NATIONAL WATER RESEARCH INSTITUTE

INSTITUT NATIONAL de RECHERCHE sur les EAUX

INTERLABORATORY STUDY NO. G-1: ANALYSIS OF SELECTED CHLORINATED HYDROCARBONS IN SEDIMENTS

W.C. Li, A.S.Y. Chau and I. Orchard

NWRI Contribution No. 91-127

INTERLABORATORY STUDY NO. G-1: ANALYSIS OF SELECTED CHLORINATED HYDROCARBONS IN SEDIMENTS

W.C. Li, A.S.Y. Chau and I. Orchard [1]

Research and Applications Branch
National Water Research Institute
Burlington, Ontario L7R 4A6

[1] Environment Canada
Conservation & Protection
Ontario Region
Toronto, Ontario M4T 1M2

NWRI Contribution No. 91-127

MANAGEMENT PERSPECTIVE

Hexachlorobutadiene (HCBD), hexachlorobenzene (HCB) and octachlorostyrene (OCS) were found to be present in the St. Clair River Delta during Upper Great Lakes Connecting Channels Study (UGLCCS). The follow-up study for the clean up of contaminated sediments form areas of concern was conducted under the auspices of Great Lakes Action Plan (GLAP).

The successful implementation of the GLAP is dependent on the availability of reliable scientific data. To assist project managers and regulating bodies to ensure the validity of analytical data, an interlaboratory study (G-1) for the analysis of selected chlorinated hydrocarbons, namely, HCBD, HCB and OCS in sediments was designed and conducted. This study will help to establish the degree of comparability of interlaboratory results among participating laboratories.

SOMMAIRE À L'INTENTION DE LA DIRECTION

La présence d'hexachlorobutadiène (HCBD), d'hexachlorobenzène (HCB) et d'octachlorostyrène (OCS) a été relevée dans le delta de la rivière St. Clair au cours de l'Étude sur les voies fluviales interlacustres du secteur supérieur des Grands Lacs. L'étude de suivi relativement à l'assainissement des sédiments contaminés des secteurs préoccupants a été effectuée dans le cadre du Plan d'action pour les Grands Lacs.

Le succès du Plan d'action pour les Grands Lacs est tributaire de la disponibilité de données scientifiques fiables. Dans le but d'aider les gestionnaires de projet et les organismes de réglementation à assurer la validité des données d'analyse, on a mené une étude interlaboratoire (G-1) portant sur le dosage de certains hydrocarbures chlorés (HCBD, HCB et OCS) dans les sédiments. Celle-ci permettra d'établir le niveau de comparabilité des résultats obtenus par les différents laboratoires participants.

ABSTRACT

As part of quality assurance/quality control (QA/QC) program for sediments under the auspices of the Great Lakes Action Plan (GLAP), an interlaboratory study (G-1) for the analysis of selected chlorinated hydrocarbons in sediments was designed and conducted. Twenty-six laboratories were sent seven test samples including two standard solutions and five naturally contaminated sediments. Each laboratory was requested to analyze the three selected chlorinated hydrocarbons, namely, hexachlorobutadiene (HCBD), hexachlorobenzene (HCB) and octachlorostyrene (OCS) in all test samples. Sixteen out of twenty-six laboratories submitted results. In general, intralaboratory precision for duplicate sediments was good for most of the participating laboratories. Interlaboratory precision for standard solutions was comparable to the previous interlaboratory studies for the analysis of chlorinated hydrocarbons. However,interlaboratory precision for sediments was more divergent since they involved more tedious sample preparation procedures for these selected chlorinated hydrocarbons. The agreement between the interlaboratory medians and the design values of HCBD, HCB and OCS in standard solutions was excellent. It suggests that in-house working standards and performance of instrumentation of participants were satisfactory. In contrast, the low recoveries of three chlorinated hydrocarbons especially HCBD, as obtained from sediment samples, were likely due to problems in tedious sample preparation procedures as mentioned earlier.

RÉSUMÉ

Dans le cadre du programme d'assurance et de contrôle de la qualité (AQ/CQ) pour les sédiments relevant du Plan d'action pour les Grands Lacs, on a mené une étude interlaboratoire (G-1) portant sur le dosage de certains hydrocarbures chlorés dans les sédiments. On a envoyé sept échantillons d'essai dont deux solutions étalons et cinq sédiments naturellement contaminés à vingt-six laboratoires. Chaque laboratoire devait doser les trois hydrocarbures chlorés choisis, soit l'hexachlorobutadiène (HCBD), l'hexachlorobenzène (HCB) et l'octachlorostyrène (OCS) dans tous les échantillons d'essai. Seize des vingt-six laboratoires ont présenté des résultats. En général, la précision intralaboratoire concernant les sédiments en double était bonne dans la plupart des laboratoires participants. La précision interlaboratoire pour les solutions étalons était comparable aux études interlaboratoires antérieures de dosage d'hydrocarbures chlorés. Toutefois, la précision interlaboratoire au niveau des sédiments différait plus étant donné que les méthodes de préparation des échantillons de sédiments en vue du dosage des ces hydrocarbures chlorés choisis étaient plus fastidieuses. La concordance entre les médianes relevées dans chacun des laboratoires et les valeurs nominales pour le HCBD, le HCB et l'OCS dans les solutions étalons était excellente. Cette concordance semble indiquer que les échantillons de référence et le fonctionnement des appareils utilisés par les participants étaient satisfaisants. Par contre, les faibles récupérations de trois hydrocarbures chlorés, en particulier du HCBD, obtenues à partir des échantillons de sédiments, étaient probablement attribuables à des problèmes au niveau des méthodes de préparation fastidieuses des échantillons mentionnées précédemment.

1 INTRODUCTION

Hexachlorobutadiene (HCBD), hexachlorobenzene (HCB) and octachlorostyrene (OCS) were found to be present in the St. Clair River Delta during Upper Great Lakes Connecting Channels Study (UGLCCS). The follow-up study for the clean up of contaminated sediments from areas of concern was conducted under the auspices of Great Lakes Action Plan (GLAP).

To assist project mangers and regulating bodies to ensure the validity of analytical data, a QA/QC (quality assurance/quality control) program for sediments was initiated in September, 1990 upon the request from Environmental Protection - Ontario Region as part of Great Lakes Action Plan. The objectives of this program are (1) to prepare sediment reference standards and reference materials for hexachlorobutadiene (HCBD), hexachlorobenzene (HCB) and octachlorostyrene (OCS) as well as any other significant sediment-associated contaminants for the support of monitoring in the St. Clair River Delta; (2) to design and conduct interlaboratory studies specific to HCBD, HCB and OCS for the evaluation of contract laboratories.

As part of this QA/QC program for sediments, a series of interlaboratory comparison studies, on a continual basis, will be designed and conducted by the Quality Assurance Project of the Research and Applications Branch at the National Water Research Institute. The goal of these studies is to assist analytical laboratories to generate accurate data. The present interlaboratory comparison study, G-1, was distributed on November 21, 1990. It involved the analysis of three selected chlorinated hydrocarbons, namely, HCBD, HCB and OCS, in standard solutions and naturally contaminated sediment samples. The original deadline for reporting results was set for January 25, 1991. However, most laboratories were late in reporting, so the study was closed on February 15, 1991. A preliminary data summary with a brief overview was prepared and distributed to those participants which had submitted their results. The summary allows participants to compare their results with those of their peers and also with the design

values. Thus corrective action can be taken if necessary in a timely manner. This final report provides more information on the data evaluation and laboratory performance of participants.

2 STUDY DESIGN

An interlaboratory study (G-1) for the analysis of HCBD, HCB and OCS in standard solutions and sediments was initiated in September, 1990. About 70 government, industrial and private laboratories were invited to participate. From the returned questionnaires, twenty-six laboratories expressed interest to participate in this study. By the time the study closed, sixteen out of twenty-six participants had submitted their results. The list of participants is given in Table 1.

The study consisted of seven test samples for the analysis of selected chlorinated hydrocarbons, namely, hexachlorobutadiene (HCBD), hexachlorobenzene (HCB) and octachlorostyrene (OCS). Description of samples is given in Table 2. Briefly, samples #1 and #2 in sealed glass ampules were mixtures of standard solutions of HCBD, HCB and OCS in iso-octane at various concentrations. The sample #2 is a ten times dilution of sample #1. These standard solutions were used to evaluate the performance of in-house calibration standards and instrumentation of participants. Samples #3 to #7 were freeze-dried naturally contaminated sediment samples for the evaluation of accuracy and precision of analytical procedures used by participants. To assess reproducibility within the same laboratory, two pairs of blind duplicates were included as shown in Table 2.

3 RESULTS AND DISCUSSION

3.1 Analytical Methodology

The participants were instructed to analyze the test samples using their in-house analytical methodology and standards. However, a known standard solution of OCS (100.0 μg/mL) was also provided to each laboratory for the preparation of OCS calibration standards.

In general, a wide variety of analytical methods, sample extractions and cleanup procedures were used by participants. Of the methods used in the extraction of HCBD, HCB and OCS from sediments, which included soxhlet, sonicator and shaker methods, the soxhlet was most commonly used. The solvent system included mixtures of acetone and hexane, acetone and dichloromethane, or dichloromethane alone. Solvent was evaporated by using rotavap, Kuderna-Danish evaporator, Snyder column, Turovap or nitrogen with a water bath. Cleanup of sediment extracts was achieved by adsorption chromatography using silica gel or Florisil. Mercury and activated copper were also used to remove sulphur interferences. All participants used either single or dual capillary columns for the separation of the HCBD, HCB and OCS. Electron capture detection for sample analysis was used by all participants. Analytical methodology used by participants is summarized in Table 3.

3.2 Data Evaluation

The data submitted by all participants for HCBD, HCB and OCS in standard solutions are summarized in Tables I-1 to I-3 in Appendix I, respectively, while the data for HCBD, HCB and OCS in sediments are summarized in Tables I-4 to I-6, respectively. Interlaboratory means and standard deviations of these samples were calculated after outliers (marked with a *) were removed by using Grubbs' test (1). One laboratory (G033) submitted data after the closing date. Their results were not included in the final data summary but their data and methodology can be found in Appendix V as late results. To determine accuracy of interlaboratory results, median values were used to compare with the design values. The design values and interlaboratory medians for HCBD, HCB and OCS in standard solutions and sediments are summarized in Tables 4-1 and 4-2, respectively. The design values for HCBD, HCB and OCS in standard solutions (samples #1 and #2) were confirmed by comparison with standard solution (MISA-230) obtained from Ultra Scientific (North Kingstown, RI, USA). The design values of sample #3 (sediment CRM #EC-3) were established by extensive in-house analysis and results

from a previous interlaboratory study QM-6 (2). The design values of samples #4 and #7 (sediment RM #EC-7) and samples #5 and #6 (sediment RM #EC-6) were obtained from our in-house analysis by using an analytical method developed by Lee et al. (3). These two sediment RMs (#EC-6 and #EC-7) had not been used in any previous interlaboratory studies. The accuracy of interlaboratory results for HCBD, HCB and OCS in test samples was evaluated by the percent recovery of interlaboratory medians. The percent recovery was calculated by dividing the interlaboratory median by the design value and multiplying by 100% as follows.

$$\% \text{ Recovery} = (\text{Interlab. Median} / \text{Design Value}) \times 100$$

For the interlaboratory studies of the QA/QC program for GLAP, values determined for test samples in an interlaboratory study, were considered to be satisfactory if they fell within a window of ±25% of the design value. These criteria of ±25% are somewhat arbitrary but have been used in other QA/QC programs (4,5). For standard solutions without matrix effect and at the higher concentration levels, these criteria could be a little generous whereas at sub ppb levels and in the presence of a large amount of co-extractive (sediments), these criteria are quite demanding. For the present study, these criteria are used for the evaluation of interlaboratory results for the three selected chlorinated hydrocarbons (namely, HCBD, HCB and OCS) in both standard solutions and sediments.

Comparison of the interlaboratory medians with the design values for standard solutions (Table 4-1) showed that agreement for all three parameters was excellent with the deviations within ±5% of the design values. Interlaboratory results for the sediment samples showed that wide deviations existed while the magnitude of the deviations varied for the different sediment samples and for the different parameters and in all cases were greater than those for the standard solutions. This was to be expected because analysis of sediment samples involved more tedious sample preparation steps such as extraction, concentration, and cleanup. Table 4-2 shows that with the exception of samples #5 and

#6 for HCBD the percent recoveries of interlaboratory medians for the three chlorinated hydrocarbons were low. However, the percent recoveries for HCB and OCS in sediment samples #3, #4 and #7 were within ±25% of the design values. For sediment samples #5 and #6 (RM #EC-6), the concentrations of three parameters were in general low or near the detection limit, the interlaboratory results was less satisfactory with the deviations more than ±25% of the design values. Of the three parameters studied, HCBD is more volatile than the other two parameters (HCB and OCS) so some HCBD could be lost during sample processing. Thus this could account for recoveries of HCBD for different sediments varying more widely than those for HCB and OCS. The range and average values of percent recoveries of interlaboratory medians for HCBD, HCB and OCS in standard solutions and sediments are summarized in Table 5-1.

Interlaboratory precision for the three selected chlorinated hydrocarbons, expressed as the relative deviation (RSD) is given in Table 5-2. As can be seen from the table, the interlaboratory precision for standard solutions was better than those of the sediment samples. Results of the unknown standard solutions indicate that of the three parameters studied, only the results for HCBD have RSD outside the ±25% range. On the other hand, results for all these three parameters in sediment samples have RSD outside the range of ±25%.

Intralaboratory precision (within-lab precision) of three selected chlorinated hydrocarbons for the two pairs of sediment samples are summarized in Tables III-1 and III-2 in Appendix III. As expected, the interlaboratory precision was usually lower (i.e. larger standard deviation) than the intralaboratory precision since interlaboratory precision involved different laboratories, analytical procedures, instrumentation, and skills of personnel. However, a few laboratories had poor intralaboratory precision and poor accuracy. It is suggested that these laboratories carefully review their internal QA/QC procedure to pay particularly attention to both their calibration standards and analytical procedures.

3.3 Comparison of Laboratory Performance

For detailed data evaluation of each laboratory, submitted results were compared with the design values. The result of each laboratory for a given parameter in a given sample was treated as "recovery" and the design value for that parameter in the sample was taken as the "true" value. Percent recovery for each parameter in a sample was then calculated. These results are summarized in Appendix II. As described previously, the ±25% of the design value was set as the satisfactory range. Outside the satisfactory range, the results were flagged very high, high, low or very low accordingly. In addition to the flagging of individual sample results, bias was evaluated for each individual parameter on all test samples. An average recovery for all results in a study for the same parameter in a given matrix regardless of sample concentrations was calculated and the same designation scheme as above was used to define bias for each individual parameter on all test results in a given matrix. Thus, the recoveries were designated as very low, low, satisfactory, high or very high based on the ranges listed below.

Average or Individual % Recovery	Individual Result Designation (Flag)	Multiple Result Designation (Bias)
≥ 150%	Very high (VH)	Very High (VH)
149% - 125%	High (H)	High (H)
124% - 76%	Satisfactory (S)	Satisfactory (S)
75% - 51%	Low (L)	Low (L)
≤ 50%	Very low (VL)	Very Low (VL)

The results for each laboratory's appraisal for flags and bias is given in Appendix IV. Summaries of flags and bias in standard solutions and sediments for the study G-1, obtained from the Tables in Appendix IV, are given in Table 6-1 and 6-2, respectively. In the calculation of the number of parameters biased and number of results flagged in Table 6-1 and 6-2, a very high (VH) or very low (VL) bias was counted as one bias while

a high (H) or low (L) bias was counted as half of a bias. Similarly, a VH or VL flag was counted as one flag while H or L flag was counted as half of a flag.

To compare the overall laboratory performance in this study, the key step was the selection of an acceptance criterion. The criterion used for this report was the average of % bias and % flags within a study and this criterion was designated as the performance index. This criterion was used in the UGLCCS (Upper Great Lakes connecting Channel Study) and CEPA (Canadian Environmental Protection Act) QA programs for comparison of the relative laboratory performance for organic parameters (4,5). It provides a simple way to evaluate laboratory performance as shown below.

Performance Index	Comment
≤ 25%	Satisfactory
26% - 50%	Moderate
≥ 51%	Poor

Results of performance index for each individual laboratory in this study are also given in Tables 6-1 and 6-2 for standard solutions and sediments, respectively. For the standard solutions, nine out of sixteen participating laboratories had satisfactory performance and only one laboratory had poor performance (Table 6-1). For the sediment samples, in contrast, only two out of sixteen participating laboratories had satisfactory performance and three laboratories had poor performance (Table 6-2). As expected, the laboratory performance of the sediment samples which involved more tedious sample preparation steps was less satisfactory than for standard solutions. It is suggested that use of available sediment reference materials such as EC-3 in in-house and interlaboratory quality control studies should prove to be beneficial in data quality on a long-term basis.

ACKNOWLEDGEMENT

The authors are grateful to the participating laboratories for the time and effort devoted to analyze the test samples and reporting the results. This interlaboratory study would not be successful without their active participation and cooperation. In addition, the authors thank Mr. M. Fox (Lakes Research Branch,NWRI) for the provision of a purified octachlorostyrene standard.

REFERENCES

Grubbs, F.E. 1969. Procedures for detecting outlying observations in samples. Technometrics, Vol. II, P 1-21.

Lee, H.B., D. Takeuchi and E. Kokotich. 1987. Upper Great Lakes connecting channels interlaboratory performance evaluation study QM-6: chlorinated hydrocarbons in sediments and ampules. NWRI Contribution 87-127.

Lee, H.B., R.L. Hong-You and A.S.Y. Chau. 1986. Analytical reference materials. Part V. Development of a sediment reference material for chlorobenzenes and hexachlorobutadiene. Analyst 111: 81-85.

Li, W.C., A.S.Y. Chau, H.B. Lee and E. Kokotich. 1990. Summary report for UGLCC interlaboratory studies on the analysis of chlorinated hydrocarbons in standard solutions, water and sediment samples. NWRI Contribution 90-122.

Li, W.C. and A.S.Y. Chau. 1991. CEPA national interlaboratory comparison study (CP-1): analysis of chlorophenols in standard solution and sediment extracts. NWRI Contribution 91-105.

Table 1. List of the participating laboratories.

Federal Government:

1. Environment Canada
   National Water Quality Laboratory
   Burlington, Ontario

2. Environment Canada
   C&P (EPS) Laboratory Services
   Wastewater Technology Centre
   Burlington, Ontario

3. Environment Canada
   Lake Research Branch
   National Water Research Institute
   Burlington, Ontario

4. Dept. Fisheries & Oceans
   Contaminants and Toxicology Research Division
   Freshwater Institute
   Winnipeg, Manitoba

Provincial Governments:

5. Environment Quebec
   Ste-Foy, Quebec

6. Alberta Agriculture
   Food Laboratory Services Branch
   Edmonton, Alberta

Private Laboratories:

7. Novalab Ltd.
   Lachine, Quebec

8. Mann Testing Laboratories Ltd.
   Mississauga, Ontario

9. Enviroclean
   London, Ontario

10. Enviro-Test Labs
    Edmonton, Alberta

11. Eli Eco Logic International Inc.
    Rockwood, Ontario

12. Zenon Environmental Laboratories
    Burlington, Ontario

13. ASL Analytical Services Laboratories Ltd.
    Vancouver, B.C.

14. Bondar-Clegg & Company Ltd.
    Ottawa, Ontario

15. Barringer Laboratories Ltd.
    Mississauga, Ontario

16. Environmental Protection Labs
    Mississauga, Ontario

Table 2. Sample distributed in study G-1.

Sample No.	Description	Design value*		
		HCBD	HCB	OCS
1	Mixed standard solution, CH-1S	200.0	200.0	200.0
2	Mixed standard solution, CH-2S	20.0	20.0	20.0
3	Freeze-dried sediment CRM, EC-3	59	254	45
4	Freeze-dried sediment RM, EC-7	10.5	59.67	18.83
5	Freeze-dried sediment RM, EC-6	0.75	4.45	3.15
6	Same as sample #5	0.75	4.45	3.15
7	Same as sample #4	10.5	59.67	18.83

Note: * The design values for samples #1 and #2 are in ng/mL and for samples #3 to #7 are in ng/g.

Table 3. Analytical methodology used by participating laboratories.

Lab	Extraction Method	Solvent System	Cleanup	Evaporation Technique	Detection	
					Separation	Measurement
G001	Sonicator	1:1 hexane/acetone	3% deactivated silica column			GC/ECD
G002	Soxhlet	51:49 acetone/hexane	Florisil column (activated at 130°C)	Snyder column	J&W DB-17, 30 m x 0.25 mm x 0.25 µm	GC/ECD
G003		Hexane			J&W DB-5, 30 m x 0.25 mm i.d. x 0.25 µm	GC/ECD
G005	Wrist shaker and Sonicator	1:2 acetone/hexane	Florisil column (activated at 130°C)	Rotary evaporator	Dual capillary columns DB-5 and DB-17, 30 m x 0.25 mm i.d., each	GC/ECD
G006		1:1 acetone/hexane	1% deactivated florisil column	K-D apparatus	Dual capillary columns DB-5 and DB-17, 30 m x 0.25 mm i.d., each	GC/ECD
G009	Soxhlet	Dichloromethane	Activated florisil column	Rotary evaporator and nitrogen evaporation	DB-5, 30 m x 0.25 mm i.d.	GC/ECD
G014	Soxhlet	Dichloromethane	(1) Copper treatment; (2) 2% deactivated florisil column	Rotary evaporator	DB-5, 60 m x 0.25 µm film thickness column	GC/ECD

Table 3.(continued) Analytical methodology used by participating laboratories.

Lab	Extraction Method	Solvent System	Cleanup	Evaporation Technique	Detection	
					Separation	Measurement
G016A	Shaker	5:8 acetone/hexane	(1) Florisil column; (2) Activated copper	Nitrogen evaporation	DB-5, 30 m x 0.25 mm i.d. x 0.25 µm	GC/ECD
G019	Wrist-action shaker and sonication bath	50:30:15 benzene/acetone/ methanol	None	Nitrogen evaporation	not reported	GC/ECD
G023	Soxhlet	45:55 acetone/hexane	5% deactivated florisil	Snyder column	DB-5 capillary column	GC/ECD
G024	Soxhlet	Dichloromethane	(1) Copper filings; (2)40% H_2SO_4; (3) 5% deactivated florisil	Snyder column and K-D apparatus	DB-5 and DB-17; 30 m x 0.25 mm i.d. x 0.25 µm, each	GC/ECD
G029	Wrist-action shaker	50:50 acetone/hexane	3% deactivated silica column		DB-1 and DB-170 capillary columns	GC/ECD
G035	Soxhlet	1:1 hexane/acetone	Florisil column	Turbo vapo apparatus	DB-5 and DB-17 capillary columns	GC/ECD
G039	Soxhlet	Dichloromethane	Florisil column	Rotary evaporator	DB-1 and DB-170 capillary columns	GC/ECD
G040		1:1 acetone/ dichloromethane	Florisil column	Ratary evaporator	DB-5 and DB-1701; 30 m x 0.25 mm i.d., each	GC/ECD
G042	Soxhlet	1:9 acetone/hexane		Rotary evaporator	DB-5 and DB-17; 30 m x 0.25 mm x 0.25 µm, each	GC/ECD

Table 4-1. Design values and interlaboratory medians for HCBD, HCB and OCS in standard solutions (all values are in ng/mL).

Parameter	Sample #1		Sample #2	
	Design Value	Interlab. Median	Design Value	Interlab. Median
HCBD	200.0	200.8 (100.4)	20.0	19.55 (97.8)
HCB	200.0	199 (99.5)	20.0	19.19 (96.0)
OCS	200.0	200.0 (100.0)	20.0	20.0 (100.0)

Note: The numbers in parentheses are the deviations from the design values, expressed as "percent recovery".

Table 4-2. Design values and interlaboratory medians for HCBD, HCB and OCS in sediments (all values are in ng/g).

Parameter	Sample #3		Samples #4 & #7			Samples #5 & #6		
	Design Value	Interlab. Median	Design Value	Interlab. Medians		Design Value	Interlab. Medians	
HCBD	59	35.3 (59.7)	10.5	6.75 (64.3)	6.675 (63.6)	0.75	1.00 (142.9)	1.15 (153.3)
HCB	254	208 (81.9)	59.67	56.0 (93.8)	48.80 (81.8)	4.45	3.10 (69.7)	3.30 (74.1)
OCS	45	36.7 (81.6)	18.83	16.29 (86.5)	15.1 (80.2)	3.15	2.26 (71.7)	2.21 (70.2)

Note: The numbers in parentheses are the deviations from the design values, expressed as "percent recovery".

Table 5-1. Range and average values of percent recoveries of interlaboratory medians for HCBD, HCB and OCS in standard solutions and sediments.

Parameter	Standard Solutions		Sediments	
	Range	Average	Range	Average
HCBD	97.8 - 100.4	99.1 (2)	59.7 - 153.3	96.8 (5)
HCB	96.0 - 99.5	97.8 (2)	69.7 - 93.8	80.3 (5)
OCS	100.0 - 100.0	100.0 (2)	70.2 - 86.5	78.0 (5)

Note: The numbers in parentheses are the numbers of samples.

Table 5-2. Range and average values of RSD of interlaboratory results for HCBD, HCB and OCS in standard solutions and sediments.

Parameter	Standard Solutions		Sediments	
	Range	Average	Range	Average
HCBD	32.6 - 38.7	35.7 (2)	47.4 - 113.0	71.3 (5)
HCB	23.1 - 26.9	25.0 (2)	27.2 - 37.6	30.7 (5)
OCS	13.7 - 26.5	20.1 (2)	25.5 - 131.5	61.0 (5)

Note: The numbers in parentheses are the numbers of samples.

Table 6-1 . Performance of individual laboratory for standard solutions in study G-1.

Lab code	Bias			Flags			Performance index**	Comment
	No. of parameter Analyzed	No. of+ parameter biased	% Bias	No. of results reported	No. of* results flagged	% Flags		
G001	3	0.5	16.7	6	2.5	41.7	29.2	Moderate
G002	3	0.5	16.7	6	0.5	8.3	12.5	Satisfactory
G003	3	0.5	16.7	6	1.5	25.0	20.9	Satisfactory
G005	3	0.5	16.7	6	2.5	41.7	29.2	Moderate
G006	3	1.0	33.3	6	2.0	33.3	33.3	Moderate
G009	3	0	0	6	0	0	0	Satisfactory
G014	3	0	0	6	0	0	0	Satisfactory
G016A	3	0	0	6	0	0	0	Satisfactory
G019	3	0.5	16.7	6	1.5	25.0	20.9	Satisfactory
G023	3	1.5	50.0	6	2.0	33.3	41.7	Moderate
G024	3	2.0	66.7	6	4.0	66.7	66.7	Poor
G029	3	0.5	16.7	6	1.5	25.0	20.9	Satisfactory
G035	3	1.0	33.3	6	2.0	33.3	33.3	Moderate
G039	3	0	0	6	0	0	0	Satisfactory
G040	3	1.5	50.0	6	3.0	50.0	50.0	Moderate
G042	3	0	0	6	0	0	0	Satisfactory

Note: + VH or VL bias was counted as one bias, while H or L bias was counted as half of a bias.
* VH or VL flag was counted as one flag, while H or L flag was counted as half of a flag.
** Performance Index = (%bias + %flags) / 2.

Table 6-1. Performance of individual laboratory for standard solutions in study G-1.

Lab code	Bias			Flags			Performance index**	Comment
	No. of parameter Analyzed	No. of+ parameter biased	% Bias	No. of results reported	No. of* results flagged	% Flags		
G001	3	0.5	16.7	6	2.5	41.7	29.2	Moderate
G002	3	0.5	16.7	6	0.5	8.3	12.5	Satisfactory
G003	3	0.5	16.7	6	1.5	25.0	20.9	Satisfactory
G005	3	0.5	16.7	6	2.5	41.7	29.2	Moderate
G006	3	1.0	33.3	6	2.0	33.3	33.3	Moderate
G009	3	0	0	6	0	0	0	Satisfactory
G014	3	0	0	6	0	0	0	Satisfactory
G016A	3	0	0	6	0	0	0	Satisfactory
G019	3	0.5	16.7	6	1.5	25.0	20.9	Satisfactory
G023	3	1.5	50.0	6	2.0	33.3	41.7	Moderate
G024	3	2.0	66.7	6	4.0	66.7	66.7	Poor
G029	3	0.5	16.7	6	1.5	25.0	20.9	Satisfactory
G035	3	1.0	33.3	6	2.0	33.3	33.3	Moderate
G039	3	0	0	6	0	0	0	Satisfactory
G040	3	1.5	50.0	6	3.0	50.0	50.0	Moderate
G042	3	0	0	6	0	0	0	Satisfactory

Note: + VH or VL bias was counted as one bias, while H or L bias was counted as half of a bias.
* VH or VL flag was counted as one flag, while H or L flag was counted as half of a flag.
** Performance Index = (%bias + %flags) / 2.

Table 6-2. Performance of individual laboratory for sediments in study G-1.

Lab code	Bias			Flags			Performance index**	Comment
	No. of parameter Analyzed	No. of+ parameter biased	% Bias	No. of results reported	No. of* results flagged	% Flags		
G001	3	0.5	16.7	15	6.0	40.0	28.4	Moderate
G002	3	1.0	33.3	15	3.5	23.3	28.3	Moderate
G003	3	1.0	33.3	13	6.5	50.0	41.7	Moderate
G005	3	1.0	33.3	15	6.5	43.3	38.3	Moderate
G006	3	2.0	66.7	13	6.5	50.0	58.4	Poor
G009	3	1.0	33.3	15	8.5	56.7	45.0	Moderate
G014	3	1.0	33.3	15	6.5	43.3	38.3	Moderate
G016A	3	0.5	16.7	15	4.0	26.7	21.7	Satisfactory
G019	3	1.5	50.0	13	6.5	50.0	50.0	Moderate
G023	3	2.0	66.7	13	8.5	65.4	66.1	Poor
G024	3	1.0	33.3	15	5.0	33.3	33.3	Moderate
G029	3	0.5	16.7	15	6.0	40.0	28.4	Moderate
G035	3	1.5	50.0	15	9.5	63.3	56.7	Poor
G039	3	0	0	10	2.0	20.0	10.0	Satisfactory
G040	3	1.0	33.3	15	7.0	46.7	40.0	Moderate
G042	3	0.5	16.7	15	6.5	43.3	30.0	Moderate

Note: + VH or VL bias was counted as one bias, while H or L bias was counted as half of a bias.
* VH or VL flag was counted as one flag, while H or L flag was counted as half of a flag.
** Performance Index = (%bias + %flags) / 2.

APPENDIX I

DATA SUMMARY

Table I-1. Results for HCBD in Standard Solutions.

Sample Results (ng/mL)		
Lab Code	**1**	**2**
G001	314	15.0
G002	209.09	29.34
G003	270	27
G005	135.3	20.5
G006	110	16
G009	216	17
G014	197.59	19.59
G016A	181	19.5
G019	160	10.0
G023	206	23
G024	260	25.0
G029	181	19.9
G035	50.0	5.0
G039	204	16
G040	54.56	15.31
G042	220	21
Mean	185.53	18.69
S.D.	71.74	6.09
Median	200.80	19.55

Table I-2. Results for HCB in Standard Solutions.

Sample Results (ng/mL)		
Lab Code	**1**	**2**
G001	145	14.6
G002	184.86	20.05
G003	220	25
G005	250.02	2.4*
G006	120	15
G009	198	21
G014	183.51	19.11
G016A	225	18.9
G019	190	24.0
G023	251	25
G024	200	30.0
G029	291	30.6
G035	173.5	17.7
G039	205	18
G040	72.16	19.19
G042	200	18
Mean	194.32	21.08
S.D.	52.28	4.87
Median	199	19.19

Table I-3. Results for OCS in Standard Solutions.

Sample Results (ng/mL)		
Lab Code	1	2
G001	248	23.0
G002	196.75	22.39
G003	230	22
G005	194.7	25.3
G006	140	19
G009	205	20
G014	176.61	18.43
G016A	194	18.4
G019	211	25.7
G023	410*	23
G024	300	32.0*
G029	224	23.9
G035	184.4	18.8
G039	202	18
G040	58.35	16.66
G042	200	19
Mean	197.65	20.91
S.D.	52.36	2.86
Median	200	20.0

Table I-4. Results for HCBD in Sediments.

Sample Results (ng/g)						
Lab code	3	4	5	6	7	D.L.
G001	6.70	6.20	1.12	0.86	8.03	1.5
G002	61.64	10.59	7.60	6.03	15.27	0.50
G003	19	4.7	3.4	3.3	3.6	0.1
G005	33.1	3.9	0.9	0.8	4.0	0.3
G006	36	8	2	3	8	1
G009	14	4	1	1	6	0.5
G014	35.23	9.21	1.23	1.21	6.46	0.06
G016A	37	7	0.9	1	7	1
G019	109*	13.0	13.5*	3.2	27.8*	2.0
G023	3	2	ND	ND	2	1
G024	39	11	1.0	1.0	11	0.03
G029	33.5	7.77	1.00	1.19	7.29	0.3
G035	6.5	2.1	0.4	0.5	1.7	0.2
G039	53	10	<2	<2	<2	2
G040	45.15	6.49	1.15	1.61	6.89	-
G042	43	13	0.48	1.1	3.4	0.20
Mean	31.05	7.44	1.706	1.84	6.47	-
S.D.	17.58	3.53	1.927	1.53	3.622	-
Median	35.23	6.75	1.00	1.15	6.675	-

Table I-5. Results for HCB in Sediments.

Sample Results (ng/g)						
Lab code	3	4	5	6	7	D.L.
G001	208	51.5	2.76	3.30	40.7	6.3
G002	203.95	43.39	3.26	4.16	45.60	0.50
G003	210	56	3.8	3.1	60	0.2
G005	580.5*	156.4*	3.0	2.8	63.9	0.3
G006	130	33	2	3	33	1
G009	163	38	3	3	42	0.5
G014	185.11	37.05	3.62	3.45	45.15	0.07
G016A	274	61	3	4	65	1
G019	329	76.0	3.0	3.2	52.0	3.0
G023	285	75	5	4	81	1
G024	170	52	6.3	6.0	53	0.05
G029	311	77.8	5.50	5.15	63.9	0.3
G035	149.8	39.4	13.3*	11.0*	34.3	1.0
G039	243	64	3.1	4.9	35	2
G040	136.12	26.92	1.195	1.83	37.39	-
G042	220	57	3.2	3.2	54	0.20
Mean	214.532	52.537	3.449	3.673	50.37	-
S.D.	62.630	16.202	1.298	1.055	13.69	-
Median	208	56.0	3.10	3.30	48.80	-

Table I-6. Results for OCS in Sediments.

Sample Results (ng/g)						
Lab code	3	4	5	6	7	D.L.
G001	56.7	25.8	1.43	2.11	18.5	2.7
G002	40.84	16.47	3.68	4.07	19.30	0.50
G003	28	22	<12	<25	26	0.2
G005	55.3	34.1	2.6	2.3	17.4	1.7
G006	32	15	ND	ND	14	1
G009	26	11	2	2	13	0.5
G014	23.09	11.06	1.83	1.65	10.15	0.07
G016A	26	17	2	2	17	2
G019	28.2	15.5	<4.0	<4.0	14.9	4.0
G023	57	30	13	9	34*	4
G024	40	22	22	8.8	22	0.07
G029	34.4	15.3	1.49	1.57	13.4	0.3
G035	32.5	16.1	4.4	4.2	11.3	1.0
G039	20	20	<2	<2	16	2
G040	47.40	14.51	2.52	2.60	15.1	-
G042	39	14	0.70	0.76	14	0.10
Mean	36.65	18.74	4.80	3.42	16.137	-
S.D.	12.09	6.56	6.31	2.74	4.122	-
Median	36.7	16.29	2.26	2.21	15.1	-

APPENDIX II

PAERCENT RECOVERY OF HCBD, HCB AND OCS IN STANDARD SOLUTIONS AND SEDIMENTS

Table II-1. Percent recovery of HCBD, HCB and OCS in standard solution.

Lab code	HCBD (%)			HCB (%)			OCS (%)		
	Sample 1	Sample 2	Avg.	Sample 1	Sample 2	Avg.	Sample 1	Sample 2	Avg.
G001	157.0	75.0	116.0	72.5	73.0	72.8	124.0	115.0	119.5
G002	104.5	146.7	125.6	92.4	100.3	96.4	98.4	112.0	105.2
G003	135.0	135.0	135.0	110.0	125.0	117.5	115.0	110.0	112.5
G005	67.5	102.5	85.0	125.0	12.0	68.5	97.4	126.5	112.0
G006	55.0	80.0	67.5	60.0	75.0	67.5	70.0	95.0	82.5
G009	108.0	85.0	96.5	99.0	105.0	102.0	102.5	100.0	101.3
G014	98.8	98.0	98.4	91.8	95.6	93.7	88.3	92.2	90.3
G016A	90.0	97.5	94.0	112.5	94.5	103.5	97.0	92.0	94.5
G019	80.0	50.0	65.0	95.0	120.0	107.5	105.5	128.5	117.0
G023	103.0	115.0	109.0	125.5	125.0	125.3	205.0	115.0	160.0
G024	130.0	125.0	127.5	100.0	150.0	125.0	150.0	160.0	155.0
G029	90.5	99.5	95.0	145.5	153.0	149.3	112.0	119.5	115.8
G035	25.0	25.0	25.0	86.8	88.5	87.7	92.2	94.0	93.1
G039	102.0	80.0	91.0	102.5	90.0	96.3	101.0	90.0	95.5
G040	27.3	76.6	52.0	36.1	96.0	66.1	29.2	83.3	56.3
G042	110.0	105.0	107.5	100.0	90.0	95.0	100.0	95.0	97.5

Table II-2. Percent recoveery of HCBD in sediments.

Lab code	Sample Results (%)					
	3	4	5	6	7	Avg.
G001	11.4	59.0	149.3	114.7	76.5	82.2
G002	104.5	100.9	1101	804	145.4	431.2
G003	32.2	44.8	453	440	34.3	200.9
G005	56.1	34.3	120.0	106.7	38.1	71.0
G006	61.0	76.2	267	400	76.2	176.1
G009	23.7	38.1	133.3	133.3	57.1	77.1
G014	59.7	87.7	164.0	161.3	61.5	106.8
G016A	62.7	66.7	120.0	133.3	66.7	89.9
G019	184.7	123.8	1800	426	264.8	559.9
G023	5.1	19.0	ND	ND	19.0	14.4
G024	66.1	104.8	133.3	133.3	104.8	108.5
G029	56.8	74.0	133.3	158.7	69.4	98.4
G035	11.0	20.0	53.3	66.7	16.2	33.4
G039	89.8	95.2	ND	ND	ND	92.5
G040	76.5	61.8	153.3	214.7	65.6	114.4
G042	72.9	123.9	64.0	146.7	32.4	88.0

Table II-3. Percent recoveery of HCB in sediments.

Lab code	Sample Results (%)					
	3	4	5	6	7	Avg.
G001	81.9	86.3	62.0	74.2	68.2	74.5
G002	79.9	72.7	73.3	93.5	76.4	79.2
G003	82.7	93.8	84.4	69.7	100.6	86.2
G005	229	262	67.4	62.9	107.1	145.7
G006	51.2	55.3	44.9	67.4	55.3	54.8
G009	64.2	63.7	67.4	67.4	70.4	66.6
G014	72.9	62.1	81.3	77.5	75.7	73.9
G016A	107.9	102.2	67.4	89.9	108.9	95.3
G019	129.5	127.4	67.4	71.9	87.1	96.7
G023	112.2	125.7	112.4	89.9	135.7	115.2
G024	66.9	87.1	141.6	134.8	88.8	103.8
G029	122.4	130.4	123.6	115.7	107.1	119.8
G035	59.0	66.0	299	247	57.5	145.7
G039	95.7	107.3	69.7	110.1	58.7	88.3
G040	53.6	45.1	26.9	41.1	62.7	45.9
G042	86.6	95.5	71.1	71.1	90.5	83.0

Table II-4. Percent recoveery of OCS in sediments.

Lab code	Sample Results (%)					
	3	4	5	6	7	Avg.
G001	126.0	137.0	45.4	67.0	98.2	94.7
G002	90.8	87.5	116.8	129.2	102.5	105.4
G003	62.2	116.8	ND	ND	138.1	105.7
G005	122.9	181.1	82.5	73.0	92.4	110.4
G006	71.1	79.7	ND	ND	74.3	75.0
G009	57.8	58.4	63.5	63.5	69.0	62.4
G014	51.3	58.7	58.1	52.4	53.9	54.9
G016A	57.8	90.3	63.5	63.5	90.3	73.1
G019	62.7	82.3	ND	ND	79.1	74.7
G023	126.7	1593	413	288	180.6	398.6
G024	88.9	116.8	698	279	116.8	259.9
G029	76.4	81.3	47.3	49.8	71.2	65.2
G035	72.2	85.5	139.7	133.3	60.0	98.1
G039	44.4	106.2	ND	ND	85.0	78.5
G040	105.3	77.1	80.0	82.5	80.2	85.0
G042	86.7	74.3	22.2	24.1	74.3	56.3

APPENDIX III

INTRALABORATORY PRECISION

(WITHIN-LAB PRECISION)

Table III-1. Intralaboratory Precision for HCBD, HCB and OCS in EC-6 (Samples #5 and #6).

Intralaboratory Mean ± S.D. (%RSD) (ng/g)			
Lab Code	HCBD	HCB	OCS
G001	0.990±0.184(18.59)	3.03±0.382(12.61)	1.77±0.481(27.18)
G002	6.815±1.110(16.29)	3.71±0.636(17.14)	3.875±0.276(7.12)
G003	3.35±0.071(2.12)	3.45±0.495(14.35)	----------
G005	0.85±0.071(8.35)	2.9±0.141(4.86)	2.45±0.212(8.65)
G006	2.5±0.707(28.28)	2.5±0.707(28.28)	----------
G009	1±0(0)	3±0(0)	2±0(0)
G014	1.22±0.014(1.15)	3.535±0.120(3.39)	1.74±0.127(7.30)
G016A	0.95±0.071(7.47)	3.5±0.707(20.20)	2±0(0)
G019	8.35±7.283(87.22)	3.1±0.141(4.55)	----------
G023	----------	4.5±0.707(15.71)	11±2.828(25.71)
G024	1.0±0(0)	6.15±0.212(3.45)	15.4±9.334(60.61)
G029	1.095±0.134(3.11)	5.325±0.247(4.64)	1.53±0.057(3.73)
G035	0.45±0.071(15.78)	12.15±1.626(13.38)	4.3±0.141(3.28)
G039	----------	4.0±1.273(31.83)	----------
G040	1.38±0.325(23.55)	1.513±0.449(29.68)	2.56±0.057(2.23)
G042	0.79±0.438(55.44)	3.2±0(0)	0.73±0.042(5.75)

Table III-2. Intralaboratory Precision for HCBD, HCB and OCS in EC-7 (Samples #4 and #7).

Intralaboratory Mean ± S.D. (%RSD) (ng/g)			
Lab Code	HCBD	HCB	OCS
G001	7.115±1.294(18.19)	46.1±7.638(16.57)	22.15±5.162(23.30)
G002	12.93±3.309(25.59)	44.495±1.563(3.51)	17.885±2.001(11.19)
G003	4.15±0.778(18.75)	58±2.828(4.88)	24.0±2.828(11.78)
G005	3.95±0.071(1.80)	110.15±65.407(59.38)	25.75±11.809(45.86)
G006	8±0(0)	33±0(0)	14.5±0.707(4.88)
G009	5.0±1.414(28.28)	40±2.828(7.07)	12.0±1.414(11.78)
G014	7.835±1.945(24.82)	41.1±5.728(13.94)	10.605±0.643(6.06)
G016A	7±0(0)	63±2.828(4.49)	17±0(0)
G019	20.4±10.465(51.30)	64±16.971(26.52)	15.2±0.424(2.79)
G023	2±0(0)	78±4.243(5.44)	32±2.828(8.84)
G024	11±0(0)	52.5±0.707(1.35)	22±0(0)
G029	7.53±0.339(4.50)	70.85±9.829(13.87)	14.35±1.344(9.37)
G035	1.9±0.283(14.89)	36.85±3.606(9.79)	13.7±3.394(24.77)
G039	----------	49.5±20.506(41.43)	18.0±2.828(15.71)
G040	6.69±0.283(4.23)	32.155±7.403(23.02)	14.805±0.417(2.82)
G042	8.2±6.788(82.78)	55.5±2.121(3.82)	14±0(0)

APPENDIX IV

LAB-SPECIFIC APPRAISAL FOR

BIAS AND FLAG STATMENTS

GLOSSARY OF TERMS

codes

VH: very high

VL: very low

H: high

L: low

S: satisfactory

ND: not detected

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G001

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	1 VH;1 L	S	1 H;1 VL;1 L	S
HCB	2 L	L	3 L	L
OCS	S	S	2 H;1 VL;1 L	S

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G002

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	1 H	H	2 VH	VH
HCB	S	S	2 L	S
OCS	S	S	1 H	S

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G003

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	2 H	H	2 VH;3 VL	VH
HCB	1 H	S	1 L	S
OCS	S	S	1 H;1 L	S

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G005

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	1 L	S	1 VL;2 L	L
HCB	1 H;1 VL	L	2 VH;2 L	H
OCS	1 H	S	1 VH;1 L	S

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G006

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	1 L	L	2 VH;1 L	VH
HCB	2 L	L	1 VL;4 L	L
OCS	1 L	S	2 L	L

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G009

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	S	S	2 H;2 VL;1 L	S
HCB	S	S	5 L	L
OCS	S	S	5 L	L

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G014

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	S	S	2 VH;2 L	S
HCB	S	S	2 L	L
OCS	S	S	5 L	L

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G016A

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	S	S	1 H;3 L	S
HCB	S	S	1 L	S
OCS	S	S	3 L	L

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G019

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	1 VL	L	4 VH	VH
HCB	S	S	2 H;2 L	S
OCS	1 H	S	1L	L

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G023

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	S	S	3 VL	VL
HCB	2 H	H	2 H	S
OCS	1 VH	VH	4 VH;1 H	VH

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G024

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	2 H	H	2 H;1 L	S
HCB	1 VH	H	2 H;1 L	S
OCS	2 VH	VH	2 VH	VH

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G029

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	S	S	1 VH;1 H;3 L	S
HCB	1 VH;1 H	H	1 H	S
OCS	S	S	2 VL;1 L	L

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G035

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	2 VL	VL	3 VL;2 L	VL
HCB	S	S	2 VH;3 L	H
OCS	S	S	2 H;2 L	S

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G039

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	S	S	S	S
HCB	S	S	2 L	S
OCS	S	S	1 VL	S

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G040

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	1 VL	L	2 VH;2 L	S
HCB	1 VL	L	3 VL;2 L	VL
OCS	1 VL	L	S	S

Lab-specific Appraisal for Flag and Bias Statement

Lab Code: G042

Parameter	Standard solutions		Sediments	
	Flags	Bias	Flags	Bias
HCBD	S	S	1 H;1 VL;2 L	S
HCB	S	S	2 L	S
OCS	S	S	2 VL;2 L	L

APPENDIX V

LATE DATA SUBMITTED BY LABORATORY G033

Results Report Form

GLAP Interlaboratory Study No. G-1

Sample	Concentration Unit	Parameter		
		HCBD	HCB	OCS
1	ng/mL	160	100	<500
2	ng/mL	ND	ND	ND
3	ng/g	378	26	ND
4	ng/g	258	86	ND
5	ng/g	248	36	ND
6	ng/g	264	28	ND
7	ng/g	284	24	ND
D.L. for Sediment	ng/g	140	20	500

Detailed Analytical Methodology on Selected Chlorinated Hydrocarbons in Sediments

GLAP Interlaboratory Study No. G-1

Please describe your analytical procedures including extraction, cleanup and methods of detection.

1. CALIBRATION

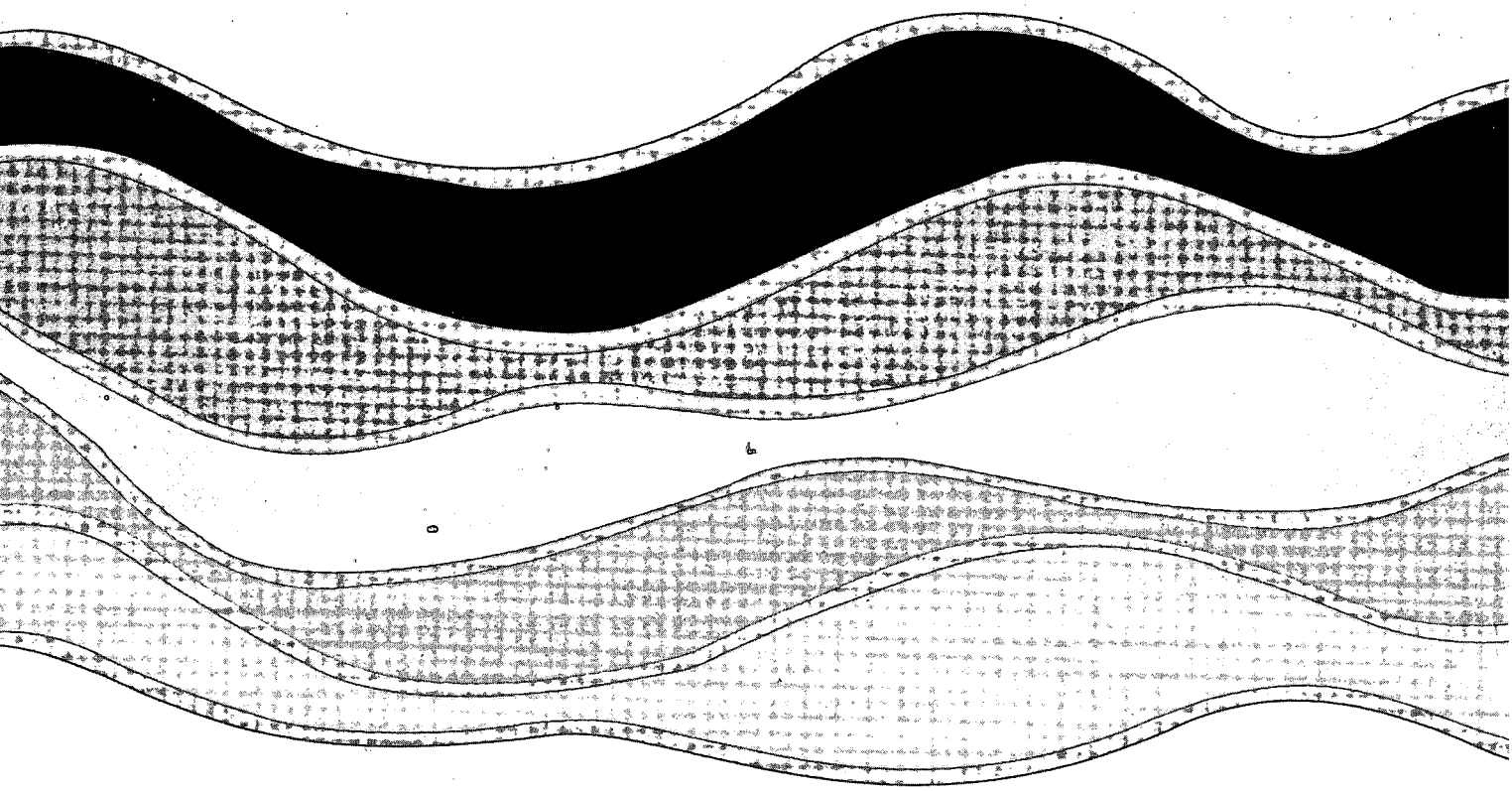
The method was calibrated for the 3 target compounds over the range 1 to 50 ug/ml using 6 calibration levels.

2. SAMPLE PREPARATION

5g of soil was soxhlet extracted with CH_2Cl_2 for 17 h, the extract washed with alkaline water (pH12); dried over Na_2SO_4 and evaporated. A blank soil was extracted at the same time.

3. ANALYSIS

Surrogate recovery was low (average 20-30%).
1 ug/l of the sample extract was injected (units ng)
The result was divided by 5 (ng/g)



NATIONAL WATER RESEARCH INSTITUTE
P.O. BOX 5050, BURLINGTON, ONTARIO L7R 4A6

Environment Canada Environnement Canada

Canada

INSTITUT NATIONAL DE RECHERCHE SUR LES EAUX
C.P. 5050, BURLINGTON (ONTARIO) L7R 4A6