	3% OV-101, 210° C	Webb-McCall	Manual	a
17	HP 5713	3% OV-101, 192° C	Webb-McCall	Automatic	a
31	Tracor 220	3% SP-2100, 200° C	Total area	Manual	d
43	HP 5710	SP-2100 cap column	No details	Manual	a,e
44	HP 5700	2% SE-30 + 4% QF-1, 210° C	Total peak height	Manual	b,f
53	Tracor 560	3% OV-1, 200° C	Total peak height	Manual	a
69	HP 5713	3% OV-17, 220° C	Webb-McCall	Automatic	g
71	Tracor 222	1.5% OV-17 + 2.0% OV-210, 180° C	Peak height of 3 selected peaks	Manual	b,f
104	HP 7520	1.5% OV-17 + 1.95% OV-210, 188° C	Area of 4 selected peaks	Manual	e
105	Tracor 550	2% OV-101 + 3% QF-1, 185° C	Total area	Manual	a

Standard used in quantitation: a - 1:1:1 or other combinations of Aroclors 1242, 1254 and 1260  
 b - 1:1:1 or other combinations of Aroclors 1248, 1254 and 1260  
 c - Individual Aroclors 1221, 1242, 1254 and 1260  
 d - Aroclor 1260  
 e - 1:1 mixture of Aroclors 1254 and 1260  
 f - 1:1 mixture of Aroclors 1248 and 1254  
 g - Individual Aroclors 1242, 1254 and 1260

### CONCLUSIONS

The quality of laboratory performance on PCB analysis varies widely in this study. Among the more comparable laboratories, the interlaboratory precision on naturally contaminated sediment samples is ca. 30%. This value is significantly higher (worse) than the interlaboratory precision observed in study No. 25 for spiked sediment samples, indicating that the laboratory results are in general less reliable on a real sample. The mean recovery of PCBs in natural sediment samples is ca. 80% with a disappointingly large interlaboratory standard deviation of ca. 40%. Apart from the recovery, quantitation of PCB mixtures, especially the natural sample extracts, remains to be a major source of error in PCB analysis.

### RECOMMENDATIONS

The periodic use of sediment reference materials such as EC-1 and EC-2 in in-house and interlaboratory quality control studies should prove to be beneficial in data quality on a long-term basis. The inclusion of these natural samples eliminates the danger of extrapolating the results based on spiked quality control samples to the quality of results of real life samples.

### ACKNOWLEDGMENTS

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## APPENDIX

### LIST OF PARTICIPANTS

#### *Environment Canada, Environmental Conservation Service*

Atlantic Region, Organic Analysis Laboratory,  
Moncton, New Brunswick

Ontario Region, Organic Analysis Laboratory,  
Burlington, Ontario

Pacific Region, Water Quality Branch Laboratory,  
Vancouver, British Columbia

Western Region, Water Quality Branch Laboratory,  
Calgary, Alberta

#### *Environment Canada, Environmental Protection Service*

Atlantic Region, Air and Water Branch, Halifax,  
Nova Scotia

#### *Fisheries and Oceans Canada*

Freshwater Institute, Analytical Chemistry Group,  
Winnipeg, Manitoba

Freshwater Institute, Industry Services Branch,  
Winnipeg, Manitoba

#### *Provincial Laboratories*

Agriculture Québec, Laboratoire d'analyses et  
d'expertises alimentaires, Ste-Foy

Alberta Department of Agriculture, Food Laboratory  
Services Branch, Edmonton

Alberta Environment, Pollution Control Laboratory,  
Edmonton

Manitoba Department of Mines, Resources and  
Environmental Management, Winnipeg

Ontario Ministry of Agriculture and Food, Provincial  
Pesticide Residue Testing Laboratory, Guelph

#### *Industrial Consulting Laboratories*

B.C. Research, Vancouver, British Columbia

Enviroclean Ltd., London, Ontario

Nova Labs, Lachine, Quebec

Environment Canada Library, Burlington



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