

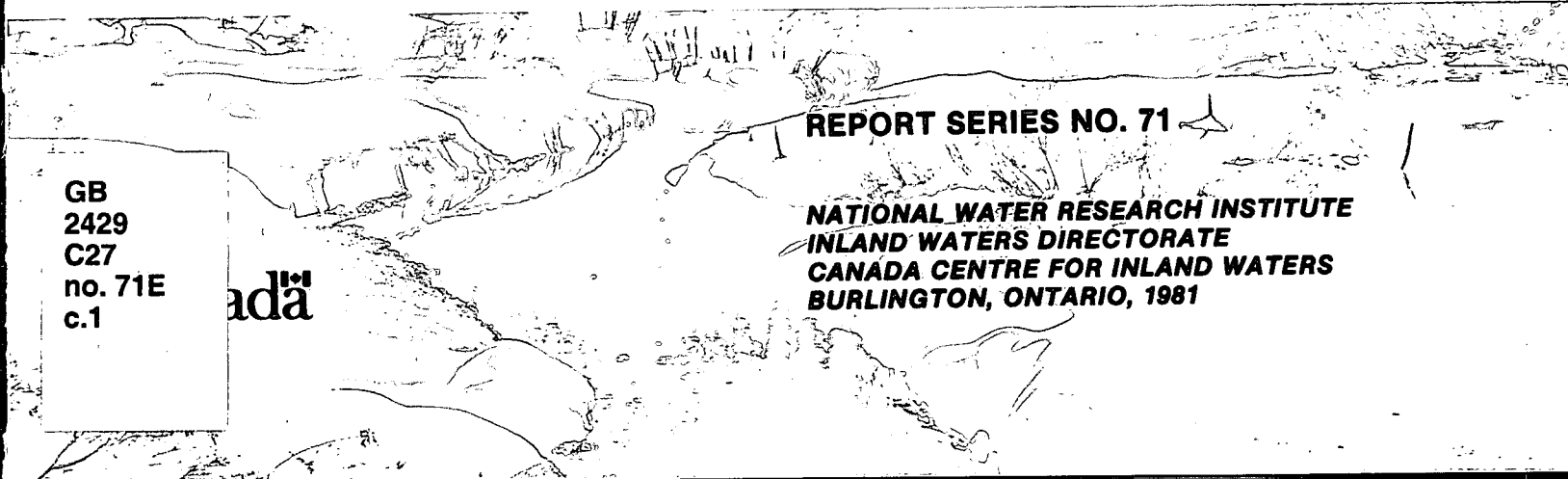
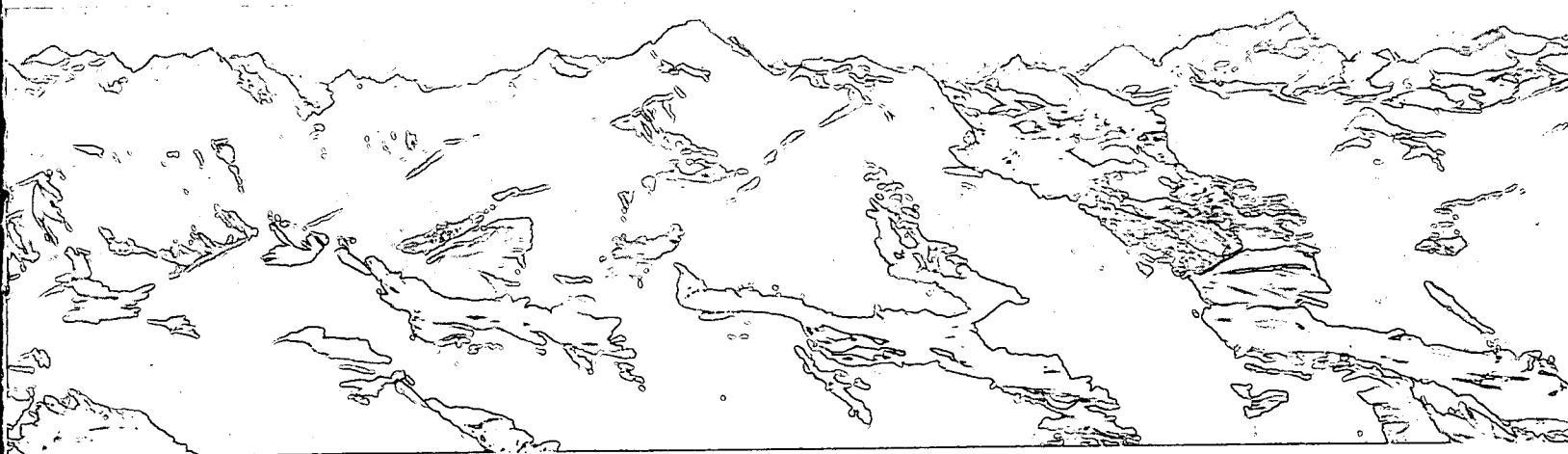
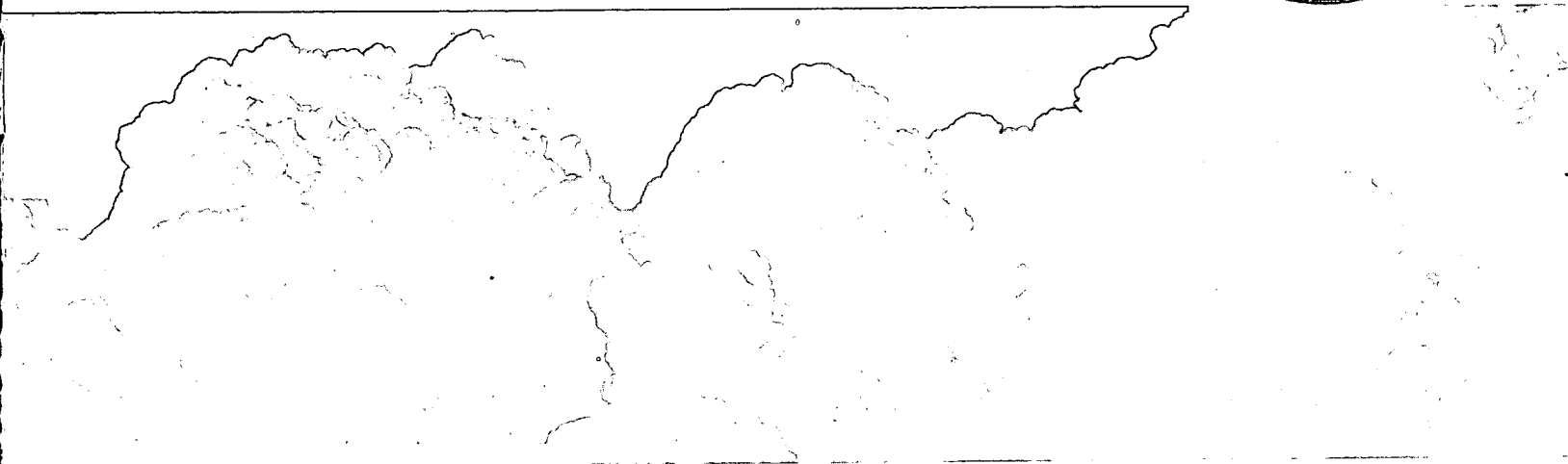


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# National Interlaboratory Quality Control Study No. 25 PCBs in Wet Sediments

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



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NATIONAL WATER RESEARCH INSTITUTE  
INLAND WATERS DIRECTORATE  
CANADA CENTRE FOR INLAND WATERS  
BURLINGTON, ONTARIO, 1981



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## Abstract

This study describes an interlaboratory comparison of the analysis of polychlorinated biphenyls (PCBs) (a 1:1 mixture of Aroclors 1254 and 1260) at concentrations of about 1 ppm in 10 to 20 g spiked wet sediment. The average coefficient of variation (16.2%) of the present study is very similar to previously reported results in other interlaboratory PCB studies at comparable levels. The variation of results among laboratories in this study appears to be due to the variation in quantitation methods more than to the variation in extraction and cleanup procedures. The laboratories that used the Webb-McCall quantitation procedure gave better accuracy and precision than those that used other quantitation procedures.

## Résumé

Dans la présente étude, on compare les résultats du dosage par différents laboratoires de concentrations d'environ 1 mg/kg de biphényles polychlorés (BPC) (un mélange 1:1 d'Aroclor 1254 et d'Aroclor 1250) dans des échantillons de 10 à 20 g de sédiments humides. Le coefficient de variation moyen (16,2 %) est très rapproché de ceux d'études antérieures semblables, pour des valeurs du même ordre. On pense que la variation interlaboratoires résultant de cette étude s'explique par les méthodes de dosage utilisées plutôt que par les procédés d'extraction et de purification. La méthode de dosage Webb-McCall a donné des résultats plus justes et plus précis que les autres méthodes utilisées.

# National Interlaboratory Quality Control Study No. 25

## PCBs in Wet Sediments

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

### INTRODUCTION

Analysis of xenobiotic organics such as PCBs (polychlorinated biphenyls) in sediment is an important activity in environmental studies. Although for many years such analysis has been carried out for international and national studies by many agencies, there have been no quality control studies using well-characterized wet sediment samples. Previous quality control studies were limited to standard solutions, solutions of sample extracts (spiked or naturally contaminated) (Aspila and Carron, 1977, 1978) or individually spiked sediment samples. The use of solutions, although suitable for certain purposes, does not reflect the "real world" situation, and in the spiking of individual sediment samples, there is always a possibility that a mechanical spiking error could exist. In addition, the organics are not at all integrated to the sediment matrix.

Ideally, for spiked or naturally contaminated sediment samples, a bulk sample is homogenized and subsamples are analyzed to check the designed or "true" value before being sent to the participants. The homogeneity of the bulk sample, however, must first be established to ensure equivalence of all subsamples. Until recently, there has been no well-documented procedure for doing this. The recent investigation of Chau *et al.* (1979) enabled the preparation of homogeneous wet sediment spiked with a PCB solution. Thus the use of spiked wet sediment is a step closer to the real life situation. It can also be used to compensate for the levels of PCBs that are difficult to find in naturally contaminated sediments.

This report summarizes the results of the first quality control study which used subsamples from a bulk wet sediment spiked with PCBs. The basic objective of this study was to determine whether or not the participating laboratories across Canada would produce comparable results in the analysis of PCBs in sediment samples on an interlaboratory basis.

A total of 33 sets of samples were distributed to laboratories that earlier had indicated a willingness to participate in the study.

### EXPERIMENTAL DESIGN

The samples distributed in this study are listed in Table 1. The Youden (Youden and Steiner, 1975) paired sample design including two sediment samples fortified to slightly different PCB levels was adopted because of its simplicity and the possibility of revealing any existing systematic and random errors. However, data from a Youden technique cannot ascribe a specific source of systematic error observed. The preferred design would consist of a series of six to eight samples which cover the entire concentration range of PCBs of interest. This option was not taken because it would have required two to three times more man-hours to prepare, distribute and analyze the samples. A blank sediment sample was also included to check any possible error due to blank determination or contamination during analysis. To monitor errors purely related to quantitation and standard solutions, a 1:1 mixture of Aroclors 1254 and 1260 in isooctane was also included. The questionnaire distributed was designed to detail the interlaboratory variability in analytical methodology.

Table 1. Samples Distributed for Interlaboratory Study No. 25 (PCBs in wet sediment)

Sample number	Total PCB content*	Type of sample
1	x ppm†	Blank wet sediment
2	x + 1.01 ppm	Spiked wet sediment
3	x + 1.22 ppm	Spiked wet sediment
4	1.00 ng/μL	Known standard in isooctane

\* PCB content expressed as a 1:1 mixture of Aroclors 1254 and 1260.  
† Original sediment used to prepare samples 1, 2 and 3 was obtained from Lake Ontario. A value of 0.02 ppm for x has been used for the calculation of sediment recovery data.

### SAMPLE PREPARATION AND INTEGRITY

All sediment samples were prepared three to four weeks before distribution. The blank sediment, which was

obtained from western Lake Ontario, was analyzed and found to contain very low concentrations of PCBs and organochlorinated insecticides. Every sediment sample was a subsample, each about 10 to 20 g, from a large homogenized bulk sample prepared according to Chau *et al.* (1979). A bulk sediment sample was accurately weighed (to the nearest gram) into a large stainless steel blender and the sample was blended for 3 min. If spiking was necessary, a known amount of PCB standard in acetone was added to the sediment and blending was continued for another 60 min. Immediately after blending, the homogenized sediment slurry was poured into 1-oz Wheaton bottles as quickly as possible.

After preparation, the tightly capped sediment samples were immediately frozen to  $-20^{\circ}\text{C}$  until distribution or in-house testing. The homogeneity and design level among the sediment subsamples were confirmed by the analysis of PCB content. The results of water content determination of the subsamples further substantiated sample homogeneity. Typical in-house coefficients of variation for replicate analysis of PCBs ( $n = 6$ ) in these wet sediment subsamples were between 3.5% and 5.5% and those for water content determinations, between 0.5% and 1.0%. Recoveries of PCBs in these samples ranged from 91% to 105% with a mean of 98%.

Since the bulk sample rather than individual subsamples was spiked and subsequently homogenized, there is no possibility of a mechanical spiking error or nonhomogeneous sediment blank from subsample to subsample. An in-house stability test of the frozen subsamples (Chau *et al.*, 1979) indicated that no observable degradation of PCB components occurred in the sediment during a three-month period. Also, samples left at room temperature were stable for at least four weeks. Therefore, no change in PCB compositions in the sediment samples during storage and transportation was expected.

## EVALUATION OF DATA

Results obtained from the participants are given in Table 2. Table 3 lists the percent recoveries of PCBs in samples 2, 3 and 4. Tables 4 and 5 summarize the analytical methodology employed by the participants. A paired sample plot for samples 2 and 3 is depicted in Figure 1. The line in Figure 1 is a  $45^{\circ}$  line passing through the design levels of the spiked sediment samples. If vertical lines were drawn from the point to the  $45^{\circ}$  line, the lengths of these vertical lines would be directly related to random errors. The lines would intersect the  $45^{\circ}$  line at various distances from the point through which the  $45^{\circ}$  line was

drawn. These distances are directly related to the systematic errors of the laboratories.

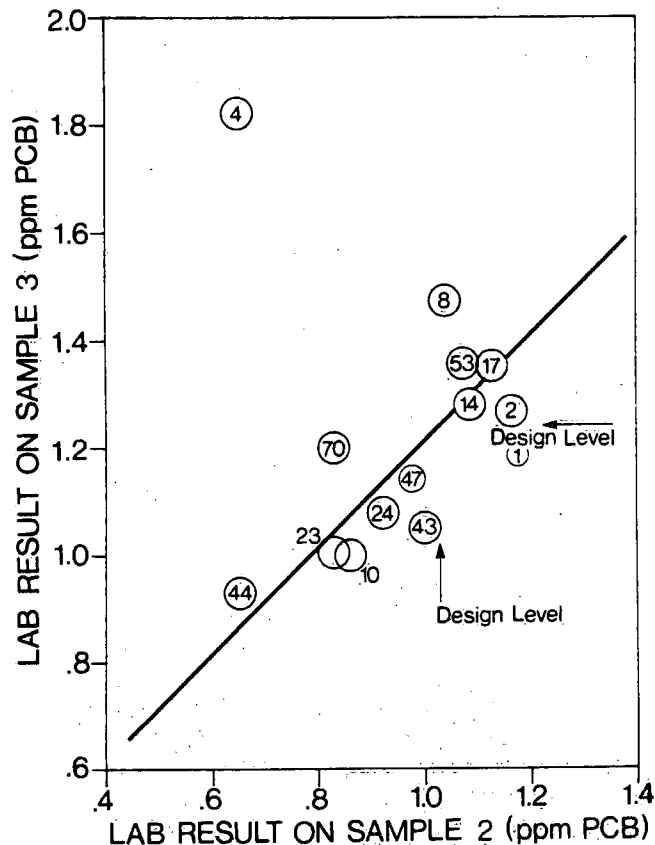


Figure 1. Paired sample plot for samples 2 and 3.

All of the laboratories reported that sample 1 had a blank PCB content of not more than 0.03 ppm. Many expressed the value in less than 0.02 or 0.01 ppm. This implies that the detection limit for PCBs in wet sediment samples is likely to be between 0.01 and 0.02 ppm per gram wet sediment for a sample size of 10 to 20 g among most participating laboratories.

The mean recoveries of sediment samples 2 and 3, without rejection of any data, are 92.8% and 98.5%, respectively, which are very close to the design levels. The corresponding coefficients of variation of these sediment results are 18.3% and 19.1%. After rejection of an outlier result as identified by Dixon's procedure (Dixon and Massey, 1969), the adjusted mean recovery and coefficient of variation (C.V.) for sample 3 are 94.8% and 14.0%, respectively. As indicated in Table 6, the average coefficient of variation of the present sediment data is similar to the results observed by Pavlou and Hom (1976) in another interlaboratory study on PCBs at comparable levels. Hence the reliability of the present data is consistent with the

Table 2. Results of Interlaboratory Study No. 25

Laboratory number	Sample 1 (ppm)	Sample 2 (ppm)	Sample 3 (ppm)	Sample 4 (ng/ $\mu$ L)
1	0.005*	0.593*	0.568*	0.524*
	0.010†	1.186†	1.136†	1.048†
2	0.015	1.165	1.265	1.04
4	None detected	0.65	1.82	1.68
8	0.02	1.04	1.48	1.28
10	<0.01	0.84	1.0	1.0
14	<0.01	1.09	1.28	1.08
17	0.027	1.126	1.353	1.009
23	<0.01	0.836, 0.819	1.007, 1.007	1.45
24	0.03	0.92	1.08	0.96
43	<0.02	1.0	1.05	1.09
44	0.019	0.654	0.931	0.868
47	None detected	0.982, 0.972	1.08, 1.20	0.995, 0.965
53	<0.02	1.08	1.36	1.15
70	<0.01	0.83	1.2	1.1
Design level	x	x + 1.01	x + 1.22	1.00

\* Results originally reported.

† Corrected results after a mistake in calculation was found.

Table 3. Percent Recovery of PCBs in Samples 2, 3 and 4

Laboratory number	Sample 2	Sample 3	Sample 4	Mean laboratory recovery $\pm$ S.D.
1	115.1	91.6	104.8	103.8 $\pm$ 11.8
2	113.1	102.0	104.0	106.4 $\pm$ 5.9
4	63.1	146.8 R	168.0 R	126.0 $\pm$ 55.5
8	101.0	119.4	128.0	116.1 $\pm$ 13.8
10	81.6	80.6	100.0	87.4 $\pm$ 10.9
14	105.8	103.2	108.0	105.7 $\pm$ 2.4
17	109.3	109.1	100.9	106.4 $\pm$ 4.8
23	84.4	81.2	145.0 R	102.2 $\pm$ 37.1
24	89.3	87.1	96.0	90.8 $\pm$ 4.6
43	97.1	84.7	109.0	96.9 $\pm$ 12.2
44	63.5	75.1	86.8	75.1 $\pm$ 11.7
47	94.9	91.9	98.0	94.9 $\pm$ 3.1
53	104.9	109.7	115.0	109.9 $\pm$ 5.1
70	80.6	96.8	110.0	95.8 $\pm$ 14.7
				$\bar{x}$ = 101.2 $\pm$ 12.6*
				$\bar{x}$ = 101.2 $\pm$ 10.1†
Mean percent recovery‡	92.8	98.5	112.4	
S.D.‡	17.0	18.9	21.5	
C.V. (%)‡	18.3	19.1	19.1	
Adjusted mean percent recovery§	92.8	94.8	105.0	
Adjusted S.D.§	17.0	13.3	10.4	
Adjusted C.V. (%)§	18.3	14.0	9.9	

\* Mean interlaboratory recovery  $\pm$  1 S.D.† Mean intersample recovery  $\pm$  1 S.D.

‡ Values calculated using all data.

§ Values calculated after rejection of outliers (R).

Table 4. Summary of Analytical Methods for Extraction and Cleanup

Laboratory number	Sample pretreatment	Method	Number of extraction	Solvent system	Cleanup*
1	None reported	Ultrasonic	3	1:1 Hexane/acetone	d,i
2	None reported	Ultrasonic	3	Acetone	a,b
4	None reported	Shaker	2	Acetonitrile	a,b,c,e
8	None reported	Blender	3	Acetonitrile	a,b
10	Air dry	Soxhlet	1	9:1 Hexane/acetone	a,b,c,f,g
14	None reported	Shaker	3	Acetone/hexane	b
17	None reported	Ultrasonic	3	1:1 Hexane/acetone	a,b,c,d
23	None reported	Shaker	1	1:1 Hexane/acetone	a,b,e
24	None reported	Ultrasonic	3	1:1 Hexane/acetone	b,c,d
43	Air dry	Soxhlet	1	1:1 Hexane/acetone	b,c
44	None reported	Ultrasonic	1	3:1 Ethyl acetate/toluene	h
47	None reported	Stirrer	3	Acetone	a,b,c
53	None reported	Soxhlet	1	1:1 Hexane/acetone	b,c
70	None reported	Ultrasonic	2	Acetone	a,b,c

\* Cleanup procedures: a—Solvent partitioning  
 b—Florisil column  
 c—Mercury  
 d—Activated copper  
 e—Charcoal column  
 f—Potassium hydroxide  
 g—Chromium trioxide/acetic acid  
 h—GPC with Bio Beads SX3  
 i—Silica gel

previous studies (Aspila and Carron, 1978; Pavlou and Hom, 1976), and variations of analytical results within ca. ±20% of the mean for total PCB content are expected in the interlaboratory comparison. However, an improvement was observed for the PCB results obtained in the present study in comparison with those of interlaboratory study No. 20 (Aspila and Carron, 1978), which included the analysis of PCBs in sediment extracts.

The mean recovery of sample 4 (PCB standards in iso-octane) is 112.4%. This result is due to two high results in a relatively small number of data sets. If the two highest results (168% and 145%), which are identified as outliers by Dixon's procedure, are rejected, the mean recovery of sample 4 is 105.0%. It is surprising that the variation of results on sample 4, without rejection of any data, is as large (C.V. 19.1%) as those of the sediment samples. With the rejection of these two results, the C.V. is still 9.9%. This implies that in PCB analysis, erroneous quantitation and standard solutions contribute a major source of analytical error. Close scrutiny of the electron-capture chromato-

grams on sample 4 and the participants' own in-house PCB standards reveals subtle but noticeable differences in PCB profile. Therefore, the lack of a reference PCB standard common to all participants adds uncertainty in interlaboratory comparison of PCB data.

Judging from the closeness of data to the 45° line in the paired sample plot (Fig. 1), it appears that most laboratories provide accurate sediment PCB analysis, with generally good precision and small random errors. More laboratories tend to under-recover slightly rather than over-recover in the sediment samples.

## DISCUSSION ON METHODOLOGY

### Sample Pretreatment

There is usually no sample pretreatment. In two laboratories, the wet sediments were air-dried before extraction. Results from three out of four air-dried samples were



Table 5. Summary of Analytical Methods for Instrumentation and Quantitation

Laboratory number	Gas chromatograph	GLC column and temperature	Quantitation	Injection	Standard* used
1	Tracor MT-220	3% OV-101, 200°C 1.5% OV-17 + 1.95% QF-1, 200°C	Webb-McCall peak area	Automatic	a
2	Tracor MT-220	3% OV-101, 195°C 6% SE-30, 205°C	Webb-McCall peak area	Manual	a
4	HP-5840	10% DC-200 160°C-210°C at 4°C per min	Total area of ten selected peaks	Manual	a
8	HP-5739A	1.5% OV-17 + 1.95% QF-1, 200°C	Total area of all peaks	Manual	a
10	HP-5710	1.5% SP-2250 + 1.95% SP-2401, 190°C	Webb-McCall	Manual	a
14	Tracor MT-220	3% OV-101, 220°C	Webb-McCall peak height	Manual	a
17	Tracor MT-220 HP-5713	3% OV-101, 192°C	Webb-McCall peak area	Automatic	b
23	Varian 3700	4% SE-30 + 6% QF-1, 200°C	Total peak height of three selected peaks	Manual	a
24	Varian 3700	3% OV-101, 200°C	Webb-McCall peak area	Automatic	b
43	HP-5710A	10% OV-1, 190°C	Webb-McCall peak area	Manual	b
44	HP-5750 HP-5700	2% SE-30 + 3% QF-1 2% OV-1 + 5% OV-210 195°C	Peak heights of selected peaks	Manual	b
47	Varian 3700	4% OV-101 + 6% OV-210 200°C	Peak heights of two selected peaks	Manual	a
53	Tracor 560	5% OV-1, 230°C	Total peak height of all peaks	Manual	a
70	HP-5700	Low loading OV-17 and QF-1, 210°C	Area summation of all peaks	Automatic	a

Note: All laboratories used electron-capture detector for detection and quantitation.

\* Standard used: a-1:1 mixture of Aroclors 1254 and 1260; b-1:1:1 mixture of Aroclors 1242, 1254 and 1260.

Table 6. Comparison of Data Variability among Several Inter-laboratory Studies on PCBs in Sediments

Investigator	Sample	Number of participants	C.V. (%)
Pavlou and Hom (1976)	Wet marine sediments	10	22
National study No. 20*	Lake sediment extracts	10	30.4
This study	Wet lake sediments	14	16.2

\*Aspila and Carron (1978).

slightly lower (ca. 80% to 85% recovery) than average. However, no significant loss of PCBs could be ascertained in those cases.

#### Extraction

Of the methods used in the extraction of PCBs from sediment, which included ultrasonic, Soxhlet, blender and shaker methods, the ultrasonic was the most popular. In this study, Soxhlet extraction did not seem to provide higher recoveries of PCBs in comparison with other extraction

techniques. Most participants used a mixture of polar and non-polar solvents, such as 1:1 acetone and hexane, as the extracting solvent, while others used a single polar solvent such as acetonitrile or acetone. No obvious relationship was observed between the recoveries and the extraction procedure. This suggests that all the extraction methods used in this study were equally efficient for the recovery of PCBs in spiked sediment samples.

### Cleanup

Judging from the results and the accompanying chromatograms, cleanup of these relatively clean sediment samples posed no problems to any of the participants. Solvent partitioning coupled with Florisil adsorption chromatography was the most widely used cleanup procedure. For sulphur removal, mercury and activated copper were generally applied.

By careful comparison of the chromatograms, it was noted that laboratory No. 43 showed a marked decrease in peak size/recovery in one of the PCB components, namely, peak RRT 103 (p,p'-DDE = 100) on the OV-101 column, in sediment samples 2 and 3 as compared with sample 4. Since this peak contributes about 7.5% of the total PCB content in a 1:1 mixture of Aroclors 1254 and 1260, the low recovery would not yield a significant error in the results, especially when the Webb-McCall method was used by this laboratory. For natural samples, however, this reduction in peak size might be erroneously interpreted as selective degradation of PCB components in the sample matrix. The reason for this decrease in recovery is suspected as being the overly active Florisil column (adsorbent activated at 170°C instead of the usual 130°C), which selectively adsorbs some PCB components. Similar findings have been observed by Lieb and Bills (1974) as well as in our laboratories.

### Quantitation

All of the laboratories invariably used gas chromatographs equipped with electron-capture detectors for detection and quantitation of PCBs. The GLC columns and conditions used were typically those employed in the analysis of PCBs and organochlorinated pesticides.

As indicated in Table 5, half of the participating laboratories employed the Webb-McCall method for PCB quantitation, while the rest utilized other methods. Thus the results on sample 4 can be divided into these two groups, as presented in Table 7. Assuming that the errors due to standard solutions and instrumentation used by all the laboratories are normally distributed, the results indicate that the group which used the Webb-McCall quantitation

method produced results with better precision and accuracy than the other group which used different quantitation techniques. Similar results have also been reported by Sawyer in a collaborative study on the quantitation of PCB mixtures (Sawyer, 1978).

Table 7. Comparison of Results on Sample 4 According to Webb-McCall Method and Other Quantitation Methods

	Webb-McCall	Other methods
	0.96	1.68
	1.009	0.868
	1.0	1.1
	1.08	1.45
	1.04	1.28
	1.09	1.15
	1.048	0.98
Design value	1.00	1.00
Mean	1.03	1.22 (1.08)*
S.D.	0.05	0.28 (0.16)*
C.V. (%)	4.5	23.0 (14.7)*

Note: Total PCB concentrations are in nanograms per microlitre. \* Results in parentheses were recalculated after the two highest results, 1.68 and 1.45 ng/μL, had been excluded.

## CONCLUSION

The results of PCB analysis in sediments obtained in this study show an improvement (better interlaboratory precision) over those obtained in the national interlaboratory quality control study No. 20 conducted two years ago. The interlaboratory coefficient of variation on the spiked sediment samples is about 20%, and hence the comparability of PCB data in sediment samples on an interlaboratory basis is confirmed. The detection limit for the participating laboratories is estimated to be between 0.01 and 0.02 ppm of PCBs in wet sediment samples. Although the extraction and cleanup procedures reported by the participants vary greatly, they appear to be equally efficient. Quantitation remains as a major source of error in the analysis of PCBs. Among the various quantitation techniques, the Webb-McCall method provides the best precision and accuracy.

## ACKNOWLEDGMENTS

The participating laboratories listed in the Appendix are gratefully acknowledged for their cooperation and patience.

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## APPENDIX

### LIST OF PARTICIPANTS

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Pacific Region, Water Quality Branch Laboratory,  
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Western Region, Water Quality Branch Laboratory,  
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#### *Environment Canada, Environmental Protection Service*

Atlantic Region, Halifax, Nova Scotia  
Wastewater Technology Centre, Burlington, Ontario

#### *Fisheries and Oceans Canada*

Freshwater Institute, Industry Service Branch,  
Winnipeg, Manitoba

#### *Provincial Laboratories*

Alberta Environment, Pollution Control Laboratory,  
Edmonton  
Manitoba Department of Mines, Resources and  
Environmental Management, Winnipeg  
Ontario Ministry of the Environment, Laboratories  
Services Branch, Toronto

#### *Industrial Consulting Laboratories*

Beak Consultants Ltd., Mississauga, Ontario  
Bondar-Clegg & Co. Ltd., Ottawa, Ontario  
B.C. Research, Vancouver, British Columbia  
Enviroclean Ltd., London, Ontario

Environment Canada Library, Burlington



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