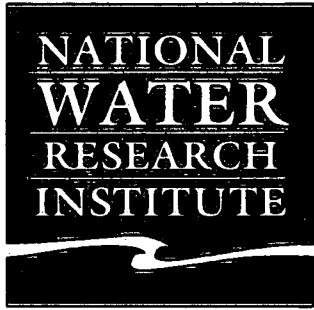
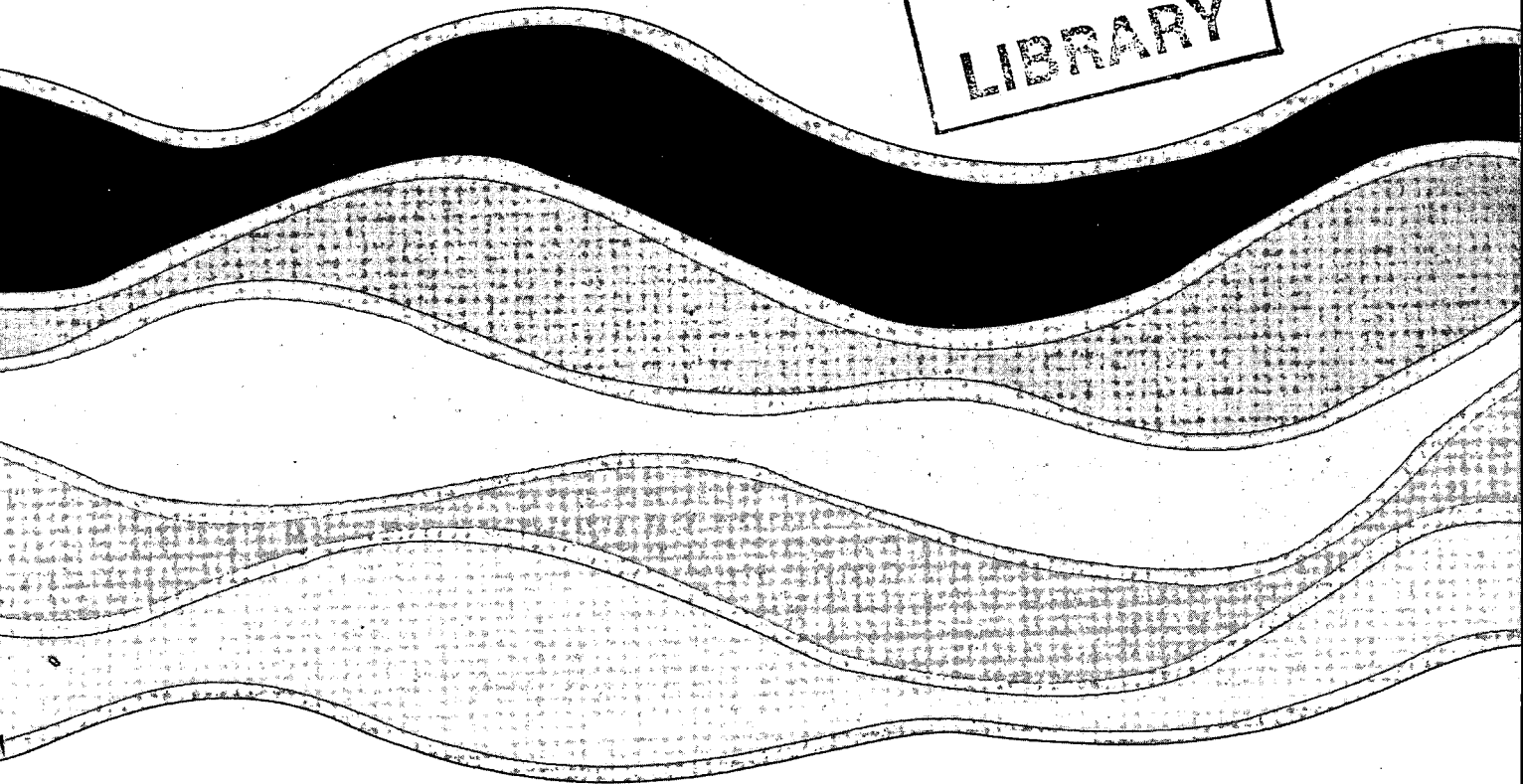


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DREDGING QUALITY CONTROL STUDY NO. 5 (DQC-5)
The Analysis of Polynuclear Aromatic Hydrocarbons, PCBs, and Toxic Trace Metals in Sediment Reference Materials in Direct- Injection Solutions
Yvonne D. Stokker and Eleanor A. Kokotich
NWRI CONTRIBUTION 91-107

MANAGEMENT PERSPECTIVE

Dredging Interlaboratory Quality Control Study No.5 (DQC-5) was conducted at the request of Public Works Canada, Ontario Region and Environment Canada's Pollution Abatement Division of Conservation and Protection, to evaluate the quality of data generated by Canadian laboratories, particularly in the Great Lakes basin, for polynuclear aromatic hydrocarbons (PAHs), total PCBs and eight toxic trace metals in sediments. As part of a larger dredging program, this study was directed toward issues related to contaminated sediments in the Great Lakes, to the Remedial Action Programs in areas of concern, and to technology treatment of contaminated sediments in support of programs under the Canada-Ontario Agreement and the International Joint Commission. The results from this round-robin study indicate that several Canadian laboratories have the capability of performing sensitive and accurate analyses for total PCBs, some PAHs and most of the trace metals in sediment at concentration levels considerably higher than those of the Ontario Ministry of the Environment (MOE) guidelines for the open-water disposal of dredge spoils. However, this study casts some doubt on the ability of many of these same laboratories to accurately measure for certain PAHs and heavy metals, especially cadmium, at the level of the MOE dredging guidelines. The data also reveals that there are several laboratories who could benefit from more stringent in-house quality control and that a select few of the participants need to seriously reevaluate their methodologies and the quality of their calibration standards, especially for the PAHs and certain trace metals. Therefore, by providing an assessment of the capabilities of potential contract laboratories to perform specific sediment analyses in a precise and accurate manner, this report may be used as a guide for Public Works Canada and other federal agencies in the granting of future contracts for the testing of sediments from dredging activities and other marine construction projects.

Dr. J. Lawrence
Director
Research and Applications Branch

Perspective-gestion

L'Étude interlaboratoire n° 5 ayant trait au contrôle de la qualité du dragage (DQC-5) a été menée à la demande de Travaux publics Canada (région de l'Ontario) et de la Division de la lutte contre la pollution du Service de Conservation et de Protection d'Environnement Canada. Cette étude avait comme objectif d'évaluer la qualité des données produites par les laboratoires canadiens, notamment les données concernant les hydrocarbures aromatiques polycycliques (HAP), les BPC totaux et neuf métaux toxiques traces dans les sédiments du bassin des Grands Lacs. Dans le cadre d'un programme de dragage plus vaste, la présente étude portait sur les questions concernant les sédiments contaminés dans les Grands Lacs, les Programmes de mesures correctives dans les zones de préoccupation et la technologie de traitement des sédiments contaminés avec pour but d'appuyer des programmes sous l'égide de l'entente Canada-Ontario et de la Commission mixte internationale. Les résultats de cette étude interlaboratoire indiquent que plusieurs laboratoires canadiens peuvent faire des dosages sensibles et précis des BPC totaux, de certains HAP et de la plupart des métaux traces dans les sédiments à des concentrations beaucoup plus élevées que celles fixées dans les lignes directrices du ministère de l'Environnement de l'Ontario (MEO) concernant le rejet des matériaux de dragage en eau de surface. Toutefois, l'étude soulève certains doutes concernant la capacité de plusieurs de ces laboratoires de mesurer précisément certains HAP et métaux lourds, notamment le cadmium aux concentrations fixées dans les lignes directrices du MEO concernant le dragage. Les données révèlent également que plusieurs laboratoires pourraient bénéficier d'un contrôle interne de la qualité plus sévère et qu'un petit groupe d'entre eux doivent réévaluer sérieusement leurs méthodes et la qualité de leurs étalons, notamment pour les HAP et certains métaux traces. Par conséquent, le présent rapport, qui présente une évaluation des capacités des laboratoires privés susceptibles d'effectuer des analyses spécifiques de sédiments de présenter des résultats précis et exacts, peut être utilisé comme guide par Travaux publics Canada et d'autres organismes fédéraux dans l'octroi des contrats futurs d'analyse des sédiments dans le cadre des activités de dragage et d'autres projets de construction en mer.

J. Lawrence
Directeur
Direction de la recherche pure et appliquée

ABSTRACT

This report describes interlaboratory study DQC-5, the fifth in a series of intercomparison studies conducted by the Quality Assurance group at the National Water Research Institute on various organic and inorganic parameters pertinent to dredging activities. The purpose of the present study was to evaluate the analytical capability and performance of contract and government laboratories on their analyses of polynuclear aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), and toxic trace metals in naturally-contaminated reference sediments and direct-injection solutions. The analytical data submitted by nineteen Canadian laboratories for study DQC-5 were evaluated by various statistical treatments to identify outlying results and to assess laboratory performance with respect to precision, accuracy and bias.

The PCB data submitted by the participants were, for the most part, satisfactory and showed considerable improvement in accuracy and in comparability between laboratories when compared to dredging studies DQC-1, DQC-3 and DQC-4. For the analysis of PAHs, several of the participants produced satisfactory and comparable data, but there were also some extremely erratic, strongly biased, or out-of-control results submitted. Low recoveries of the PAHs from the sediment samples were one of the more common problems observed, but there were also some divergent results submitted for the direct-injection solutions that would generally be more indicative of poor calibration standards and quantitation techniques. This confirms our previous experience that one should interpret the data for PAHs with caution.

Except for cadmium, the trace metal results were generally quite acceptable. Interlaboratory results for copper, lead, nickel and zinc demonstrated a high degree of accuracy and interlaboratory and intralaboratory precision with only a few exceptions. Chromium and arsenic results were slightly more divergent, likely due, in part, to the diversity in methodologies. Moderate difficulties with the analysis for mercury were particularly prevalent at the lower concentration levels. Cadmium results were the least satisfactory of the trace metal data, being neither comparable, nor reliable at less than 2 ug/g. In addition, more than a third of the participants reported detection limits for cadmium that would not have enabled them to quantitate this metal at the guideline levels suggested by the Ontario Ministry of the Environment for the open-water disposal of dredge spoils.

RÉSUMÉ

Le présent rapport présente l'étude interlaboratoire DQC-5, la cinquième d'une série d'études effectuées par le groupe d'assurance de la qualité de l'Institut national de recherche sur les eaux visant à comparer les résultats des laboratoires ayant trait à divers paramètres organiques et inorganiques mesurés dans le cadre des activités de dragage. L'étude avait pour objectif d'évaluer les capacités d'analyse et la performance des laboratoires privés et gouvernementaux dans leur dosage des hydrocarbures aromatiques polycycliques (HAP), des biphényles polychlorés (BPC) et des métaux traces toxiques dans des sédiments témoins contaminés naturellement et dans de simples solutions. On a évalué à l'aide de diverses méthodes statistiques les résultats de dosages présentés par dix-neuf laboratoires canadiens dans le cadre de l'étude DQC-5 dans le but de repérer les résultats aberrants et de déterminer la performance des laboratoires en ce qui a trait à la précision, à l'exactitude et aux biais reliés à leurs dosages.

Les résultats concernant les BPC présentés par les participants étaient, pour la plupart, satisfaisants et présentaient une amélioration considérable du point de vue de l'exactitude et de la comparabilité entre les laboratoires par rapport aux études DQC-1, DQC-3 et DQC-4. Dans le cas du dosage des HAP, plusieurs des participants ont obtenu des résultats satisfaisants et comparables, mais des résultats extrêmement erratiques, fortement biaisés ou non contrôlés ont également été présentés. Les faibles de taux de récupération des HAP dans les échantillons de sédiments étaient l'un des problèmes les plus courants, mais certains résultats divergents présentés pour les simples solutions pourraient en général indiquer de mauvais étalons et de piètres techniques de mesure. Ces résultats confirment notre expérience précédente qu'il faut interpréter avec précaution les résultats concernant les HAP.

Sauf dans le cas du cadmium, les résultats pour les métaux traces étaient en général plutôt acceptables. Les résultats interlaboratoires pour le cuivre, le plomb, le nickel et le zinc témoignent d'un degré élevé d'exactitude et de précision, tant interlaboratoire qu'intralaboratoire, à quelques exceptions près. Les résultats pour le chrome et l'arsenic étaient un peu plus divergents, probablement en partie à cause de la diversité des méthodes utilisées. Les dosages du mercure présentaient certains problèmes, particulièrement aux faibles concentrations. Les résultats pour le cadmium étaient les moins satisfaisants dans la catégorie des métaux traces; ils étaient en effet peu comparables et peu fiables à moins de 2 µg/g. En outre, plus du tiers des participants ont signalé des limites de détection pour le cadmium qui ne les auraient pas permis de mesurer ce métal conformément aux lignes directrices proposées par le ministère de l'Environnement de l'Ontario pour le rejet des matériaux de dragage en eau de surface

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DREDGING QUALITY CONTROL STUDY No.5 (DQC-5)

The Analysis of Polynuclear Aromatic Hydrocarbons, PCBs, and Toxic Trace Metals in Sediment Reference Materials and Direct-Injection Solutions

by

Yvonne D. Stokker and Eleanor A. Kokotich

INTRODUCTION

Contaminated sediments have long been a major environmental concern in many areas of the Great Lakes. Extensive assessments of the levels and significance of contaminants in in-situ sediments, particularly in harbours and nearshore areas of the lakes, have been the focus of many scientific surveys¹, but the evaluation of dredge spoils for open-water disposal has also become an issue of considerable concern². In the early 1970's, the Ontario Water Resources Commission developed sediment guidelines for evaluating dredged material for open-water disposal. These guidelines, which are based on total concentrations of contaminants (eleven heavy metals, nutrients, PCBs, cyanide and oil and grease) in the sediments, were later formalized in an Ontario Ministry of the Environment (MOE) report published in 1976³. In the absence of federal regulations, Environment Canada (DOE) uses these MOE guidelines to evaluate the acceptability of dredgeate for open water disposal in the Great Lakes. However, since 1984, a joint committee, formed by DOE and MOE, has been reviewing these guidelines as well as regulations formulated by other agencies, and, by including a biological impact factor, hopes to establish more comprehensive sediment quality objectives and criteria for specific contaminants or contaminant classes^{4,5,6}.

In 1982, the Dredging Quality Control Program was initiated and funded by DOE's Environmental Protection Service, Ontario Region (EPS-OR). This new program, implemented over the next four years by the Quality Assurance and Methods Section (QAMS) at the National Water Research Institute (NWRI), was part of a larger dredging project that ensured that the organic and inorganic contaminants in dredged sediments were within the MOE guidelines and those of the United States Environmental Protection Agency. The program was designed

to help various federal agencies, such as the Department of Public Works, Small Craft Harbours, Environmental Protection and Fisheries and Oceans Canada, in those projects where data collection and interpretation were essential to the implementation of measures for abatement and control of pollution from dredging activities. Since these measures or decisions were based on data generated by contract laboratories, the capability of these laboratories to perform specific analyses in a precise and accurate manner, had to be established beforehand and their overall performance assessed. The continued use of interlaboratory QC studies could also then provide an ongoing evaluation of a laboratory's performance for use by these same agencies as a selection criterion. Hence, the goal for each of the individual QC studies was to ensure that potential contract laboratories gave reliable analytical results for specific analyses on the dredged sediments. From 1982 to 1986, four round-robin studies were designed and conducted by the QA group at NWRI as follows: DQC-1 evaluated the analysis of PCBs in naturally contaminated sediments and in standard solutions⁷; DQC-2 assessed the analysis of ten toxic trace metals in sediments⁸; DQC-3 again assessed the analysis of PCBs in naturally contaminated dry sediments and in standard solutions⁹; and DQC-4 evaluated the analysis of both PCBs and eight toxic trace metals in dry sediment reference materials¹⁰.

In October 1989, the Quality Assurance Group at NWRI was requested by Public Works Canada, Ontario Region and DOE's Pollution Abatement Division of Conservation and Protection, Ontario Region, to design and conduct another 'dredging' interlaboratory quality control study, this time on the analysis of polynuclear aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), and toxic trace metals in sediments. The program would be directed toward issues related to contaminated sediments in the Great Lakes, to the Remedial Action Programs in areas of concern, and to technology treatment of contaminated sediments in support of programs under the Canada-Ontario Agreement and the International Joint Commission. Public Works Canada would also use this study as a guide in granting future contracts to private laboratories for the testing of sediments for dredging and other marine construction projects.

Forty laboratories across Canada, who had the capability of analyzing for all three classes of pollutant of interest, were invited to participate in the round-robin study. A list of these forty laboratories is provided in Appendix I. Of those invited, twenty-two laboratories agreed to participate and, in early December, sample sets consisting of eleven sediment samples and four direct-injection ampules were sent to these participants. Over the next four months, 19 sets of results were received. A list of the participating laboratories is given in Table 1.

In this report on interlaboratory study DQC-5, the quality of sediment data for sixteen PAHs, total PCBs and eight trace metals, submitted by 19 government and private laboratories, is evaluated.

STUDY DESIGN

In the fall of 1989, a telephone survey of more than fifty government and private laboratories, located primarily in the Great Lakes basin, was conducted to assess the interest and capabilities of these laboratories to participate in a dredging interlaboratory round-robin study. The basic requirements for participation were (a) to have the capability to analyze for polynuclear aromatic hydrocarbons (PAHs), PCBs and eight toxic trace metals in sediments; and (b) to be able to provide results on a set of four to six sediments for each of the above classes by mid-January, 1990. A desirable element for the study was to include as many private laboratories as possible from southern Ontario since Public Works Canada would likely use this study as a guide in the granting of future contracts to private laboratories for the testing of sediments for dredging and other marine construction projects being conducted in the Great Lakes region. A list of forty qualified laboratories was compiled from the telephone survey. Written invitations were then sent to these forty laboratories to participate in the study, but only twenty-two responded positively. Samples were sent to the twenty-two participants in early December with a request for results by January 22, 1990. When only one set of results was received by that date, the study deadline was extended. By early March, nineteen laboratories had provided full or partial results.

The identities and a brief description of the samples distributed in study DQC-5 are given in Table 2. The sample set was comprised of eleven freeze-dried sediment samples and four sealed glass ampules. The sediment samples were fully homogeneous, naturally-contaminated reference materials (RMs) or certified reference materials (CRMs) specially developed for either PAH and PCB analyses or for trace metal analyses. With the exception of BCSS-1, all sediments originated from the Great Lakes basin and had been prepared at the National Water Research Institute in Burlington, Ontario¹¹⁻¹⁶. BCSS-1 was a marine sediment reference material purchased from the National Research Council of Canada (NRCC) and had been certified by them for its trace metal content.* The reference values for the various parameters in each of these materials are listed in Tables 3 to 5 along with each of the interlaboratory medians calculated from the results submitted in this study. The reference values for EC-2, EC-3 and WQB-1 are based on numerous multi-method, multi-operator in-house and contract analyses and have been confirmed by up to five external round-robin studies. The preparation and development of these and other similar reference sediments at NWRI are reported elsewhere¹¹⁻¹³. TH-1, TH-2 and HR-1 were derived from typical dredging sites in Ontario but have not yet been fully characterized. Consequently, the true concentrations of the parameters of interest in these materials are not known with absolute certainty. However, while the reference values listed in Tables 3 to 5 for these latter sediments are based on a lesser number of in-house and external analyses, they too, have been verified by up to five round-robin studies.

The participants were requested to analyze for sixteen PAHs and for total PCBs in five of the sediment samples and for eight trace metals in the remaining six sediments. Specifically, the parameters of interest were: naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene,

* Since the distribution of the samples in this study, the NRCC has withdrawn its certification for mercury in sediment BCSS-1¹⁷. (Sample #15 in this study).

benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-c,d)pyrene, dibenzo(a)anthracene, benzo(g,h,i)perylene, total PCBs, arsenic, cadmium, chromium, copper, lead, mercury, nickel and zinc. Each sediment was to be extracted and analyzed using the laboratory's own routine methods of analysis. Sediment samples #2 and #5 and sediments #11 and #14 were provided as blind duplicate samples for the organic and inorganic analyses, respectively, in order that the participants could estimate their own in-house precision of analysis.

For the analysis of the ampules in this study, the participants were requested to determine the concentrations of the PAHs and PCBs using their own in-house standards and calibration procedures. Samples #6 and #7 were injection-ready 'standard' solutions of a 1:1:1 mixture of Aroclors 1242, 1254 and 1260 in isooctane and samples #8 and #9 were mixtures of the sixteen PAHs of interest. The 'A' series of ampules #8 and #9 were prepared in toluene for those participants who had indicated that they would be analyzing for the PAHs by gas chromatography. Similarly, ampules #8B and #9B were prepared at the same concentration levels in acetonitrile for those participants who would be using HPLC techniques. All ampules, whether for PCB or PAH analysis, were to be analyzed directly to give an indication of the quality and accuracy of in-house standards, quantitation technique and selection of proper standards.

RESULTS AND DISCUSSION

Analytical Methodologies

Summaries of the analytical procedures employed by the participants for the PCB, PAH and trace metal analyses in this study are presented in Tables 6, 7 and 8, respectively. A wide variety of different techniques were used for the extraction of both the organics and the metals from the sediment samples as well as for their analytical measurement. Only seven laboratories of the nineteen who participated in this study, coextracted the PAHs and PCBs from the sediment samples.

Extraction of the sediment samples for PCBs was done primarily by sonication or soxhlet extraction although mechanical shakers and vortex mixers were also employed. A variety of solvents were used for the extractions including acetone/hexane mixtures, dichloromethane, acetone, hexane, carbon disulfide and cyclohexane. Cleanup of the PCB-containing extracts was mostly achieved by column chromatography using either Florisil or silica gel. As listed in Table 6, some participants also used either a base-partitioning step, an alumina column or sulfuric acid in addition to either Florisil or silica gel columns. Sulfur and sulfur compounds were removed by metallic mercury or activated copper.

Fifteen of the nineteen laboratories used capillary columns for the analysis of PCBs in this study. Three participants used packed OV-1 or OV-101 columns and one used a megabore HP-1 column. Five participants employed dual-column GC-ECD, each using a different combination of two capillary columns. Among the remaining ten laboratories, eight different capillary columns were employed. Three of these latter participants quantitated the PCBs using a mass selective detector (MSD). See Table 6 for details. Inspection of the ampule and sediment sample results in this study gave no clear indication of which technique or column provided more accurate PCB results.

The most commonly used method of extraction of the PAHs from the sediment samples was sonication although soxhlet extraction, and shaking were also used by a few laboratories. As seen in Table 7, dichloromethane was employed by eleven of the nineteen participants while five used acetone/hexane mixtures. Carbon disulfide, acetone and an acetone/dichloromethane mixture were each used by one laboratory. Ten participants applied their extracts to either silica gel columns or silica gel solid-phase extraction tubes for cleanup while only one chose SEP PAK and alumina. The remaining laboratories did not use any cleanup column steps.

For the detection and quantitation of the PAHs, thirteen laboratories used either GC-MSD or GC/MS techniques, three used GC-FID, and the remaining

three participants employed HPLC-UV, HPLC-fluorescence, or HPLC with a combination of the two detectors. All participants using gas chromatography analyzed for the PAHs by means of bonded phase fused silica capillary columns such as DB-1 or DB-5 in various lengths.

The extraction and digestion methods used by the participants in this study for the analysis of the eight trace metals in the sediment samples were quite varied. As can be seen in Table 8, the methods differed in most cases in their combination of acids for the digestion step, but several also differed in their analytical instrumentation. For the metals other than mercury and arsenic, the sediment samples were digested with aqua regia by eight of the eighteen laboratories, or a combination of HNO_3 , HCl , HF , HClO_4 , and H_2O_2 by the other ten. The extract was then analyzed by either flame atomic absorption spectroscopy (flame-AAS) or inductively coupled plasma atomic emission spectroscopy (ICP-AES). However, one participant used graphite furnace atomic absorption spectroscopy (GFAAS) for low level lead and cadmium analyses, and one employed direct current atomic emission spectroscopy (DCP-AES) for the analysis of all six of these metals.

Digestion of the samples for mercury analysis was accomplished by a combination of HNO_3 , HCl , H_2SO_4 and HClO_4 in the presence of KMnO_4 , V_2O_5 , or $\text{K}_2\text{S}_2\text{O}_8$. The mercury in the digest was then reduced to elemental mercury by SnCl_2 , SnSO_4 or hydroxylamine. For mercury, the most popular technique for analysis was cold vapour atomic absorption (AAS), although one participant used a mercury monitor and three employed ICP-AES.

The most popular procedure for digesting the sediment samples for the extraction of arsenic was by means of aqua regia. Two laboratories used alkaline fusion and the remaining eight used various combinations of different acids and reagents. As seen in Table 8, the extract was then analyzed by hydride generation AAS or ICP-AES. One laboratory used a colorimetric technique and two employed GFAAS. None of the participants in this study used X-ray fluorescence or neutron activation techniques for any of the metals.

Data Evaluation - Total PCBs

Table 9 lists the raw data reported by the participants for total PCBs in samples #1 to #7. The overall performance for PCB analysis in this study was, for the most part, satisfactory. As can be seen in Table 3, the interlaboratory medians of the seven test samples agreed closely with the reference values (76-102%) and the between-lab standard deviations ranged from 18 to 49% (after rejection of only the most extreme outliers).

To evaluate the accuracy of the PCB data in this study, the "per cent recoveries" (reported results vs. reference values or interlaboratory medians) were calculated for each sample and are tabulated by laboratory in Appendix IV. To provide a semi-quantitative evaluation of this data, the individual results were deemed to be satisfactory if they were within +50% of the interlaboratory median but were designated as very low, low, high, or very high, if this "recovery" value was beyond this range. Appendix III outlines, in more detail, the procedures and criteria used in this study to assess laboratory performance for PCB and PAH analysis. Interlaboratory medians rather than reference values were used as evaluation criteria for data accuracy in the flagging procedure since not all the reference values for the different sediments have been finalized. However, in this case, laboratory performance evaluated against either a consensus standard (medians) or an absolute standard (reference values) would be valid since the interlaboratory medians for total PCBs in each sample were in close agreement with the reference values. Only in a few cases did the two values differ by more than 15%. A complete summary of the PCB results flagged to the interlaboratory medians is provided in Table 27.

It becomes obvious when studying the data in Appendix IV, that the recoveries obtained for ampule solutions #6 and #7 were considerably better than those obtained for sediment samples #1 to #5. Because the ampule solutions required no treatment their "recovery" values may be used as a measure of the accuracy of each participant's in-house calibration standards and their quantitation technique for total PCBs. As seen in Table 9, most results for the ampules were within +25% of the design values except for the

data from laboratories F064, F065, F066, F084 and N197. It should be emphasized that the results obtained on injection-ready solutions are less indicative of a laboratory's performance in real life situations than those on sediment samples since the ampule solutions require no extraction and cleanup and are easier to quantitate than sediment extracts with their coextractives.

In Table 9, the PCB data for the five sediment samples show good comparability between most laboratories with only a few participants in this study submitting outlying results. When these few extreme results (i.e. those flagged either VH or VL) are rejected, the range of reported values becomes considerably narrower and the remaining data falls within two to three-fold of the reference values. Sediment samples #2, #3 and #5 originated from Lake Ontario in the Niagara River plume area and were naturally contaminated with high levels of chlorinated hydrocarbons. Perhaps because of these coextractives, the interlaboratory standard deviation for these three sediments ranged as high as 42% to 49%. Samples #1 and #4, on the other hand, were derived from typical dredging sites around Lake Ontario, and, while they were naturally contaminated with Aroclors 1254 and 1260, they contained much lower concentrations of other organic contaminants. The interlaboratory results for these two samples were much better than those for sediment samples #2, #3 and #5. After rejection of extreme outliers, the relative standard deviations for these two samples were 33% and 26%, respectively, and were consistent with the results submitted in studies DQC-3 and DQC-4.

Assessing the precision of PCB analysis was made possible in this study by the provision of duplicate sediment samples. The range of differences between duplicate samples #2 and #5 ranged from less than 5% (laboratories F066, N041, N107 and N158) to almost 200% (laboratory N197). Only nine of the eighteen participants achieved better than 25% precision of analysis between duplicate sediments #2 and #5.

Several of the PCB analyses conducted by laboratories F057 and N197 were out of control as some results were more than 2000-fold different from the reference values and the medians while others were considerably more accurate. N197 was also deemed to be erratic in their sediment analyses for PCBs because

they received both very high and very low flags. The extremely high sediment results for these two participants could have been due to poor quantitation and/or insufficient cleanup of their extracts so that non-PCB peaks (such as the chlorinated hydrocarbons in samples #2, #3 and #5) were being misinterpreted as PCB components. Laboratories F064, F065 and F084 provided very poor results for the direct-injection samples but were better in their analyses of the sediments. Laboratory N196, on the other hand, achieved good results for the direct-injection solutions but had extreme difficulty in recovering any more than 10% of the PCBs in the sediment samples. Since this participant was able to quantitate the Aroclor mixtures in ampules #6 and #7, it is believed that their low sediment recoveries were likely due to inadequate extraction and cleanup techniques or other random errors rather than to poor or inaccurate PCB standards or quantitation techniques. Laboratories F057 and F066 were considered to be biased high in these PCB analyses and F040, N196 and N208 were biased low in that more than a third of their results were flagged.

It is of interest to note that the detection limits reported by the participants ranged from 0.3 to 100 ng/g, a more than 300-fold difference. As listed in Table 25, six of the eighteen participants in this study reported detection limits for total PCBs in sediment at or above the 50 ng/g level listed by the Ontario Ministry of the Environment in their guidelines for evaluating dredge spoils for open-water disposal³.

In summary, despite the various quantitative techniques used by the participants, the PCB data in this study were satisfactory and comparable. Three laboratories, in particular, demonstrated severe analytical problems but the majority experienced, at most, only minor difficulties with these samples. Nevertheless, adequate precision in the analysis of the duplicate samples was achieved by only half of the participants. Therefore, while most laboratories provided acceptable data for total PCBs in the sediment samples, only laboratories F087, N041, N107 and N158 demonstrated both good precision and accuracy, without any evidence of bias in their PCB data. However, laboratories F063, F064, F094, and N200 provided PCB data that were satisfactory for the most part, but which demonstrated some imprecision in the duplicate samples and/or slight bias in their entire set of PCB results.

Data Evaluation - PAH analyses

The raw data reported by the participants for PAHs in the five sediment samples and two direct-injection mixtures may be found in Tables 10 to 16. Interlaboratory medians for each parameter in the samples were determined using all data (except the 'less than' or 'not detected' values) reported by the participants. Outliers were not rejected when calculating these medians since most of the results fell within two to three-fold of the reference values for each parameter in both the sediment samples and the injection-ready samples. In Table 3, a summary of the interlaboratory medians, the reference values and the recovery of the medians against the reference values, is provided.

Most laboratories analyzed for all sixteen PAHs, but only half of the participants could resolve isomeric pairs such as benzo(b)fluoranthene and benzo(k)fluoranthene. Laboratory F084 had difficulty resolving eight of the sixteen PAHs under study. One participant (F033) omitted five of the PAHs in the sediment analyses and another five PAHs in the direct-injection ampules. Laboratories F007, N107 and N196 submitted incomplete sets of PAH data (i.e. not all samples were analyzed). Four laboratories (F057, F084, N041 and N200) found that more than a third of the PAHs in the sediment samples were below their detection limits but only laboratories F084 and N041 were unable to measure any of the PAHs in the lowest concentration direct-injection sample (Ampule #9).

Evaluation of the accuracy of the PAH results was accomplished in the same manner as was done for the PCB data, using the "percent recoveries" calculated for each laboratory. Appendix IV provides a complete listing of each participant's recovery data in relation to both the reference values and the interlaboratory medians. However, since a few of the medians were not in close agreement with the reference values, and because this technique of evaluating a laboratory's data is a peer appraisal assessment, flags were assigned to the data based on recoveries calculated as a percentage of the median. Table 28 provides a summary of each laboratory's performance based on the percentage of their results that were flagged.

In previous organic interlaboratory studies, erratic in-house standards have been shown to be the single major source of error. Therefore, in the present study, samples #8 and #9 were designed to contain known quantities of each PAH of interest in order to evaluate the accuracy of the participants' in-house calibration standards and quantitation techniques for PAHs. Each of these 'standard' solutions could be quantified by direct injection into a gas or liquid chromatograph. As shown in Tables 15 and 16, the interlaboratory medians agreed with the design values for most of the PAHs in the higher concentration ampule but wider variation in results was seen in the lower concentration ampule. A comparison with Table 25, which lists the participants' detection limits for PAHs in sediment shows that many of the laboratories were at, or approaching, their limits of quantitation for many of the PAHs in sample #9. Laboratories F084 and N041 were unable to detect any of the 16 PAHs in this sample. For the two ampules, laboratory F057 was biased low while each of F065, F066, F094, and N196 were biased moderately high. Nevertheless, the overall performance of the participants on the two ampule mixtures was satisfactory since less than 25% of the results provided, were flagged. Moreover, the comparability of the laboratories with their different methodologies was good, as the lowest and the highest results reported, rarely exceeded a factor of two from the design values in each of the ampules.

The data submitted for the analysis of PAHs in the sediment samples were satisfactory for several of the participants, but as expected, were not as comparable as those for the direct-injection ampules. From 30% to 50% of the PAH results were flagged in each of the five sediments with sample #3 showing the widest range of results. Because the direct-injection results were more accurate for the majority of the participants, many of the problems encountered with the sediment samples were likely due to the extraction or cleanup steps of the procedure rather than the instrumental analysis portion of the method. Losses of the PAHs due to improper sample preservation or due to suspect storage conditions were avoided by providing the participants with freeze-dried sediments for which the stability of the parameters of interest has been monitored and verified for up to ten years.

In general, most of the interlaboratory medians for the PAHs in the sediment samples agreed fairly well with their corresponding reference values. (Refer to Table 3.) More than 80% of the individual results reported by the participants were within two to three-fold of the interlaboratory medians. Surprisingly, however, as can be seen in Table 3, the agreement of the medians to the reference values in sample #5 were far better than in its duplicate sample, sediment #2. In sample #2, six of the medians were less than 70% of their corresponding reference values, whereas in sample #5, all PAHs except chrysene had medians that were greater than 70% of the reference value. In total, half of the PAH medians in sample #5 were from 10% to 30% greater than the corresponding medians in sample #2. Low values for pyrene and chrysene were provided by many of the participants on all five sediment samples. Fluoranthene and benzo(a)anthracene in the lower-concentration sediment samples were also recovered in reduced amounts by several participants. The PAHs for which the interlaboratory medians were not in close agreement with the reference values were those same compounds for which the widest range of concentrations were reported. It appears then, that these data reflect a problem of erratic recoveries, rather than one of consistently low recoveries.

Careful examination of Tables 10 to 16 reveals a significant difference in the results for benzo(b)fluoranthene and benzo(k)fluoranthene reported individually and as a sum of the two isomers. Most analysts who had difficulty resolving these two peaks reported total values that were considerably higher than the total concentrations calculated for the two parameters analyzed separately. Therefore, the agreement with the reference values would be considerably better if all B(b)F and B(k)F data had contributed to the interlaboratory medians. Furthermore, the intralaboratory precision of analysis averaged 15% and 30% for B(b)F and B(k)F reported individually, but was better than 10% between the duplicate samples for those reporting the sum of these two PAHs. However, since the results reported as a sum of the two parameters could not be statistically evaluated, they were not used in the calculation of the medians and the preparation of the data summaries.

The performance of a particular laboratory for the analysis of any or all of the PAHs can be readily assessed from the tables included in Appendix IV. A glance at the summary of flags in Table 28 will tell, overall, whether the results reported by the laboratory were satisfactory (few flags), biased high (presence of multiple VH or H flags), biased low (presence of multiple VL or L flags), or erratic (presence of both VH and VL flags). No matter which approach is used, (calculation of recoveries to the medians or to the reference values), results that are flagged VH or VL are usually indicative of significant analytical errors occurring in that laboratory on that particular sample. From Table 28, it becomes apparent, then, that laboratories F063, F064, F065, F066, and N158 provided the most comparable and most accurate data for PAH analyses in this study, as the calculated '% flagged' appraisals for these participants were less than 20%. (The 14% flagged value calculated for laboratory N200 does not necessarily reflect good accuracy because this participant submitted 'not detected' results for more than half of the PAH analyses.)

In Appendix IV, the tables for laboratories F040, N196 and N197 reveal that these participants were biased low in their sediment analyses because they had several L and VL flags on samples #1 to #5. Because their data were not flagged as severely on the direct-injection ampules, this would therefore indicate a possible problem with their PAH recoveries rather than one of poor calibration standards. N196 may have also had a problem with their low level calibration standard as many of their results for ampule #9 were flagged H and VH. Laboratory N041 would also be considered to be biased low on both their sediment and ampule analyses, because, despite the numerous 'not detected' data, 17 of 37 results provided by them were flagged L or VL. Laboratories F007 and N208 were biased moderately low because of low results submitted on several specific PAHs.

Laboratories F084, F087, F094 and N107 were each considered to be biased high in their PAH analyses because most or all of their PAH results were flagged H or VH. For laboratory F087, the ampule data submitted was quite good, suggesting that their calibration standards were adequate but that they

may have experienced interference problems or other difficulties in the quantitation of the PAHs in their sediment extracts. Laboratories F084 and F094, on the other hand, likely had low in-house calibration standards since they had received H and VH flags on both their ampule and sediment results. N107 was biased high for all but one of the PAHs in the sediment samples, but they did not submit any data for the direct-injection samples. Laboratories F033 and N090 were biased moderately high because of the numerous H and VH flags that they received on their results for a few of the PAHs in the sediment samples.

Generally, a laboratory's analyses are labelled erratic when they are assigned VH and VL flags for the same parameter on different samples. Their analytical system for a particular parameter is considered to be out-of-control when single, extreme outlying results are reported. Thus, laboratory F057 was deemed to be erratic on several specific PAHs, while N196 was considered to be erratic overall on their PAH analyses in this study because their results for ampule #9 were flagged H and VH while all of their sediment PAH data were flagged L or VL.

Blind duplicate samples were provided so that the in-house precision of the participating laboratories might be assessed. In this study, sediment samples #2 and #5 were the duplicate samples for both the PAH and PCB analyses. From the data in Tables 11 and 14, the most precise laboratories for PAH analysis were F040, F063, F094, N090, N107 and N158 with each achieving less than 15% difference, on average, for all PAH results provided on these two samples. The poorest intralaboratory precision was demonstrated by laboratories F033, N197 and N208. (Laboratories F057, F084, N041, N196 and N200 could not be assessed for precision on their PAH analyses because of incomplete data sets.) Among the individual PAHs, the most precise results were obtained for phenanthrene, fluoranthene, benzo(b)fluoranthene and indeno(1,2,3-c,d)pyrene while those who were erratic in their extraction or cleanup procedures saw this reflected primarily in the recoveries of naphthalene, fluorene, benzo(a)pyrene and benzo(g,h,i)perylene. For those

participants who experienced poor precision of analysis between samples #2 and #5 (laboratories F033, N197 and N208), improved in-house quality control and verification of extraction recoveries with external reference standards and spike solutions are strongly recommended.

It seems apparent then, that the overall interlaboratory results for PAHs were only moderately comparable. As with the data submitted for the PCBs in this study, there was a wide-ranging (more than 3300-fold) difference in the detection limits reported by the participants for PAHs in sediments. (Refer to Table 25.) Nevertheless, the interlaboratory medians, in most cases, were in relatively close agreement to their corresponding reference values and the majority of the data fell within a narrow range of these medians. Some of the participants demonstrated very poor precision on the duplicate sediment samples while a few others exhibited problems with severely biased results for either the direct-injection ampules or the sediment samples or both. This would indicate a general need to reevaluate the quality of their calibration standards and quantitation techniques. Many of the participating laboratories also seemed to have considerably more difficulty analyzing sediment sample #3 since close to 50% of the results were flagged. It is speculated that the presence of high levels of chlorobenzenes and PCBs in this sediment, coupled with the lower PAH concentrations, might have caused interpretation problems to some participants.

Therefore, while a select few of the laboratories need to seriously reevaluate their PAH calibration standards and/or methodologies, many of the participants in this study were able to provide satisfactory and comparable data for the PAHs in these freeze-dried sediment samples. Specifically, laboratories F063 and N158 provided the most accurate and precise data for PAHs in this study, while laboratories F007, F064, F065, F066 and N090 were either slightly less precise, or provided moderately biased data.

Data Evaluation - Trace Metals

All raw data received for the trace metal analyses in this study are summarized in Tables 17 to 24. An existing computer program was used to sort the data by parameter and to arrange them by increasing laboratory and sample number. The mean, standard deviation and median values for each sample are also given in these tables. For the trace metals reported as 'not detected', a 'less than' code was inserted in the data summary. The medians listed were calculated without these 'less than' values, except where the computerized Youden ranking technique had flagged some of the more extreme 'less than' values. The mean and standard deviation were calculated without any data rejection.

Each laboratory in this study analyzed for all eight trace metals. The detection limits reported by the participants for these metals in sediments, are listed in Table 26, along with the guideline levels issued by the Ontario Ministry of the Environment for evaluating dredge spoils for open-water disposal¹. As can be seen in this table, eight laboratories listed a detection limit for cadmium that was at, or above, the guideline level. Laboratories N196 and N197 also listed detection limits above the guideline level for mercury. According to the detection limits provided by the participants in this study, each of the remaining laboratories should have been capable of analyzing sediments for the other seven metals at the level of the MOE guidelines. Therefore, the sediment samples used in this study were selected so as to cover the concentration ranges slightly below, at, and above the MOE guidelines for each of the trace metals of interest.

Two of the sediments that were used in this study were reference materials that had been certified for trace metals. Sample #12 was a sediment certified by the National Water Research Institute of Environment Canada for arsenic, selenium and mercury, and sample #15 was a certified sediment obtained from the National Research Council of Canada. The remaining reference values listed in Table 5 for sediment samples #10 to #14 were based

on numerous in-house and external analyses, and have been confirmed in other interlaboratory round-robin studies, including dredging studies DQC-2⁸ and DQC-4¹⁰.

Overall, most of the trace metal data provided in this study were satisfactory except for sample #15, which was the sediment obtained from NRCC. The recoveries for cadmium, chromium, lead and mercury in this sample were very wide-ranging and showed poor comparability between laboratories. However, since the initiation of this study, the NRCC has withdrawn its certification of mercury in this sediment due to the recent discovery of a contamination problem in a few of the bottles¹⁷. Therefore, the variability in results on this sample may not necessarily be indicative of poor laboratory performance. For the other five sediments, most of the participants provided acceptable data for all eight trace metals, such that, with the exception of the above-mentioned parameters in sample #15, each interlaboratory median was within 25% of its corresponding reference value.

The original instructions for this study requested total metal analysis of the six sediment samples. The results submitted by many of the participants did not appear to reflect this request. (Refer to Table 8 for a listing of the different methods employed by each of the participants for their trace metal analyses). Most digestion procedures for 'total' metal analysis entail complete destruction of the sediment matrix including the silica lattice. The 'extractable' metal dissolution procedures usually extract only the more readily available metals without destruction of the silica lattice. In this study, the digestion procedures varied from complete destruction using HF, HClO₄, and HNO₃, to weak leaching with aqua regia. The more rigorous digestions using hydrofluoric acid yielded the most consistent recoveries. Previous studies have shown that aqua regia digestions, with or without H₂O₂, yielded incomplete recoveries of most metals, especially chromium. For this reason, the participants in this study whose chromium data have been labelled as biased high, or who have received H or VH flags on their chromium results (Table 19), have probably obtained more accurate results than

those whose data were not flagged high. However, because this technique uses the deviation from the interlaboratory medians for calculation, the chromium flags do not accurately reflect the laboratory's performance with respect to their recoveries of total chromium. For this reason, the chromium flags have been excluded in the calculation of the laboratory performances in Tables 29 and 30. To correct this anomaly, the chromium flags could be calculated with respect to the reference value.

Both chromium and cadmium proved to be relatively difficult elements to analyze in this study as the number of flags and the interlaboratory standard deviations were very high for both metals. Despite the larger BAE that was used for chromium (50% at the LLBAE), 47% of the results were flagged. (Refer to Appendix III for an explanation of these terms and the technique of flagging). As discussed above, the variation in these results could be related to the number of different methodologies employed for extraction, with the lower chromium recoveries most likely reflecting incomplete digestion of the sediment samples. (Refer to the list of 'Acids used for Digestion' in Table 19). Therefore, for laboratories F087, N090 and N158, the statements of low bias in their data become even more critical.

Similar to the findings in dredging studies DQC-2⁹ and DQC-4¹⁰, cadmium results were probably the least satisfactory of the eight trace metals in this study in terms of both accuracy and precision. Even after excluding the data for sample #15, the interlaboratory standard deviations averaged 45%, indicating poor comparability among the participants. Furthermore, 16% of the results were submitted as 'not detected' and a third of the remaining results were flagged despite the larger BAE (50% at the LLBAE) assigned to this data set. The poor results may be due, in part, to a combination of high detection limits for cadmium (Table 26) and the low levels of cadmium in the sediment samples under study. Thus, a smaller degree of comparability between the participants was expected and seen. However, as in the case of the chromium data, the wide range in results could also be a reflection of the diversity in the methods employed in extracting the sediments.

Mercury data were the most divergent in this study with an average interlaboratory precision of 80%, indicating poor comparability among the participants. After excluding the data for sample #15, the variability among results remains considerable, for both the higher and lower concentration samples. In addition, the intralaboratory precision for mercury in samples #11 and #14 was the worst of the eight trace metals under study. Nevertheless, less than a quarter of the data were flagged, which suggests that the variability in mercury analysis, while widespread among the study participants, was moderate, and not due to a few extreme results from one or two laboratories.

The interlaboratory results for copper, lead and zinc were excellent as less than 20% of these data were flagged. Very good comparability among the participants was demonstrated by average interlaboratory precisions of better than 20% for each of these metals. In the case of lead, a wider variation in results was obtained on only the very lowest level sample. A previous study, National Interlaboratory QC Study No.35²¹, which had used the same sediments (except for #15 from the NRCC), had shown very little difference between the 'extractable' and 'total' results submitted for cobalt, copper, lead and zinc. In this study, it was therefore not unexpected to find that good 'total' results were achieved by less rigorous digestion procedures than those required to extract a metal that was well-embedded in the silica lattice. Hence, the data in this study confirm that copper, lead and zinc were not well-embedded in the silica lattice of these particular sediment samples.

National Study #35²¹ also suggested that from 85% to 97% of the nickel in sediments #10 to #14 was in the 'extractable' form. As anticipated, the nickel results in this study were relatively accurate and precise despite the variation in methodologies used for extraction. The 30% interlaboratory precision calculated for the nickel results in sediment samples #11 and #14 was likely due to the extremely high concentration of nickel (approximately 900 ug/g) that was present in this sediment from Sudbury.

Lastly, arsenic data were also generally satisfactory in this study except for the erratic results reported by laboratories F064, F066 and F084. None of the participants provided biased data for this metal. Moreover, despite the imprecision of some of the submitted results and the few extreme outlying data points, the interlaboratory medians for arsenic were still very close to their corresponding reference values.

A summary of the performance of the participants in this study for their analyses of the eight trace metals in sediment is presented in Table 29. In this table, the number of results reported, the sum of the results flagged VL, L, H and VH, the percentages of results flagged (both including and excluding the chromium data for reasons described below), and a statement of biases, are presented for each participating laboratory. Laboratory N041 did not send any results for the trace metal portion of this study and laboratory F065 submitted their data well past the extended deadline, after the preliminary data summary had been released. Hence, these latter results could not be included in the interlaboratory evaluations, but have been listed in Appendix II. The remaining results were evaluated for accuracy and bias by the ranking and flagging procedures outlined in Appendix III. These procedures, which semi-quantitatively evaluate data accuracy and bias in a set of results, have been widely used in other interlaboratory studies.

Individual results were flagged VL, L, H or VH when they deviated significantly from the interlaboratory median. The most accurate laboratory was therefore the one with the least number of results flagged. As a comparison with their peers, the percentage of results flagged with respect to the total number of results reported is also given in Table 29. (Please note that the H and L flags are counted as only half of a VH or VL flag in order to add emphasis to the very extreme deviations from the median). This comparison, however, should be applied with caution as some participants did not provide sufficient data for assessment of their performance on all eight trace metals. From Table 29, it can be seen that the most accurate trace metal analyses were conducted by laboratories F007, F033, F040, F057, F094 and

N107. Participants F066, F087, N090 and N208 had slightly higher percentages of flagged results but would also be considered as having produced, on average, relatively accurate results for the trace metals in sediment in this study.

Bias was determined by Youden's ranking technique¹⁸⁻²⁰, as described in Appendix III. The bias statements listed for each laboratory in Table 29 refer to the tendency of their entire set of results for that particular metal to be higher or lower than those of the other participants in the study. Laboratories F063, F064, F087, F094 and N090 each produced biased results for one of the metals under study, while four laboratories received biased statements on more than one of the eight trace metals of interest. The copper and zinc data submitted by laboratory F066 were biased low while their set of mercury data was biased high. Laboratory N158 was biased low on their chromium and nickel results and N197 was deemed to be biased high on both their cadmium and mercury results. Laboratory N200 submitted biased low cadmium data while producing biased high results for chromium, copper and lead. These statements of biased results are strong evidence of systematic errors and are the areas that these laboratories should look to for improvement. The participants who provided unbiased data for all eight trace metals were laboratories F033, F040, F057, F084, N107 and N208.

The intralaboratory precisions of the participants on their trace metal analyses were evaluated in a similar manner to those of the organic analyses in this study. Sediments #11 and #14 were duplicate samples of a sediment reference material from Sudbury in which the concentrations of six of the eight trace metals of interest were well above all of the participants' detection limits, while its cadmium and mercury concentrations were at or above the majority of the reported detection limits. These latter two metals, because of their relatively lower concentration levels, had, as expected, the largest average difference between the duplicate samples, while nickel and copper were the two metals on which the participants obtained the best precision of analysis. When the average repeatability was calculated for each

laboratory on all eight metals, ten participants had achieved better than 10% difference, on average, between the duplicate sample analyses. The most precise laboratories, by these calculations, were F007, F033, F040, F057, F064, F094, N107, N196, N197 and N208.

A laboratory's analyses were labelled erratic when their results for a particular parameter were assigned both VH and VL flags on different samples. In this study, laboratory F084 was deemed to be erratic on their arsenic and zinc analyses, and F087 was erratic on the results they submitted for lead and nickel. Laboratories F064 and F066 also submitted erratic results for arsenic, while N208 submitted some erratic mercury data.

Other than the biased and erratic results described above, satisfactory and accurate data were obtained from most participants for arsenic, copper, lead, nickel and zinc. Moderate difficulties with the analysis of mercury were experienced by many of the laboratories but may have been due, in part, to the low concentration levels in the sediment samples analyzed in this study. Sediment results for chromium and cadmium were indicative of considerably more problems than the other parameters. In agreement with the earlier dredging interlaboratory studies, DQC-2³ and DQC-4¹⁰, cadmium results were neither comparable nor reliable at less than 2 ug/g. The digestion procedures used by several participants were likely the cause for their low recoveries of these two metals as they were not exhaustive enough to recover 'total' chromium and cadmium. The bias high statements for metals other than chromium were likely due to contamination, poor standards or poor background correction. For the majority of participants, the results for copper, lead, nickel and zinc indicated a high degree of accuracy and interlaboratory and intralaboratory precision with only a few minor exceptions. This suggests that reproducible results on the analysis of these trace metals can be expected from most laboratories who participated in this study. Overall, the participants who submitted the most precise, accurate and unbiased data for all eight trace metals, were laboratories F033, F040, F057 and N107. The data submitted by laboratories F007, F063, F064, F094, N090, N158, N196, and N208 were generally satisfactory but showed some degree of imprecision, reduced accuracy and/or bias in the trace metals analyses.

Performance Summary for Total PCB, PAH and Trace Metal Analyses

In order to assess the overall performance of the participants on both their organic and inorganic analyses, a summary was prepared (Table 30) of the bias and precision statements and percentages of flagged results assigned to each laboratory for the three classes under study. This table also lists the 'performance scores' of each laboratory and their 'overall performance rating' on the organic and inorganic samples as determined by the procedure described in Appendix III. These assessments can serve as a rough indication of the overall accuracy of the PCB, PAH and trace metal data since lower percentages of results flagged reflect more accurate results. In this study, seven laboratories (F007, F063, F064, F087, N041, N158 and N200) received a 'satisfactory' performance score of less than 20 and thus were considered to be the most accurate. Another group of nine laboratories received performance scores between 20 and 40 and were therefore declared to be 'moderate' with respect to accuracy in their overall performance. A final group of three laboratories (F084, N196 and N197) obtained performance scores greater than 40, which assigned them an overall performance rating of 'poor' in terms of accuracy for the three classes of parameter in this study.

Our scoring system for the preparation of Table 30 is tentative and is based on established flagging techniques which use the recoveries or deviations of the results relative to the interlaboratory medians as a basis to provide a semi-quantitative evaluation of the accuracy of the data. Thus, each laboratory was rated for accuracy relative to the performance of the other participants. However, there are several limitations to this technique. When scrutinizing the data in more detail, it becomes apparent that participants F007, F033, F065, N041, N090, N107 and N196 did not provide complete sets of data for all parameters or samples, so that their performance scores do not reflect their capabilities on the full scope of samples provided in this study. Also, laboratories F057, F084, N041 and N200 submitted a significant proportion of their organic data as 'not detected'. These limitations in a participant's detection capabilities, as well as any statements of bias and imprecision, or the presence of erratic results, were

not considered in the assignment of overall performance ratings. Finally, despite the fact that the analyses for each of the three classes were of a different nature, they were averaged on an equal basis when calculating the performance scores. Therefore, while these ratings are generally indicative of accuracy, it is not sufficient to use the assessment of the participants in Table 30 as the sole criterion for evaluation of their performance.

Of the seven participants in Table 30 who received a satisfactory rating with respect to accuracy, three laboratories (F007, N041 and N200) provided incomplete or insufficient data in this study. Furthermore, the PCB data submitted by laboratory F007, were severely imprecise for the duplicate sediment samples, and poor recoveries were seen in several of their PAH results. Both N041 and N200 submitted more than 50% of their PAH results as 'not detected' while demonstrating poor recoveries on much of their remaining PAH data. Laboratory F087 also submitted a considerable number of 'not detected' results, but their performance shortcomings were more apparent in their biased high and out-of-control results submitted for several of the PAHs. Therefore, of the seven laboratories who were assigned a 'satisfactory' overall performance rating with respect to accuracy, only laboratories F063, F064 and N158 did not demonstrate any severe problems in the analysis of the three classes under study. Several of the participants who received a 'moderate' assessment rating, would also be considered to have generally performed well, but, as seen in Table 30, their data for at least one of the three classes of parameter under study were either incomplete, imprecise, or demonstrated strong bias.

Clearly then, no one laboratory performed exceptionally well in their analyses for all three classes of parameter in this study. Overall, laboratories F063, F064 and N158 submitted the best results for PCBs, PAHs and trace metals, as their data revealed only minor problems with imprecision or bias in some of the samples. Laboratories F007, F094, N090, N107 and N208 performed well on two of the three classes, but provided incomplete or somewhat less satisfactory results for the third set of parameters. All other participants in this study demonstrated more severe problems with their analyses of at least two of the three classes of parameters in this study.

CONCLUSION

The results of this round-robin study indicated that there are some laboratories capable of accurately analyzing for PAHs, PCBs, and trace metals in sediments. However, the study also revealed some extreme outlying and/or very erratic results produced by certain participating laboratories for total PCBs, arsenic and PAHs as well as some common, but less severe problems with the analysis of certain PAHs, chromium, cadmium and mercury.

The PCB data were, for the most part, satisfactory in this study. The interlaboratory medians of the seven test samples agreed closely with the reference values and good comparability between most laboratories was seen with only a few participants in this study providing outlying results. Since the concentrations of PCBs in these naturally-contaminated sediments have been established by extensive in-house analyses and further confirmed by several interlaboratory studies, the comparison of a participant's results to the reference values is a good indication of accuracy. The calculated recoveries listed in Appendix IV would also, therefore, reflect well the true capabilities of a laboratory, for total PCB analysis in real sediment samples at such levels. Precision was not as good as expected in this study, in that less than half the participants were able to achieve better than 25% precision of analysis between the duplicate sediment samples. The detection limits reported by the laboratories for total PCBs in sediments spanned a 300-fold range with one-third of the participants reporting detection limits that were at, or above, the 50 ng/g level listed by the Ontario Ministry of the Environment in their guidelines for evaluating dredge spoils for open water disposal³. Nevertheless, the results from this study indicate that one could place confidence in the PCB data being produced by the majority of the participating laboratories in this study.

Many of the participants produced satisfactory and comparable PAH data in this study, but some erratic, heavily biased, or out-of-control results were also observed. Incomplete sets of results were submitted by four participants, while another four reported more than a third of their results

as 'not detected'. Again, as with the PCBs in this study, there was a very wide range (more than 3000-fold) in the detection limits reported by the participants. Nevertheless, for those who did submit a complete set of PAH results, the majority of their sediment data were satisfactory in comparison to both the medians and the reference values. Furthermore, the precision of analysis for the PAHs was considerably better than that obtained for the PCBs, despite the fact that the same sediment samples were used for both determinations. Most of the laboratories who provided biased or imprecise results appeared to have experienced difficulties with recovering the PAHs from the sediments rather than with poor calibration standards. However, for those who performed poorly on both the ampule and the sediment analyses, their wide range of results and poor precision for several of these parameters would suggest a need for more accurate analytical standard solutions as well as external reference solutions with which they could monitor their accuracy. Therefore, while several of the participants were able to provide precise, accurate and relatively unbiased PAH data, the strongly biased, erratic and out-of-control results submitted by some of the other participants confirm our previous experience that one should interpret the data for these compounds with caution.

There are a wide variety of methods currently in use for the analysis of trace metals in sediment and this is illustrated well in this study. However, while the instructions for these samples requested 'total' metal analysis, the list of methodologies used by the participants reveals that several laboratories were employing digestion techniques that would not have been exhaustive enough to recover any more than the 'extractable' metal content. For those metals which had a significant proportion of their total content well-embedded in the silica lattice of the sediment, these less exhaustive procedures could have produced low recoveries. Nevertheless, the interlaboratory medians for the sediment samples were for the most part, in good agreement with the reference values and, for most parameters, the range of results was quite reasonable. The results for copper, lead, nickel and zinc demonstrated a high degree of accuracy and interlaboratory and intralaboratory precision with only a few minor exceptions. Chromium results

were less comparable, due in part, most likely, to the diversity in extraction methods. Moderate difficulties with the analysis of mercury were experienced by many of the laboratories especially at the very low concentration levels in some of the sediment samples analyzed in this study. Cadmium results were the least satisfactory of the trace metal data, being neither comparable, nor reliable at less than 2 ug/g. In addition, more than a third of the participants reported detection limits for cadmium that would not have enabled them to quantitate this metal at the guideline level issued by the Ontario Ministry of the Environment.

In summary, no laboratory performed exceptionally well on all of their analyses for PCBs, PAHs and trace metals in sediment. However, three participants (F063, F064 and N158) performed significantly better, on average, and an additional four (F007, F094, N107, and N208) were considered to have performed adequately well, having demonstrated only moderate difficulties with bias or imprecision in some of the samples. All other participants in this study appeared to have experienced more severe problems with their analyses of at least two of the three classes under study. Three laboratories, in particular, were so inaccurate, erratic or heavily biased on some of their analyses that it is recommended that they closely reevaluate the data being produced by their laboratory for the analyses of these particular classes of compound.

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Table 1. List of Participants in Interlaboratory Study DQC-5.

Federal Government:

- | | |
|---|----------------------------------|
| 1. Environment Canada
C&P (EPS) Laboratory Services
River Road Environmental Technology Centre
Ottawa, Ontario | No results submitted for
PCBs |
| 2. Environment Canada
C&P (EPS) Laboratory Services
Wastewater Technology Centre
Burlington, Ontario | Incomplete PAH results |
| 3. Environment Canada
National Water Quality Laboratory
Burlington, Ontario | |

Provincial Governments:

- | | |
|--|--|
| 4. Ontario Ministry of the Environment
Trace Organics QA/QC Section
Rexdale, Ontario | |
|--|--|

Private Laboratories:

- | | |
|---|-----------------------------------|
| 5. (a) AccuTest Laboratories Ltd. (inorganics)
Nepean, Ontario | No results submitted |
| (b) Paracel Labs (organic analyses)
Nepean, Ontario | No results submitted |
| 6. Acres Analytical Ltd.
Niagara Falls, Ontario | |
| 7. Areco Canada Inc.
Nepean, Ontario | Incomplete PAH and PCB
results |
| 8. Barringer Laboratories
Mississauga, Ontario | |
| 9. Beak Consultants Ltd.
Mississauga, Ontario | No results submitted |
| 10. Bondar-Clegg & Co. Ltd.
Ottawa, Ontario | |
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continued

Table 1 (continued). List of Participants in Interlaboratory Study DOC-5.

11. Canviro Analytical Laboratories Ltd. Kitchener, Ontario	
12. Clayton Environmental Consultants Ltd. Windsor, Ontario	
13. DILLON Environmental Laboratories Mississauga, Ontario	
14. Eco-Recherches Pointe-Cl�aire, P.Q.	No results submitted for trace metals
15. Enviroclean London, Ontario	
16. Environmental Applications Group Markham, Ontario	Incomplete PAH results
17. Fine Analysis Laboratories Hannon, Ontario	No results submitted
18. Mann Testing Laboratories Mississauga, Ontario	Late results submitted for trace metals, see Appendix II
19. OceanChem Group Dartmouth, Nova Scotia	
20. Proctor & Redfern Ltd. Don Mills, Ontario	
21. Technical Services Laboratories Mississauga, Ontario	
22. Zenon Environmental Inc. Burlington, Ontario	

Table 2. Description of Samples.

Sample #	Identification Code	Origin
1	TH-1	Toronto Harbour
2	EC-3	Niagara River Plume
3	EC-2	Lake Ontario
4	HR-1	Humber River
5	EC-3	Niagara River Plume
6	EC-107	standard solution of 1:1:1 mixture of Aroclors 1242, 1254 and 1260 in isooctane
7	EC-109	standard solution of 1:1:1 mixture of Aroclors 1242, 1254 and 1260 in isooctane
8A	EC-417	standard solution of 18 PAHs in toluene (prepared from neat materials)
8B	EC-423	standard solution of 18 PAHs in acetonitrile (prepared from neat materials)
9A	EC-420	standard solution of 18 PAHs in toluene (prepared from neat materials)
9B	EC-426	standard solution of 18 PAHs in acetonitrile (prepared from neat materials)
10	TH-2	Toronto Harbour
11	SUD-1	Sudbury
12	WQB-1	Lake Ontario
13	HR-1	Humber River
14	SUD-1	Sudbury
15	BCSS-1	Gulf of St. Lawrence: Baie des Chaleurs

Note 1: All samples prepared at the National Water Research Institute except for BCSS-1 which is a reference material from the National Research Council of Canada.

Note 2: Sediments 2 and 5 and sediments 11 and 14 are pairs of duplicate freeze-dried sediments for the organic and inorganic analyses, respectively.

Table 3. Reference Values and Interlaboratory Medians for PAHs and PCBs in sediment samples. (All values in ng/g).

Parameter	Sediment #1		Reference Value		Sediments #2 & #5		Reference Value		Sediment #5	
	Median	% Recovery	Value		Median #2	% Recovery #2	Median #5	% Recovery #5	Median #5	% Recovery #5
Naphthalene	127	---	---	---	120	---	111	---	111	---
Acenaphthylene	79.4	---	---	---	33	---	30	---	30	---
Acenaphthene	46	---	---	---	34	---	34	---	34	---
Fluorene	61	---	---	---	30	---	28	---	28	---
Phenanthrene	489	80	293	---	280	96	281	96	281	96
Anthracene	111	99	58	---	66.6	115	71	122	71	122
Fluoranthene	1030	96	579	---	357	62	431	74	431	74
Pyrene	788	70	466	---	324	70	443	95	443	95
Benzo (a) anthracene	449	70	323	---	246	76	284	88	284	88
Chrysene/Triphenylene	530	57	456	---	281	62	290	64	290	64
Benzo (b) fluoranthene	780	94	578	---	306	53	467	81	467	81
Benzo (k) fluoranthene	320	65	260	---	178	68	218	84	218	84
Benzo (a) pyrene	547	79	408	---	352	86	388	95	388	95
Indeno (123-cd) pyrene	665	105	480	---	386	80	414	86	414	86
Dibenzo (ah) anthracene	200	101	119	---	126	106	145	122	145	122
Benzo (ghi) perylene	607	93	493	---	300	61	370	75	370	75
Total PCBs	500.5	91	822	---	725.5	88	805	---	805	98

Parameter	Sediment #3		Reference Value		Sediment #4		Reference Value	
	Median	% Recovery	Value		Median	% Recovery	Value	
Naphthalene	2570	---	---	---	81.4	---	---	---
Acenaphthylene	363	---	---	---	30	---	---	---
Acenaphthene	68	---	---	---	47.3	---	---	---
Fluorene	500	---	---	---	50	---	---	---
Phenanthrene	1630	114	528	---	468	89	---	---
Anthracene	220	195	104	---	108	104	---	---
Fluoranthene	2505	72	817	---	580	71	---	---
Pyrene	1780	60	829	---	552	67	---	---
Benzo (a) anthracene	927	69	427	---	359	84	---	---
Chrysene/Triphenylene	1069	58	512	---	320	63	---	---
Benzo (b) fluoranthene	1810	81	466	---	526	113	---	---
Benzo (k) fluoranthene	1190	104	262	---	211	81	---	---
Benzo (a) pyrene	1006	98	426	---	440	103	---	---
Indeno (123-cd) pyrene	1795	100	332	---	400	120	---	---
Dibenzo (ah) anthracene	602	108	111	---	120	108	---	---
Benzo (ghi) perylene	1312	84	331	---	360	109	---	---
Total PCBs	930	76	544	---	420	77	---	---

Table 4. Reference Values and Interlaboratory Medians for PAHs and PCBs in Ampule Solutions.

Parameter	Ampule #6 (pg/ul)		Ampule #7 (pg/ul)		Design Value	Design Value	Ampule #8 (ng/ul)		Ampule #9 (ng/ul)		
	Design Value	Median	Median	Median			Design Value	Median	Design Value	Median	Design Value
Total PCBs	200	175.5	88	60	61	102					
Parameter	Design Value	Median	#8A	Median	#8B	% Recovery	Design Value	Median	#9A	Median	% Recovery
Naphthalene	19.89	15.74	79	27.7	139	0.497	0.497	0.444	89	0.49	99
Acenaphthylene	9.87	8.60	87	28.1	285	0.493	0.493	0.38	77	1.39	282
Acenaphthene	0.945	1.20	127	22.4	2370	0.472	0.472	0.43	91	0.38	80
Fluorene	37.84	30.9	82	37.0	98	0.473	0.473	0.322	68	0.38	80
Phenanthrene	16.05	13.8	86	16	100	0.502	0.502	0.1	80	0.5	100
Anthracene	0.974	0.93	95	2.36	242	0.0973	0.0973	0.1	103	0.20	206
Fluoranthene	39.06	27.1	69	39	100	0.976	0.976	0.64	66	0.87	89
Pyrene	29.48	25.72	87	30	102	0.983	0.983	0.67	68	0.95	97
Benzo(a)anthracene	11.54	5.74	50	6.80	59	0.481	0.481	0.21	44	0.28	58
Chrysene/Triphenylene	19.17	14.68	77	16	83	0.767	0.767	0.42	55	0.64	83
Benzo(b)fluoranthene	19.03	13.8	73	24.1	127	0.571	0.571	0.27	47	1.0	175
Benzo(k)fluoranthene	9.10	5.65	62	7.5	82	0.364	0.364	0.2	55	0.30	82
Benzo(a)pyrene	9.69	8.51	88	9.8	101	0.484	0.484	0.325	67	0.49	101
Indeno(123-cd)pyrene	15.70	14	89	15.9	101	0.491	0.491	0.260	53	2.15	438
Dibenzo(ah)anthracene	3.95	3.02	76	8.95	227	0.0987	0.0987	0.083	84	1.35	1368
Benzo(ghi)perylene	13.97	14.4	103	15.9	114	0.437	0.437	0.285	65	0.75	172

8A, 9A - GC data for PAHs in toluene
8B, 9B - HPLC data for PAHs in acetonitrile

Table 5. Reference Values and Interlaboratory Medians for Trace Metals in sediment samples. (All values in ug/g).

Parameter	Sediment #10		Sediments #11 & #14		Sediment #15	
	Reference Value	Median	Reference Value	Median	Reference Value	Median
Chromium	---	104	---	68	---	---
Nickel	44.2	41	941	922	921	98
Copper	105.0	120	565.0	570	565	100
Zinc	---	893	771	785	750	97
Arsenic	9.7	8.6	32.5	32	31	95
Cadmium	5.30	5.5	2.30	1.75	1.76	77
Mercury	---	0.605	0.11	0.103	0.093	85
Lead	---	189	58.0	51	53	91

Parameter	Sediment #12		Sediment #13		Sediment #15	
	Reference Value	Median	Reference Value	Median	Reference Value	Median
Chromium	---	61	---	116	123	54
Nickel	59.3	55	38.5	34	55.3	47
Copper	80.0	76.4	81.0	77	18.5	17
Zinc	279.0	260	1100	1080	119	101
Arsenic	23.00	22.8	6.85	5.85	11.1	9.8
Cadmium	2.10	1.9	3.80	3.75	0.25	0.40
Mercury	1.09	1.04	0.35	0.365	0.129	0.160
Lead	85.7	84	143.5	139	22.7	24

Table 6. Summary of Methodologies for the Analysis of PCBs in Sediments.

Lab. No.	Extraction & Cleanup	Analysis & Comments
F007	-soxhlet extraction with acetone-hexane (59:41); base partition with 2% KHCO ₃ ; back-extract into hexane; Florisil column cleanup; Hg cleanup	dual GC-ECD, 30m DB-5 and 30m DB-17
F033	-coextract with PAHs ultrasonically using 1:1 acetone:hexane; wash with XAD mill-Q water, then CH ₂ Cl ₂ ; dry through Na ₂ SO ₄ ; silica gel column cleanup	dual GC-ECD, 30m SPB-1 and 30m SPB-5
F040	-coextract with PAHs by ultrasonic extraction using acetone; wash with water, then CH ₂ Cl ₂ ; Florisil column cleanup	dual GC-ECD, 15m DB-5 and 15m DB-1701
F057	-coextract with PAHs by ultrasonic extraction with carbon disulfide	dual GC-ECD, 30m DB-1 and 30m DB-1701
F063	-extract with acetone on a mechanical shaker; Florisil column cleanup	dual GC-ECD, 30m DB-5 and 30m DB-1701
F064	-extraction on a wrist shaker with 1:1 acetone:hexane; Florisil mini column cleanup	GC-ECD, 6ft OV-1
F065	-extract with hexane by ultrasonication; dry through Na ₂ SO ₄ ; cleanup using Florisil, silica and acid silica	GC-ECD, 30m DB-17
F066	-extract twice with 50/50 acetone/hexane on a wrist shaker; dry through Na ₂ SO ₄ ; silica gel column cleanup; cleanup with copper	GC-ECD, 30m DB-1
F084	-extract five times with hexane on a vortex mixer; Florisil column cleanup	GC-ECD, 60m DB-5
F087	-coextract with PAHs with 50:50 acetone:hexane ultrasonically three times; silica gel solid phase extraction tube cleanup; Florisil solid phase extraction tube cleanup; further cleanup with H ₂ SO ₄ then Hg	GC-ECD, 30m SPB-5
F094	-coextract with PAHs by soxhlet extraction with CH ₂ Cl ₂ ; Florisil column cleanup	GC-ECD, megabore HP-1
N041	-extract three times with acetone and hexane ultrasonically; cleanup with H ₂ SO ₄	GC-ECD, 6ft OV-1
N090	-coextract with PAHs using CH ₂ Cl ₂ ; silica gel and alumina column cleanups; Hg cleanup	GC-MSD, 30m DB-5
N107	-soxhlet extraction with hexane/acetone; washed with NaCl/H ₂ O; dry through Na ₂ SO ₄ ; Florisil column cleanup; Hg cleanup	GC-ECD, 25m DB-5
N158	-coextract with PAHs with CH ₂ Cl ₂ by ultrasonication; dry through Na ₂ SO ₄	GC-MSD, 30m DB-5
N196	-soxhlet extraction with CH ₂ Cl ₂ ; dry through Na ₂ SO ₄ ; Florisil solid phase extraction tube cleanup	GC-ECD, 15m DB-5
N197	-soxhlet extraction with hexane/acetone; Florisil column cleanup; cleanup with H ₂ SO ₄	GC-ECD, 4ft OV-101
N200	-extract with cyclohexane on a vortex mixer; silica gel column cleanup; Hg cleanup	GC-ECD, 15m SPB-5
N208	-soxhlet extraction with CH ₂ Cl ₂ ; Florisil column cleanup	GC-MSD, 30m DB-1

Table 7. Summary of Methodologies for the Analysis of PAHs in Sediments.

Lab. No.	Extraction & Cleanup	Analysis & Comments
F007	-soxhlet extraction with acetone-hexane (59:41); base partition with 2% KHCO ₃ ; back-extract into hexane; dry through Na ₂ SO ₄	GC/MS
F033	-coextract with PCBs ultrasonically using 1:1 acetone:hexane; wash with XAD milli-Q water, then CH ₂ Cl ₂ ; dry through Na ₂ SO ₄ ; silica gel column cleanup	GC-MSD, 30m DB-5
F040	-coextract with PCBs by ultrasonic extraction using acetone; silica gel and alumina column cleanups	GC-MSD, 60m DB-5
F057	-coextract with PCBs by ultrasonic extraction with carbon disulfide	GC/MS
F063	-extract with 50:50 acetone:CH ₂ Cl ₂ on a mechanical shaker	GC/MS, 30m DB-5
F064	-extract with acetone/hexane on a wrist shaker; SEP PAK and alumina cleanups	GC/MS, 30m DB-1
F065	-extract with 1:1 acetone:hexane by ultrasonication; silica gel column cleanup	GC-MSD, 30m DB-5
F066	-extract twice by soxhlet extraction with CH ₂ Cl ₂ ; dry through Na ₂ SO ₄	GC/MS
F084	-extract five times with CH ₂ Cl ₂ ultrasonically; dry through Na ₂ SO ₄ ; silica gel column cleanup	GC-FID, 30m DB-5
F087	-coextract with PCBs with 50:50 acetone:hexane ultrasonically three times; silica gel solid phase extraction tube cleanup	GC-FID, 30m SPB-5
F094	-coextract with PCBs by soxhlet extraction with CH ₂ Cl ₂	GC-MSD, 30m DB-5; Results corrected for recoveries
N041	-extract three times with CH ₂ Cl ₂ ultrasonically; dry through Na ₂ SO ₄	GC-FID, 30m DB-5
N090	-coextract with PCBs using CH ₂ Cl ₂ ; silica gel column cleanup	GC-MSD, 30m DB-5
N107	-soxhlet extraction with CH ₂ Cl ₂ ; dry through Na ₂ SO ₄	GC-MSD, 12m DB-1
N158	-coextract with PCBs with CH ₂ Cl ₂ by ultrasonication; dry through Na ₂ SO ₄	GC-MSD, 30m DB-5
N196	-soxhlet extraction with CH ₂ Cl ₂ ; dry through Na ₂ SO ₄ ; silica gel solid phase extraction tube cleanup	HPLC-UV
N197	-ultrasonic extraction with CH ₂ Cl ₂ ; silica gel column cleanup	HPLC-UV; first 10 PAHs HPLC-Fluorescence; last 6 PAHs
N200	-ultrasonic extraction with CH ₂ Cl ₂ ; silica gel column cleanup	HPLC-Fluorescence
N208	-soxhlet extraction with CH ₂ Cl ₂ ; silica gel column cleanup	GC-MSD, 30m DB-1

Table 8. Summary of Methodologies for the Analysis of Trace Metals in Sediments.

Lab. No.	Digestion	Detection & Comments
F007	-Pb, Zn, Cr, Cu, Cd, Ni: digest with HF, aqua regia and H ₂ O ₂ -Hg: hot block digestion with HNO ₃ , H ₂ SO ₄ and V ₂ O ₅ ; SnCl ₂ reduction -As: NaOH fusion; HCl dissolution of the flux	DCP cold vapour AAS hydride AAS
F033	-Pb, Zn, Cr, Cu, Cd, Ni: digest with HF, HClO ₄ and HNO ₃ -Hg: digest with HNO ₃ , H ₂ SO ₄ , KMnO ₄ and K ₂ S ₂ O ₈ -As: alkaline fusion	flame AAS cold vapour AAS hydride formation, ICP
F040	-Pb, Zn, Cr, Cu, Cd, Ni: hot block digestion with aqua regia -Hg: digest with HNO ₃ and H ₂ SO ₄ ; SnCl ₂ reduction -As: digest with HClO ₄ , HNO ₃ and H ₂ SO ₄ ; NaBH ₄ reduction	ICP-AES cold vapour mercury monitor hydride generation, flameless AAS
F057	-Pb, Zn, Cr, Cu, Cd, Ni: digest with HF, HCl, HClO ₄ and HNO ₃ -Hg: digest with HNO ₃ -As: digest with HClO ₄ and HNO ₃ ; KI and SnCl ₂ reduction	flame AAS cold vapour AAS hydride generation, colorimetric
F063	-Pb, Zn, Cr, Cu, Cd, Ni, As: digest with aqua regia -Hg: digest with HNO ₃ , H ₂ SO ₄ , KMnO ₄ and K ₂ S ₂ O ₈	flame AAS (Pb); ICAP; hydride generation, AAS (As) cold vapour AAS
F064	-Pb, Zn, Cr, Cu, Cd, Ni, As: digest with aqua regia -Hg: digest with aqua regia and KMnO ₄ ; SnCl ₂ reduction	ICP-AES; GFAAS (As) cold vapour AAS
F065	-Pb, Zn, Cr, Cu, Cd, Ni, As: digest with aqua regia -Hg: digest with HClO ₄ , HNO ₃ and H ₂ SO ₄	ICAP; GFAAS (As) cold vapour AAS
F066	-all metals: digest with aqua regia	ICP
F084	-Pb, Zn, Cr, Cu, Cd, Ni: digest with HF, HClO ₄ and HNO ₃ -Hg: digest with HNO ₃ , H ₂ SO ₄ , HCl, KMnO ₄ and K ₂ S ₂ O ₈ ; hydroxylamine sulphate-NaCl; SnSO ₄ reduction -As: digest with HF, HClO ₄ , HNO ₃ , KMnO ₄ and K ₂ S ₂ O ₈	flame AAS cold vapour AAS hydride, flameless AAS
F087	-Pb, Zn, Cr, Cu, Cd, Ni: digest with aqua regia -Hg: digest with HNO ₃ , H ₂ SO ₄ , KMnO ₄ and K ₂ S ₂ O ₈ -As: digest with HNO ₃ and H ₂ SO ₄	flame AAS cold vapour AAS hydride generation, AAS
F094	-Pb, Zn, Cr, Cu, Cd, Ni, As: digest with HF, HClO ₄ and HNO ₃ -Hg: digest with HNO ₃ and H ₂ SO ₄ and KMnO ₄ ; hydroxylamine-HCl reduction; SnCl ₂ reduction	flame AAS; GFAAS (Pb & Cd, low level); hydride generation, AAS (As) cold vapour AAS

continued

Table 8 (continued). Summary of Methodologies for the Analysis of Trace Metals in Sediments.

Lab. No.	Digestion	Detection & Comments
N090	-Pb, Zn, Cr, Cu, Cd, Ni: digest with HNO ₃ and H ₂ O ₂ then HCl -Hg: digest with aqua regia then KMnO ₄ ; NaCl-hydroxylamine sulphate; SnSO ₄ reduction -As: digest with HNO ₃ and H ₂ SO ₄ ; KI and SnCl ₂ reduction	flame AAS cold vapour AAS hydride generation, AAS
N107	-Pb, Zn, Cu, Cd and Ni: digest with aqua regia -Cr: digest with HF and aqua regia -Hg: digest with HCl, HNO ₃ , H ₂ SO ₄ , KMnO ₄ and K ₂ S ₂ O ₈ , -As: digest with HF, HCl and HNO ₃	AAS AAS cold vapour AAS hydride generation, AAS
N158	-Pb, Zn, Cr, Cu, Cd and Ni: aqua regia and H ₂ O ₂ -Hg: digest with HNO ₃ and H ₂ SO ₄ , KMnO ₄ and K ₂ S ₂ O ₈ ; NaCl-hydroxylamine sulphate; SnSO ₄ reduction -As: digest with HCl, HNO ₃ and aqua regia	ICAP cold vapour AAS hydride generation, ICP
N196	-Pb, Zn, Cr, Cu, Cd and Ni: digest with HF, HCl, HClO ₄ and HNO ₃ -Hg, As: aqua regia	ICP ICP; hydride generation-ICP(As)
N197	-Pb, Zn, Cr, Cu, Cd and Ni: digest with HCl, HNO ₃ and H ₂ O ₂ -Hg: digest with HNO ₃ and H ₂ SO ₄ ; KMnO ₄ and NaCl-hydroxylamine sulphate -As: HCl, HClO ₄ and H ₂ SO ₄	flame AAS cold vapour AAS hydride generation, AAS
N200	-all metals: digest with aqua regia	ICP; cold vapour AAS(Hg); hydride generation-ICP(As)
N208	-Pb, Zn, Cr, Cu and Ni: digest with HNO ₃ and H ₂ O ₂ -Cd, Hg, As: digest with aqua regia	ICP-AES flame AAS(Cd); cold vapour AAS(Hg); hydride generation, AAS(As) QC: CRM calibration verification

Table 9. PCB results for Sediment Samples #1-5 and Ampules 6 & 7.

Lab No.	Total PCBs						
	Sediment Sample (ng/g)					Ampules (pg/ul)	
	1	2	3	4	5	6	7
F007	456.8	1072.4	1560.9	450.5	594.0	148.8	48.4
F033	511	661	1820	420	2070	198	64.4
F040	210	265	480	200	280	125	47
F057	376	15415	25169	388	11699	233.1	63.3
F063	402	500	740	417	700	137	46
F064	737	800	1190	599	669	107	306
F065	490	1400	810	400	1500	71	nd
F066	699	1120	1380	984	1140	349	170
F084	620	790	930	620	1300	0.03	<0.01
F087	714	889	864	576	805	208	75
F094	530	880	1030	390	1500	190	67
N041	590	580	670	580	560	200	<100
N090	-	-	-	-	-	-	-
N107	548	896	1170	534	887	181	60
N158	560	587	835	530	620	182	42.3
N196	32.1	25.8	-	-	-	171	48.7
N197	370	170	29000	330	7900	69	<50
N200	200	420	580	360	580	180	62
N208	300	260	360	330	300	130	53
<hr/>							
Interlab							
Median	500.5	725.5	930	420	805	175.5	61
<hr/>							
Reference							
Value	552	822	1220	544	822	200	60

For an explanation of terms and symbols, see Appendix III.

Table 10. PAH results for Sediment Sample #1 (ng/g).

Lab No.	Parameter															
	Naph-	Acen-	Ace-	Fl-Phen-	Anth-	Fluor-	Pyrene	B(a)A	Chrys-	B(b)F	B(k)F	B(a)P	I(cd)P	D(ah)A	B(ghi)P	
F007	276	nd	75	78	700	151	790	877	556	530	754	414	613	746	373	973
F033	82.7	-	-	127	518	92.8	1030	1000	763	1020	941	1360	1390	-	-	-
F040	36	nd	nd	nd	130	31	245	230	95	140	160	153	110	200	35	157
F057	<330	<330	<330	<330	977	510	1580	1240	348	749	1104	1540	973	1170	556	613
F063	129	163	35	58	477	177	1540	1640	473	424	(903)**	480	345	143	336	
F064	83	42	32	61	611	111	1030	1000	355	654	593	267	368	250	72	282
F065	143	83.7	40.9	36.8	439	96.3	653	693	158	169	(373)**	162	288	81.1	361	
F066	120	51	25	56	500	86	610	610	660	610	(1100)**	860	710	240	810	
F084	<100	<100	<100	<100	450	<100	1400	1700	(4200)**	(5700)**	<200	(4900)**			3800	
F087	180	750	170	720	190	<100	1090	470	330	280	780	1090	4280	490	<100	2300
F094	370	75	55	126.7	755	120	1115	1120	860	624.45	(2140)**	1310	1110	200	1100	
H041	<20	<20	<20	<30	80	<40	<40	300	<70	<80	<70	<60	470	<160	<220	<220
H090	-	18	46	21	653	264	1288	1269	424	968	(1886)**	762	689	109	370	
H107	312	123	98	88	796	192	1305	1324	764	1078	1495	320	1125	665	252	780
H158	125	121	47.4	68.9	571	147	687	699	477	495	663	686	450	745	148	544
H196	9.38	nd	16.4	2.72	4.17	2.48	25.9	16.7	nd	25.7	12.5	2.18	30.9	3.17	nd	12.3
H197	240	<100	200	<10	100	20	670	470	230	310	790	240	440	1100	3800	1200
H200	<100	<100	<100	<100	<100	<100	1090	<100	380	260	<100	280	710	(<200)**	<100	
H208	59	14	10	23	270	34	650	660	540	540	920	320	430	640	200	600
Interlab																
Median	127	79.4	46	61	489	111	1030	788	449	530	780	320	547	665	200	607
Reference																
Value	---	---	---	---	611	112	1071	1118	640	923*	826	493	696	635	199	655

For an explanation of terms and symbols for tables 10 - 16, see Appendix III.

Table 11. PAH results for Sediment Sample #2 (ng/g).

Lab No.	Parameter															
	Naph-	Acen-	Ace-	Fl- Phen-	Anth-	Fluor-	Pyrene	B(a)A	Chrys-	B(b)F	B(k)F	B(a)P	I(cd)P	D(ab)A	B(ghi)P	
F007	288	21	48	43	431	97	391	327	172	233	306	146	204	262	113	354
F033	nd	-	-	nd	289	71.3	357	367	434	416	726	489	613	-	-	-
F040	28	nd	nd	nd	62	22	125	104	55	82	120	130	86	85	nd	75
F057	2140	<330	<330	<330	<330	<330	624	352	<330	<330	572	<330	<330	<330	<330	<330
F063	83	48	21	27	245	61	743	652	263	243	(585)**	349	282	113	313	
F064	69	16	17	111	284	56	573	476	227	405	463	209	333	262	88	300
F065	136	39.2	35.9	28.5	262	54.4	312	257	339	354	(727)**	354	800	126	992	
F066	120	36	17	21	280	49	350	320	380	380	(750)**	460	570	170	63	
F084	<100	<100	130	<100	200	700	300	700	(600)**	(700)**	500	(600)**	400			
F087	310	<100	<100	300	210	210	660	280	300	760	(1180)**	4090	460	<100	1230	
F094	340	30	40	70	350	70	580	540	440	260	(1740)**	1210	910	190	1010	
N041	<20	<20	<20	<30	<30	60	<40	170	70	<80	<70	<60	120	<150	<210	<210
N090	-	16	26	18	334	164	668	513	238	458	(1190)**	518	588	200	299	
N107	252	92	59	49	424	106	713	614	490	652	950	355	728	578	179	655
N158	99.4	71.1	32.1	34.6	338	66.6	349	310	253	235	(819)**	100	442	82.4	300	
N196	2.44	nd	nd	11.1	2.36	nd	29.2	8.62	nd	21.7	8.07	nd	19.9	2.49	nd	12.2
N197	<90	<100	100	30	<9	<9	140	29	150	62	210	52	100	330	600	230
N200	<100	<100	<100	<100	<100	<100	<100	<100	214	302	<100	392	471	(<200)**	<100	
N208	5	4	3	5	73	6	160	130	120	150	300	105	70	290	75	260
Interlab																
Median	120	33	34	30	280	66.6	357	324	246	281	306	178	352	386	126	300
Reference Value																
Value	---	---	---	---	293	58	579	466	323	456*	578	260	408	480	119	493

Table 12. PAH results for Sediment Sample #3 (ng/g).

Lab No.	Parameter															
	Naph-	Acen-	Ace-	Fl-	Phen-	Anth-	Fluor-	Pyrene	B(a)A	Chrys-	B(b)F	B(k)F	B(a)P	I(cd)P	D(ab)A	B(ghi)P
F007	5651	108	60	nd	1799	896	2022	439	112	171	141	141	66	93	nd	124
F033	1600	-	-	230	1630	83.2	2100	1970	991	2080	3560	3560	3430	-	-	-
F040	525	60	nd	55	258	175	55	480	215	340	620	450	230	850	107	600
F057	<330	<330	<330	633	453	1380	2620	1390	341	937	2280	<330	709	<330	<330	<330
F063	2430	639	27	560	1440	210	4340	3730	1020	924	(2480)**		649	718	312	642
F064	1960	163	23	300	1990	196	4460	3540	927	2710	3830	1680	1380	2770	523	2430
F065	2710	356	45.5	767	1280	116	2020	1660	1190	1510	(3413)**		713	2690	737	2940
F066	4500	370	37	1100	2100	220	3300	3000	2200	2900	(4900)**		3600	2800	1100	2900
F084	2100	<100	200	500	5800	1000	2800	1900	(1900)**		(2800)**		3600	(<200)**		800
F087	3590	370	1210	2480	8890	1050	2390	1150	3050	4920	4450	1350	4480	1650	<100	4040
F094	6480	390	80	1130	2040	180	3760	2920	2160	2410	(7544)**		3660	3570	680	3520
N041	1100	<20	<20	200	1700	310	870	1100	620	740	780	<60	<80	380	<200	520
N090	-	40	<8	374	1478	446	4229	2999	778	2839	(5652)**		1314	2011	<16	957
N107	6146	573	75	136	2276	496	4200	3476	1750	3102	3826	1452	1747	2042	689	2046
N158	3589	500	1236	1145	479	654	2968	2408	879	1069	(4800)**		790	1940	248	1312
N196	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
N197	<90	<100	100	<10	140	10	900	1100	1300	680	910	320	350	1300	4400	1400
N200	<100	<100	<100	<100	<100	<100	2030	1120	724	1020	<100	1190	982	(<200)**		<100
N208	400	21	4	11	420	38	990	820	550	650	1340	590	350	970	310	800
Interlab																
Median	2570	363	68	500	1630	220	2505	1780	927	1069	1810	1190	982	1795	602	1312
Reference																
Value	---	---	---	---	1433	113	3480	2957	1347	1857*	2228	1147	1006	1790	559	1555

Table 13. PAH results for Sediment Sample #4 (ng/g).

Lab No.	Parameter															
	Naph-	Acen-	Ace-	Fl-	Phen-	Anth-	Fluor-	Pyrene	B(a)A	Chrys-	B(b)F	B(k)F	B(a)P	I(cd)P	D(ah)A	B(ghi)P
F007	212	16	54	55	601	113	609	555	337	289	78	46	62	44	nd	62
F033	81.4	-	-	140	533	69.1	532	549	311	488	526	526	686	-	-	-
F040	24	nd	nd	nd	95	33	170	150	60	90	110	110	68	61	nd	70
F057	<330	<330	<330	<330	<330	<330	<330	<330	855	<330	<330	<330	<330	<330	<330	<330
F063	20	49	26	50	430	126	1170	1150	367	259	(615)**		386	233	100	236
F064	60	18	27	49	511	107	815	737	261	412	412	191	310	179	57	216
F065	44.7	37.3	38.5	41.4	468	108	580	549	432	355	(691)**		333	458	120	529
F066	67	27	16	29	370	72	440	450	440	380	(620)**		480	400	120	450
F084	<100	<100	200	<100	300	400	300	900	(500)**		(1700)**		3700	(4100)**		<200
F087	130	260	<100	<100	280	<100	860	780	640	1710	(1140)**		2640	510	<100	1510
F094	330	30	60	120	630	130	910	890	530	320	(1530)**		1110	560	160	620
H041	<20	<20	<20	<20	<20	70	160	260	<60	<70	<60	<60	270	<140	<190	<190
H090	-	15	54	35	574	254	952	832	418	554	(1233)**		513	417	101	244
H107	244	87	59	88	743	195	1140	1108	692	784	985	385	834	516	179	579
H158	110	67.1	40.6	56.9	519	99.8	468	444	312	254	(759)**		252	323	65.8	235
H196	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
H197	90	<100	300	<10	29	<9	350	470	290	290	590	190	350	850	2500	880
H200	<100	<100	<100	<100	<100	224	1020	<100	234	314	<100	342	390	(<200)**		<100
H208	7	6	8	20	240	34	450	440	350	320	570	230	440	380	120	360
Interlab																
Median	81.4	30	47.3	50	468	108	580	552	359	320	526	211	440	400	120	360
Reference																
Value	---	---	---	---	528	104	817	829	427	512*	466	262	426	332	111	331

Table 14. PAH results for Sediment Sample #5 (ng/g).

Lab No.	Parameter															
	Naph-	Acen-	Ace-	Fl-	Phen-	Anth-	Fluor-	Pyrene	B(a)A	Chrys-	B(b)F	B(k)F	B(a)P	I(cd)P	D(ab)A	B(ghi)P
F007	226	21	40	32	372	78	354	310	250	217	292	216	261	300	70	390
F033	116	-	-	nd	343	84.6	481	453	654	695	918	918	1580	-	-	-
F040	28	nd	nd	nd	70	22	127	105	60	85	125	110	100	137	nd	115
F057	<330	<330	<330	<330	<330	<330	366	<330	<330	<330	616	<330	<330	349	<330	<330
F063	105	72	25	34	272	87	846	748	295	249	(552)**	330	268	106	280	
F064	19	22	17	23	290	54	527	436	200	354	414	173	292	254	64	249
F065	79.1	73.0	34.6	22.2	291	70.0	462	427	382	355	(838)**	388	734	183	777	
F066	93	30	15	20	230	43	280	250	340	300	(580)**	430	500	180	540	
F084	<100	<100	<100	<100	200	<100	400	900	(500)**	(<200)**	1300	(2600)**	<200			
F087	220	<100	<100	<100	260	340	620	450	410	2550	(1330)**	4360	480	<100	1160	
F094	320	30	40	70	360	70	590	540	440	290	(1700)**	1180	1020	190	970	
H041	<20	<20	<20	<30	100	40	130	490	<70	<70	<70	<60	210	<150	<210	<210
H090	-	14	36	23	354	158	678	535	284	532	(1328)**	544	666	<4	332	
H107	268	98	45	56	479	126	820	689	557	746	1041	378	1071	687	210	746
H158	132	73.5	33.3	39.9	345	71.0	355	314	261	241	(834)**	273	417	83.5	317	
H196	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
H197	<90	<100	<90	<10	21	<9	150	200	230	120	220	51	91	180	200	90
H200	<100	<100	<100	<100	<100	<100	524	<100	160	250	<100	314	370	(<200)**	<100	
H208	51	8	9	14	150	21	280	230	240	240	520	220	370	410	110	370
Interlab																
Median	111	30	34	28	281	71	431	443	284	290	467	218	388	414	145	370
Reference																
Value	---	---	---	---	293	58	579	466	323	456*	578	260	408	480	119	493

Table 15. PAH results for Ampules 8A and 8B*** (ng/ul).

Lab No.	Parameter															
	Naph-	Acen-	Ace-	Fl-	Phen-	Anth-	Fluor-	Pyrené	B(a)A	Chrys-	B(b)F	B(k)F	B(a)P	I(cd)P	D(ah)A	B(ghi)P
8A: GC results																
F007	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
F033	-	9.93	1.16	32.8	17.1	-	22.0	12.6	-	-	24.9	11.9	13.6	10.3	-	14.8
F040	8.0	5.0	nd	14.0	8.0	1.0	14.0	11.0	2.0	6.0*	9.0	5.0	5.0	14	nd	9.0
F057	10.3	3.6	0.9	18.9	8.6	0.2	17.1	10.1	3.0	6.1	10.7	4.0	1.4	8.4	0.7	0.0007
F063	25.4	9.1	0.8	39.4	16.5	0.3	38.6	34.5	6.5	21.4	(42.1)**	12.0	19.1	7.6	24.2	
F064	22	10	1.4	39	17	1.1	39	30	6.0	16	16	5.4	7.1	11	2.0	14
F065	14.2	11.7	1.63	22.1	14.1	0.85	27.1	22.4	9.81	14.5	(24.5)**	8.51	22.3	6.29	27.6	
F066	25	10	1.8	43	25	1.8	40	35	5	16	(28)**	28	14	0.95	20	
F084	6	6.9	0.7	58	28	(54)**	27	(28)**	(38)**	28	(30)**	28	(30)**	26		
F087	16.69	8.09	1.20	34.33	13.70	0.85	1.56	24.94	5.74	14.27	18.25	6.74	8.24	13.58	2.96	15.95
F094	20.60	9.51	1.38	34.40	(20.00)**	33.40	26.50	7.52	16.80	(23.80)**	12.10	16.00	3.07	14.90		
N041	7.2	2.8	0.67	18	6.4	<0.41	32	27	<0.82	15	13	<0.71	<1	<1.7	<2.4	8.1
N090	-	12.50	1.40	13.45	13.80	2.60	37.85	26.55	6.50	14.85	22.35	7.40	10.15	15.50	3.65	11.35
N107	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
N158	14.8	7.45	1.33	24.3	13.8	2.98	18.7	15.1	5.39	9.79	13.8	5.90	6.90	14.9	3.24	13.4
N208	18.4	5.02	0.77	29.0	9.05	0.43	21.2	16.1	3.94	9.99	7.85	4.61	4.99	7.81	1.74	7.80
Interlab																
Median	15.74	8.60	1.20	30.9	13.8	0.93	27.1	25.72	5.74	14.68	13.8	5.65	8.51	14	3.02	14.4
Design																
Value	19.89	9.87	0.945	37.84	16.05	0.974	39.06	29.48	11.54	19.17	19.03	9.10	9.69	15.70	3.95	13.97
8B: HPLC results																
N196	54.5	50.7	30.7	40.9	15.9	4.02	34.3	26.0	6.80	18.6	23.6	8.77	9.81	17.7	8.95	14.7
N197	0.8	3.6	14	33	16	0.69	39	30	6.9	16	27	7.5	9.8	14	<0.05	17
N200	<10	28.1	<20	<10	18.4	<1	40	48.8	5.7	9.4	24.1	7.2	8.1	<1	(15.3)**	
Interlab																
Median	27.7	28.1	22.4	37.0	16	2.36	39	30	6.80	16	24.1	7.5	9.8	15.9	8.95	15.9
Design																
Value	19.89	9.87	0.945	37.84	16.05	0.974	39.06	29.48	11.54	19.17	19.03	9.10	9.69	15.70	3.95	13.97

*** A(toluene); B(acetonitrile)

Table 16. PAH results for Ampules 9A and 9B*** (ng/ul).

Lab No.	Parameter															
	Naph-	Acen-	Ace-	Fl-	Phen-	Anth-	Fluor-	Pyrene	B(a)A	Chrys-	B(b)F	B(k)F	B(a)P	I(cd)P	D(ah)A	B(ghi)P
9A: GC results																
F007	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
F033	-	0.36	0.38	0.32	0.37	-	0.71	0.70	-	-	0.20	0.17	0.22	0.13	-	0.17
F040	0.2	0.3	0.3	0.2	0.4	nd	0.4	0.4	0.9	0.3*	0.2	0.2	0.3	0.5	nd	0.3
F057	0.22	0.11	<0.4	0.08	0.49	0.36	0.52	0.18	0.14	0.41	0.24	0.20	0.30	0.15	0.0009	0.0007
F063	0.5	0.4	0.5	0.4	0.5	nd	0.9	1.0	0.2	0.6	(0.6)**	0.4	0.2	nd	0.4	
F064	0.5	0.5	0.5	0.5	0.5	0.1	0.9	0.9	0.2	0.4	0.3	0.2	0.2	0.2	0.1	0.2
F065	0.428	0.586	0.640	0.323	0.480	0.034	0.582	0.624	0.463	0.779	(0.825)**	0.329	0.299	0.185	0.270	
F066	0.82	0.29	0.43	0.26	0.31	0.022	0.54	0.62	0.52	0.42	(0.95)**	0.32	0.22	0.025	0.31	
F084	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
F087	0.41	0.20	0.20	0.22	0.24	<0.20	0.62	0.68	0.24	0.40	0.47	0.25	0.39	0.38	<0.02	0.48
F094	0.56	0.44	0.49	0.49	(0.82)**	0.66	0.82	0.19	7.83	(12.20)**	11.20	11.30	1.50	15.90		
N041	<0.22	<0.2	<0.2	<0.31	<0.31	<0.41	<0.4	<0.59	<0.82	<0.85	<0.8	<0.71	<1	<1.7	<2.4	<2.4
N090	-	0.46	0.38	0.12	0.31	0.13	0.73	0.66	0.21	0.44	0.51	0.22	0.37	0.37	0.07	0.26
N107	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
N158	0.425	0.449	0.457	0.462	0.506	0.204	0.854	0.884	0.312	0.636	0.623	0.379	0.494	0.653	0.096	0.578
N208	0.46	0.24	0.34	0.33	0.30	0.04	0.41	0.44	0.11	0.26	0.21	0.11	0.16	0.14	0.05	0.15
Interlab																
Median	0.444	0.38	0.43	0.322	0.4	0.1	0.64	0.67	0.21	0.42	0.27	0.2	0.325	0.260	0.083	0.285
Design																
Value	0.497	0.493	0.472	0.473	0.502	0.0973	0.976	0.983	0.481	0.767	0.571	0.364	0.484	0.491	0.0987	0.437
9B: HPLC results																
N196	0.49	0.58	0.38	0.35	0.78	0.33	2.98	2.03	1.75	1.40	1.40	1.40	1.26	3.84	1.35	0.95
N197	<0.3	<0.5	<0.3	0.41	0.47	0.071	0.87	0.95	0.28	0.64	1.0	0.30	0.49	0.45	<0.05	0.54
N200	<10	2.2	<20	<10	0.5	<1	0.8	0.7	0.2	0.2	0.3	0.2	0.3	<1	(0.4)**	
Interlab																
Median	0.49	1.39	0.38	0.38	0.5	0.20	0.87	0.95	0.28	0.64	1.0	0.30	0.49	2.15	1.35	0.75
Design																
Value	0.497	0.493	0.472	0.473	0.502	0.0973	0.976	0.983	0.481	0.767	0.571	0.364	0.484	0.491	0.0987	0.437

*** A(toluene); B(acetonitrile)

Table 17. Arsenic results for sediment analyses (ug/g).

SAMPLE LAB NO	10		11		12		13		14		15	
	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK
F007	8.5	6.50	29.1	4.50	19.1 L	3.00	6.0	11.00	30.2	6.00	11.4 H	15.00
F033	8.6	8.50	29.1	4.50	22.8	9.00	6.3	14.00	26.1 L	4.00	10.7	12.00
F040	8.6	8.50	33	12.00	22	6.50	6.2	13.00	32	11.50	9.8	8.50
F057	8.50	6.50	34.0	13.00	24.0	15.00	5.0	4.50	31.0	8.00	9.0	5.50
F063	6.4 VL	3.00	34.2	14.00	19.7 L	4.00	4.9	3.00	32	11.50	7.0 VL	3.00
F064	15. VH	17.00	27. L	3.00	22	6.50	13. VH	17.00	27. L	5.00	0.16 VL	1.00
F066	10. H	14.50	40. VH	17.00	22	6.4	6.4	15.00	3.1 VL	1.00	10	10.50
F084	0.9 VL	1.00	1.6 VL	9.00	23	11.50	0.7 VL	1.00	5.5 VL	2.00	17. VH	17.00
F087	9	11.50	32	9.00	23	11.50	5	4.50	31	8.00	11	13.50
N090	10. H	14.50	30	6.50	22	6.50	6	11.00	31	8.00	9	5.50
F094	8.02	5.00	32.5	10.00	23.1	14.00	6.85	9.00	34.8 H	16.00	11.0	13.50
N107	8.9	10.00	36. H	15.00	23	11.50	5.6	7.00	34	15.00	9.3	7.00
N158	12.3 VH	16.00	32.6	11.00	26.5 H	17.00	9.34 VH	16.00	31.4	10.00	13.5 VH	16.00
N196	7. L	4.00	31	8.00	17. VL	1.00	6	11.00	33	14.00	8. L	4.00
N197	9.0	11.50	30	6.50	25	16.00	5.8	8.00	25. VL	3.00	10	10.50
N200	9.6	13.00	37. H	16.00	23	11.50	5.4	6.00	35. H	17.00	9.8	8.50
N208	5.5 VL	2.00	26.9 L	2.00	18.2 VL	2.00	3.7 VL	2.00	32.2	13.00	6.3 VL	2.00
MEAN	8.6		30.4		22.1		5.95		27.9		9.6	
Std.Dev.	2.9		8.2		2.4		2.47		9.3		3.4	
MEDIAN	8.6		32		22.8		5.85		31		9.8	
REFERENCE VALUE	9.7		32.5		23.00 ± 1.84		6.85		32.5		11.1 ± 1.4	

LAB NO.	TOTAL RANK	AVERAGE RANK	NO. OF SAMPLES RANKED	SUMMARY OF FLAGGING	METHOD OF DETECTION
N208	23.00	3.833	6	VLLVLLVLL	AAS, hydride
F084	33.50	5.583	6	VLLVLLVLLVH	AAS, hydride
F063	38.50	6.417	6	VLLVLL	AAS, hydride
N196	42.00	7.000	6	LVLL	ICP, hydride
F007	46.00	7.667	6	LH	AAS, hydride
F064	49.50	8.250	6	VHLLVLLVLL	GFAAS
F033	52.00	8.667	6	L	ICP, hydride
N090	52.00	8.667	6	H	AAS, hydride
F057	52.50	8.750	6		colorimetric, hydride
N197	55.50	9.250	6	VL	AAS, hydride
F087	58.00	9.667	6		AAS, hydride
F040	60.00	10.000	6	HVHVL	AAS, hydride
F066	64.50	10.750	6	H	AAS, hydride
N107	65.50	10.917	6	H	AAS, hydride
F094	67.50	11.250	6	HH	ICP, hydride
N200	72.00	12.000	6	HH	ICP, hydride
N158	86.00	14.333	6	VHHVHVVH	ICP, hydride
OVERALL AVERAGE RANK IS		9.000			

NOTE: Certified reference values on tables 17 - 24 are given with their uncertainties representing the 95% tolerance limits for an individual sample. All other reference values are recommended values only and are subject to change as more data become available.

Table 18. Cadmium results for sediment analyses (ug/g).

LOWER LIMIT FOR USE OF BASIC ACCEPTABLE ERROR= 1.00 ug/g BASIC ACCEPTABLE ERROR= 0.50 ug/g CONCENTRATION ERROR INCREMENT= 0.10

SAMPLE LAB NO.	10		11		12		13		14		15	
	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK
F007	<5.0	.00	<5.0	.00	<5.0	.00	<5.0	.00	<5.0	.00	<5.0	.00
F033	5.3	8.00	<1.0	.00	<1.0 VL	1.50	3.13	4.00	1.13 L	3.00	<1.0	.00
F040	4.7	5.00	2.0	10.50	1.9	8.00	3.7	8.00	2.0	11.00	0.4	5.00
F057	5.70	9.00	1.55	5.00	1.85	6.00	3.80	9.00	1.55	6.00	<0.50	.00
F063	0.5 VL	1.00	<0.3 VL	1.00	<0.3 VL	1.50	<0.3 VL	1.00	<0.3 VL	1.00	<0.3	.00
F064	6.3	13.00	2.0	10.50	2.0	11.00	4.4	11.00	1.9	9.00	0.2	2.00
F066	7.00	14.00	1.1 L	4.00	2.3	14.00	3.3	6.00	1.5	5.00	0.4	5.00
F084	7.1 VH	17.00	2.7 VH	13.00	2.2	12.50	5.9 VH	15.00	7.6 VH	15.00	<0.1	1.00
F087	4.33 L	3.00	0.56 VL	2.00	1.81	4.00	4.33	10.00	1.96	10.00	<0.50	.00
N090	8.3 VH	15.00	1.7	7.00	1.9	8.00	4.5	12.50	2.9 VH	13.00	1.5 VH	9.00
F094	5.74	10.00	1.80	8.00	1.83	5.00	3.65	7.00	1.76	8.00	0.250	3.00
N107	4.6	12.00	1.6	6.00	1.9	8.00	3.2	5.00	1.6	7.00	0.77	8.00
N158	6.22	4.00	1.81	9.00	1.91	10.00	5.48 VH	14.00	1.42	4.00	<1.00	.00
N196	5.	6.00	<2.	.00	<2.	.00	3.	3.00	<2.	.00	<2.	.00
N197	14. VH	16.00	5. VH	14.00	3. VH	15.00	6. VH	16.00	5. VH	14.00	6. VH	10.00
N200	3.8 VL	2.00	1.00 L	3.00	1.6	3.00	2.0 VL	2.00	0.80 VL	2.00	0.60	7.00
N208	6.2	11.00	2.2	12.00	2.2	12.50	4.5	12.50	2.2	12.00	0.4	5.00
MEAN	5.81		1.92		2.03		4.06		2.38		1.17	
Std.Dev.	2.76		1.08		0.35		1.12		1.81		1.85	
MEDIAN	5.5		1.75		1.9		3.75		1.76		0.40	
REFERENCE VALUE	5.30		2.30		2.10		3.80		2.30		0.25 ± 0.4	

LAB NO.	TOTAL RANK	AVERAGE RANK	NO. OF SAMPLES RANKED	SUMMARY OF FLAGGING
F007	0.00	.000	0	
F063	5.50	1.100	5	VHVLVLVL
N200	19.00	3.167	6	VLLVLVL
F033	16.50	4.125	4	VLL
N196	9.00	4.500	2	
F087	29.00	5.800	5	LVL
N107	38.00	6.333	6	
F094	41.00	6.833	6	
F066	41.00	6.833	6	
F057	35.00	7.000	5	
F040	47.50	7.917	6	
F064	56.50	9.417	6	
N158	49.00	9.800	5	VHVVH
N090	64.50	10.750	6	VHVVHVVH
N208	65.00	10.833	6	VHVVHVVH
F084	70.50	11.750	6	VHVVHVVHVVH
N197	85.00	14.167	6	
OVERALL AVERAGE RANK IS	7.814			BIASED HIGH

INSUFFICIENT DATA
INSUFFICIENT DATA
BIASED LOW
INSUFFICIENT DATA

Table 19. Chromium results for sediment analyses (ug/g).

LOWER LIMIT FOR USE OF BASIC ACCEPTABLE ERROR=50.00 ug/g BASIC ACCEPTABLE ERROR=25.00 ug/g CONCENTRATION ERROR INCREMENT= 0.10

SAMPLE LAB NO	10		11		12		13		14		15	
	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK
F007	148. H	14.00	99. H	13.00	100. H	12.00	160. H	15.50	100. H	12.00	104. VH	11.00
F033	141. H	13.00	104. H	14.00	115. VH	16.00	150. H	13.00	103. H	14.00	109. VH	13.00
F040	96.	7.00	62.	8.00	60.	8.00	110.	8.00	65.	9.00	52.	8.00
F057	167. VH	16.00	98. H	12.00	104. VH	13.00	143.	12.00	99. H	11.00	131. VH	17.00
F063	88.	5.50	50.9	5.00	45.4	5.00	94.3	7.00	54.9	6.00	48.3	6.00
F064	104.	9.00	68.	9.00	53.	7.00	116.	9.00	61.	7.50	51.	7.00
F066	86.	4.00	47.6	4.00	40.	3.00	91.	5.00	47.	4.00	38.	3.00
F084	97.2	8.00	87.6	11.00	94.9 H	11.00	87.8	4.00	102. H	13.00	117. VH	14.00
F087	74.2	3.00	43.4	2.00	40.1	4.00	72.3 L	2.00	43.7 L	3.00	40.1	4.00
N090	66. L	1.00	39. L	1.00	31. L	1.00	70. L	1.00	37. L	1.00	30.	1.00
F094	149. H	15.00	105. H	15.50	114. VH	14.50	160. H	15.50	106. VH	15.00	108. VH	12.00
N107	116.	11.00	105. H	15.50	114. VH	14.50	158. H	14.00	114. VH	16.00	130. VH	16.00
N158	69.5 L	2.00	45.4	3.00	32.5 L	2.00	77.8 L	3.00	40.2	2.00	33.0	2.00
N196	118.	12.00	72.	10.00	93. H	10.00	127. H	11.00	88.	10.00	89. H	10.00
N197	110.	10.00	61.	7.00	61.	9.00	120.	10.00	61.	7.50	54.	9.00
N200	260. VH	17.00	150. VH	17.00	120. VH	17.00	270. VH	17.00	150. VH	17.00	120. VH	15.00
N208	88.	5.50	53.	6.00	48.	6.00	93.	6.00	52.	5.00	43.	5.00
MEAN	116		76		74		124		78		76	
Std.Dev.	47		31		33		49		32		38	
MEDIAN	104		68		61		116		65		54	
REFERENCE VALUE	---		---		---		---		---		123 ± 14	

LAB NO.	TOTAL RANK	AVERAGE RANK	NO. OF SAMPLES RANKED	SUMMARY OF FLAGGING	ACIDS USED FOR DIGESTION
N090	6.00	1.000	6	LLLLL	HCl, HNO ₃ , H ₂ O ₂
N158	14.00	2.333	6	LLLLL	aqua regia, H ₂ O ₂
F087	18.00	3.000	6	LLL	aqua regia
F066	23.00	3.833	6	L	aqua regia
N208	33.50	5.583	6		HNO ₃ , H ₂ O ₂
F063	34.50	5.750	6		aqua regia
F040	48.00	8.000	6		aqua regia
F064	48.50	8.083	6		aqua regia
N197	52.50	8.750	6		HCl, HNO ₃ , H ₂ O ₂
F084	61.00	10.167	6	HHVH	HF, HClO ₄ , HNO ₃
N196	63.00	10.500	6	HH	HF, HCl, HClO ₄ , HNO ₃
F007	77.50	12.917	6	HHHHVH	HF, aqua regia, H ₂ O ₂
F057	81.00	13.500	6	VHHVHVH	HF, HCl, HClO ₄ , HNO ₃
F033	83.00	13.833	6	HVVHVH	HF, HClO ₄ , HNO ₃
N107	87.00	14.500	6	VVHVHVH	HF, aqua regia
F094	87.50	14.583	6	HVVHVH	HF, HClO ₄ , HNO ₃
N200	100.00	16.667	6	VHVHVHVH	HF, HClO ₄ , HNO ₃
OVERALL AVERAGE RANK IS		9.000			aqua regia

Table 20. Copper results for sediment analyses (ug/g).

SAMPLE LAB NO	10		11		12		13		14		15	
	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK
F007	122	11.00	573	11.00	62.5 L	1.00	70.5	2.00	565	8.50	16.0	4.50
F033	119	4.00	572	10.00	75.4	7.00	75.9	7.00	565	8.50	22.1 H	17.00
F040	110	1.50	570	9.00	76	8.00	76	8.00	570	10.00	17	9.50
F057	121	10.00	612	13.00	83	14.00	84	14.00	616	14.00	22	16.00
F063	123	12.50	553	7.00	76.4	9.00	78.9	13.00	437	16.00	16.3	6.00
F064	144	VH	738	VH	98	16.00	94	16.00	728	VH	17	9.50
F066	110	1.50	530	4.00	73	2.50	71	3.00	530	5.00	14	1.50
F084	145	VH	621	14.00	73.9	4.00	77.6	11.00	682	VH	19.9	14.00
F087	118	3.00	509	1.00	76.9	11.00	70.4	1.00	511	4.00	16.5	7.00
N090	124	14.00	540	5.50	78	12.00	77	9.00	535	6.00	17.4	9.50
F094	120	7.00	592	12.00	76.8	10.00	78.6	12.00	602	13.00	17.4	13.00
N107	136	H	640	H	90	15.00	87	15.00	583	12.00	16.4	4.50
N158	120	7.00	510	2.00	74.6	6.00	75.8	6.00	504	2.00	14.8	3.00
N196	123	12.50	560	8.00	79.4	13.00	77.3	10.00	573	11.00	17.1	12.00
N197	120	7.00	540	5.50	74	5.00	73	4.00	540	7.00	17	9.50
N200	120	7.00	790	VH	100	17.00	100	17.00	780	VH	20	15.00
N208	120	7.00	517	3.00	73	2.50	74	5.00	506	3.00	14	1.50
MEAN	123		586		78.9		78.9		577		17.3	
Std.Dev.	10		77		9.3		8.2		84		2.4	
MEDIAN	120		570		76.4		77		565		17	
REFERENCE VALUE	105.0		565.0		80.0		81.0		565.0		18.5 ± 2.7	

LAB NO.	TOTAL RANK	AVERAGE RANK	NO. OF SAMPLES RANKED	SUMMARY OF FLAGGING
F066	17.50	2.917	6	
N208	22.00	3.667	6	
N158	26.00	4.333	6	L
F087	27.00	4.500	6	L
N197	38.00	6.333	6	
F007	38.00	6.333	6	L
F040	46.00	7.667	6	
F063	48.50	8.083	6	VL
F033	53.50	8.917	6	H
N090	56.00	9.333	6	
N196	66.50	11.083	6	
F094	67.00	11.167	6	VHVH
F084	75.00	12.500	6	HHH
N107	76.50	12.750	6	
F057	81.00	13.500	6	
F064	89.50	14.917	6	VHVHVHVH
N200	90.00	15.000	6	VHVHVHVH
OVERALL AVERAGE RANK IS	9.000			

Table 21. Lead results for sediment analyses (ug/g).

SAMPLE LAB NO.	10		11		12		13		14		15	
	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK
F007	193.	11.00	54.0	12.00	77.5	6.00	152.	14.50	52.0	7.50	28.0	14.50
F033	162. L	2.00	42.8	3.00	67.8 L	1.00	129.	5.00	48.3	4.00	14.6	5.00
F040	160. L	1.00	51.	8.50	80.	7.00	120.	2.00	53.	9.00	22.	8.00
F057	204.	13.00	69. H	16.00	87.	11.00	147.	12.00	62.	17.00	25.	12.00
F063	196.	12.00	51.	8.50	82.	8.00	142.	10.50	51.	5.50	24.	10.00
F064	189.	9.00	46.	6.00	75.	4.00	139.	9.00	51.	5.50	12. L	2.00
F066	180.	4.00	49.	7.00	84.	9.00	130.	6.50	52.	7.50	26.	13.00
F084	181.	6.50	58.2	14.00	74.6	3.00	83.9 VL	1.00	53.6	10.00	29.7	16.00
F087	181.	6.50	34. L	1.00	95.	16.00	142.	10.50	20. VL	1.00	48. VH	17.00
N090	213.	16.00	52.	10.50	85.	10.00	151.	13.00	56.	14.00	24.	10.00
F094	208.	14.00	69.9 H	17.00	88.2	13.00	158.	16.00	58.4	15.00	21.8	7.00
N107	210.	15.00	44.	4.00	90.	15.00	152.	14.50	40. L	2.00	14.	4.00
N158	181.	6.50	45.9	5.00	89.9	14.00	132.	8.00	55.9	13.00	18.6	6.00
N196	168.	3.00	52.	10.50	72.	2.00	127.	3.00	55.	12.00	13. L	3.00
N197	190.	10.00	42.	2.00	76.	5.00	130.	6.50	41.	3.00	10. L	1.00
N200	260. VH	17.00	62.	15.00	99.	17.00	180. VH	17.00	61.	16.00	24.	10.00
N208	181.	6.50	56.	13.00	88.	12.00	128.	4.00	54.	11.00	28.	14.50
MEAN	192		51.7		83.0		137		50.8		22.5	
Std.Dev.	24		9.4		8.5		20		9.8		9.0	
MEDIAN	189		51		84		139		53		24	
REFERENCE VALUE ---			58.0		85.7		143.5		58.0		22.7 ± 3.4	

LAB NO.	TOTAL RANK	AVERAGE RANK	NO. OF SAMPLES RANKED	SUMMARY OF FLAGGING
F033	20.00	3.333	6	LL
N197	27.50	4.583	6	L
N196	33.50	5.583	6	L
F040	35.50	5.917	6	L
F064	35.50	5.917	6	L
F066	47.00	7.833	6	L
F084	50.50	8.417	6	VL
F087	52.00	8.667	6	LVLVH
N158	52.50	8.750	6	L
N107	54.50	9.083	6	L
F063	54.50	9.083	6	L
N208	61.00	10.167	6	
F007	65.50	10.917	6	
N090	73.50	12.250	6	
F057	81.00	13.500	6	H
F094	82.00	13.667	6	H
N200	92.00	15.333	6	VHVH
OVERALL AVERAGE RANK IS		9.000		BIASED HIGH

Table 22. Mercury results for sediment analyses (ug/g).

SAMPLE LAB NO.	10					11					12					13					14					15				
	REPORTED VALUE	RANK	REPORTED VALUE	RANK	NO. OF SAMPLES RANKED	AVERAGE RANK	SUMMARY OF FLAGGING	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK			
F007	0.620	10.00	0.120	11.00	6	3.417	VLL	1.12	12.00	0.315	5.00	0.100	10.50	0.205	13.00															
F033	0.61	9.00	0.10	7.50	6	3.667	VLLVHVL	1.04	9.00	0.36	8.00	0.09	8.00	0.18	11.00															
F040	0.56	6.00	0.04	2.50	6	4.583	VL	0.98	4.00	0.32	6.00	0.03	2.00	0.16	7.00															
F057	0.476	2.00	0.105	9.00	6	4.667	VH	0.277 VL	1.00	0.277	3.00	0.095	9.00	0.158	4.00															
F063	0.704	13.00	0.092	5.50	6	7.417	VH	1.037	7.00	0.528 VH	14.00	0.082	7.00	0.154	2.00															
F064	0.66	11.00	0.06	4.00	6	8.083	VH	1.04	9.00	0.47	12.00	0.07	6.00	0.16	7.00															
F066	1.2 VH	16.00	0.16	15.00	6	8.167		1.5 VH	15.00	0.52 H	13.00	0.19	15.00	0.35 VH	14.00															
F084	0.6	7.50	0.13	13.50	6	8.583		1.1	11.00	0.4	10.50	0.11	12.50	0.16	7.00															
F087	0.538	4.00	0.120	11.00	6	8.750	VHVVH	1.000	5.50	0.328	7.00	0.125	14.00	0.178	10.00															
N090	0.7	12.00	0.1	7.50	6	9.083		1.0	5.50	0.4	10.50	0.1	10.50	0.2	12.00															
F094	0.528	3.00	0.092	5.50	6	9.333		0.748 VL	2.00	0.244 L	2.00	0.068	5.00	0.155	3.00															
N107	0.60	7.50	0.12	11.00	6	9.667		1.04	9.00	0.37	9.00	0.11	12.50	0.16	7.00															
N158	0.54	5.00	0.13	13.50	6	10.000		0.94	3.00	0.29	4.00	0.059	4.00	0.16	7.00															
N196	<2.0	15.00	<2.5 VH	16.00	6	10.333		2.0 VH	16.00	<2.0	16.00	<2.0	16.00	2.0 VH	15.00															
N197	1.0 VH	14.00	0.5 VH	16.00	6	10.667		1.4 VH	14.00	0.9 VH	16.00	0.5 VH	16.00	<0.5	7.00															
N200	0.99 VH	14.00	0.04	2.50	6	11.000		1.3 H	13.00	0.78 VH	15.00	0.04	3.00	0.16	7.00															
N208	0.083 VL	1.00	<0.001 L	1.00	2	15.400		4.12 VH	17.00	0.079 VL	1.00	<0.001	1.00	0.131	1.00															
MEAN	0.651		0.127			16.000		1.26		0.411		0.118		0.379																
Std. Dev.	0.252		0.108			16.000		0.83		0.201		0.112		0.553																
MEDIAN	0.605		0.103			16.000		1.04		0.365		0.093		0.160																
REFERENCE VALUE	---		0.11			16.000		1.09 ± 0.15		0.35		0.11		0.129 ± 0.012																
OVERALL AVERAGE RANK IS						8.588																								

BIASED HIGH
BIASED HIGH
INSUFFICIENT DATA

Table 23. Wickel results for sediment analyses (ug/g).

LOWER LIMIT FOR USE OF BASIC ACCEPTABLE ERROR=25.00 ug/g BASIC ACCEPTABLE ERROR= 6.25 ug/g CONCENTRATION ERROR INCREMENT= 0.20

SAMPLE LAB NO.	10		11		12		13		14		15	
	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK
F007	30. L	2.00	981.	12.00	36. VL	1.00	25. L	1.00	971.	12.00	40.	2.00
F033	39.7	8.00	974.	11.00	54.7	7.00	35.6	10.00	969.	11.00	45.3	6.00
F040	35.	4.00	850.	16.00	55.	8.50	33.	7.00	880.	8.00	47.	9.00
F047	41.	9.00	1027.	14.00	50.	4.50	29.	2.50	1025.	15.00	46.	7.50
F063	36.	5.00	895.	8.00	49.	3.00	33.	7.00	712. L	3.00	45.	5.00
F064	39.	7.00	989.	13.00	63.	12.00	34.	9.00	956.	10.00	51.	10.00
F066	33.	3.00	750.	3.00	51.	6.00	29.	2.50	770.	5.00	44.	4.00
F084	48.3	12.00	1330.	17.00	59.1	11.00	39.0	12.00	1402. VH	17.00	55.7	13.00
F087	45.	10.00	57. VL	1.00	58.	10.00	51. VH	16.00	72. VL	1.00	55.	12.00
N090	59. VH	17.00	871.	7.00	70. H	15.50	63. VH	17.00	781.	7.00	64. VH	17.00
F094	51. H	14.50	1030.	15.00	70. H	15.50	46. H	14.00	1060.	16.00	58. H	14.00
N107	50.	13.00	922.	9.00	64.	13.00	41.	13.00	921.	9.00	52.	11.00
N158	27.8 L	1.00	580.	2.00	48.8	2.00	31.3	5.00	610.	2.00	37.9	1.00
N196	54. H	16.00	965.	10.00	79. VH	17.00	47. VH	15.00	981.	13.00	59. H	15.00
N197	51. H	14.50	780.	4.00	50.	4.50	33.	7.00	780.	6.00	43.	3.00
N200	46.	11.00	1100.	16.00	69. H	14.00	38.	11.00	1000.	14.00	62. H	16.00
N208	36.	5.50	790.	5.00	55.	8.50	30.	4.00	760.	4.00	46.	7.50
MEAN	42.5		876		57.7		37.5		862		50.1	
Std.Dev.	9.0		267		10.5		9.6		270		7.8	
MEDIAN	41		922		55		34		921		47	
REFERENCE VALUE	44.2		941		59.3		38.5		941		55.3 ± 3.6	

LAB NO.	TOTAL RANK	AVERAGE RANK	NO. OF SAMPLES RANKED	SUMMARY OF FLAGGING
N158	13.00	2.167	6	LVLVL BIASED LOW
F066	23.50	3.917	6	
F007	30.00	5.000	6	LVL L
F063	31.50	5.250	6	L
N208	34.50	5.750	6	H
N197	39.00	6.500	6	
F040	42.50	7.083	6	VLVHL
F087	50.00	8.333	6	
F057	52.50	8.750	6	
F033	53.00	8.833	6	
F064	61.00	10.167	6	
N107	68.00	11.333	6	VHHVHH
N090	80.50	13.417	6	HH
N200	82.00	13.667	6	VHVH
F084	82.00	13.667	6	VHVHH
N196	85.00	14.167	6	HHH
F094	89.00	14.833	6	BIASED HIGH
OVERALL AVERAGE RANK IS	9.000			

Table 24. Zinc results for sediment analyses (ug/g).

LOWER LIMIT FOR USE OF BASIC ACCEPTABLE ERROR=99.00 ug/g BASIC ACCEPTABLE ERROR=20.00 ug/g CONCENTRATION ERROR INCREMENT= 0.10														
SAMPLE LAB NO	10		11		12		13		14		15			
	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK	REPORTED VALUE	RANK		
F007	839	6.00	834	12.00	283	11.00	1122	13.00	778	11.00	123	H	16.00	
F033	893	9.00	737	2.00	249	5.00	1080	7.50	749	7.00	90	5	2.00	
F040	820	3.50	760	7.00	260	8.50	1100	11.00	770	10.00	98		7.50	
F057	945	14.00	822	11.00	305	H	1145	14.00	820	14.00	116		13.50	
F063	727	1.00	739	3.00	227	2.00	1030	1.50	585	VL	95		5.50	
F064	886	8.00	904	H	290	13.50	1080	7.50	910	VH	114		11.00	
F066	820	3.50	710	1.00	240	3.00			710	3.00	90		1.00	
F084	1569	17.00	1205	VH	35.4	VL	1504	16.00	1043	VH	150	VH	17.00	
F087	1008	5.00	758	5.00	261	10.00	1060	5.00	720	5.00	94		3.50	
N090	934	16.00	829	13.00	258	6.00	1057	4.00	718	4.00	101		9.00	
F094	930	13.00	785	9.00	285	12.00	1220	H	817	12.00	116		13.50	
N107	814	12.00	930	VH	320	VH	1040	3.00	946	VH	104		10.00	
N158	930	2.00	786	7.00	247	4.00	1080	7.50	725	6.00	95	0	5.50	
N196	901	10.00	786	10.00	299	H	1080	7.50	818	13.00	115		12.00	
N197	910	11.00	760	7.00	260	8.50	1100	11.00	750	8.50	98		7.50	
N200	950	15.00	840	14.00	290	13.50	1100	11.00	240	VL	120		15.00	
N208	880	7.00	740	4.00	259	7.00	1030	1.50	750	8.50	94		3.50	
MEAN	921		817		257		1114		756		107			
Std.Dev.	180		116		62		114		169		16			
MEDIAN	893		785		260		1080		750		101			
REFERENCE VALUE	---		771		279.0		1100		771		119 + 12			

LAB NO.	TOTAL RANK	AVERAGE RANK	NO. OF SAMPLES RANKED	SUMMARY OF FLAGGING
F066	11.50	2.300	5	
F063	15.00	2.500	6	
N208	31.50	5.250	6	VLVL
N158	32.00	5.333	6	
F033	34.50	5.750	6	
F087	33.50	5.583	6	
F040	47.50	7.917	6	H
N090	52.00	8.667	6	
N197	53.50	8.917	6	H
N196	67.50	11.250	6	H
F007	69.00	11.500	6	H
N200	69.50	11.583	6	VL
F064	70.00	11.667	6	VH
N107	74.00	12.333	6	VHVVH
F094	74.50	12.417	6	H
F057	82.50	13.750	6	H
F084	85.00	14.167	6	VHVVLVHVVH
OVERALL AVERAGE RANK IS	8.921			

BIASED LOW
BIASED LOW

Table 25. Detection Limits Reported for PAHs and PCBs in Sediments (ng/g).

Lab No. Parameter

Lab No.	PAHs														PCBs		
	Naph-	Acen-	Ace-	Fl-	Phen-	Anth-	Fluor-	Pyr-	B(a)A	Chrys-	B(b)F	B(k)F	B(a)P	I(cd)P	D(ah)A	B(ghi)P	Total PCBs
F007	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	100
F033	-	10	10	15	15	-	15	15	-	-	30	30	30	30	-	30	77
F040	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	20
F057	330	330	330	330	330	330	330	330	330	330	330	330	330	330	330	330	3
F063	100	100	100	100	100	100	100	100	200	200	200	200	200	200	200	200	10
F064	40	50	40	40	70	10	20	60	20	20	60	60	40	40	40	40	5
F065	10	10	10	10	10	10	10	10	10	10	10	10	10	10	20	20	100
F066	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	50
F084	100	100	100	100	100	100	100	100	(---200---	(---200---	(---200---	(---200---	200	(---200---	200	200	20
F087	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	10
F094	10	10	10	10	10	10	10	10	10	10	10	10	10	10	30	30	5
N041	10	10	10	10	10	10	10	10	10	10	10	10	10	10	30	30	50
N090	-	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5	2-5
N107	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	10
N158	24.0	9.6	9.6	9.6	3.2	4.8	8.0	8.0	8.0	33.6	22.4	22.4	19.2	24.0	19.2	19.2	3.2
N196	30	10	10	15	20	15	15	15	10	15	30	40	40	30	15	15	0.3
N197	90	100	90	10	9	9	10	9	9	9	10	9	9	9	100	10	80
N200	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	10
N208	0.1	0.1	0.1	0.1	0.2	0.2	0.1	0.2	0.2	0.2	0.2	0.2	0.2	0.3	0.2	0.2	10

Note: Ontario Ministry of the Environment guidelines for evaluating dredge spoils for open-water disposal' list a concentration level of 50 ng/g for Total PCBs.

Table 26. Detection Limits Reported for Trace Metals in Sediments (ug/g).

Lab No.	Trace Metals							
	Hg	Pb	Zn	Cr	Cu	Cd	Ni	As
F007	0.1	5	5	5	2	5	5	0.1
F033	0.01	5.0	1.0	1.0	1.0	1.0	3.0	0.2
F040	0.01	1.0	2.0	1.0	0.5	0.05	0.2	0.03
F057	0.005	0.05	0.05	0.05	0.01	0.005	0.05	0.5
F063	0.002	2	0.3	0.3	0.3	0.2	2	1
F064	0.01	0.1	0.1	0.1	0.1	0.05	0.1	0.1
F065	0.1	4	1	2	1	1	4	0.4
F066	0.05	10	5	5	5	0.2	5	0.5
F084	0.10	1	1	1	1	0.1	1	0.05
F087	0.005	0.50	0.25	0.10	0.10	0.50	0.25	0.2
F094	0.01	0.2	0.5	1	0.5	0.01	1	0.05
N041	0.02	0.2	3	3	3	1	5	0.2
N090	0.003	0.004	0.004	0.010	0.006	0.007	0.004	0.002
N107	0.01	1.0	0.5	0.5	0.5	0.2	0.5	0.1
N158	0.010	0.500	0.040	0.200	0.040	1.00	0.200	0.300
N196	2	0.001	0.001	0.001	0.001	2	0.001	0.1
N197	0.5	20	10	20	20	2	20	0.3
N200	0.02	1.0	1.0	1.0	1.0	1.0	1.0	1.0
N208	0.01	5	1	1	1	0.1	2	1
MOE Guidelines*	0.3	50	100	25	25	1.0	25	8

* Ontario Ministry of the Environment guidelines for evaluating dredge spoils for open-water disposal.³

Table 27. Summary of PCB Results Flagged to the Interlaboratory Medians.

Lab #	Total No. of Results Reported	No. of Results "Not Detected"	No. of Detectable Values	No. of Results Flagged				% Flagged*
				VH	H	L	VL	
F007	7	0	7	0	1	0	0	7
F033	7	0	7	1	1	0	0	21
F040	7	0	7	0	0	4	0	29
F057	7	0	7	3	0	0	0	43
F063	7	0	7	0	0	0	0	0
F064	7	0	7	1	0	0	0	14
F065	7	1	6	0	2	1	0	25
F066	7	0	7	2	2	0	0	43
F084	7	1	6	0	1	0	1	25
F087	7	0	7	0	0	0	0	0
F094	7	0	7	0	1	0	0	7
N041	7	1	6	0	0	0	0	0
N090	0	0	0	-	-	-	-	-
N107	7	0	7	0	0	0	0	0
N158	7	0	7	0	0	0	0	0
N196	4	0	4	0	0	0	2	50
N197	7	1	6	2	0	1	1	58
N200	7	0	7	0	0	1	0	7
N208	7	0	7	0	0	3	0	21

* H and L flags are counted as half of VH and VL flags, respectively.

Less than values and "not detected" results are not included in the calculation of the % Flagged.

Refer to Appendix IV for a complete listing of the individual flags by parameter for each laboratory.

Table 28. Summary of PAH Results Flagged to the Interlaboratory Medians.

Lab #	Total No. of Results Reported	No. of Results "Not Detected"	No. of Detectable Values	No. of Results Flagged				% Flagged*
				VH	H	L	VL	
F007	80	4	76	6	4	2	13	29
F033	77	3	74	14	11	4	0	29
F040	112	20	92	1	1	37	29	53
F057	112	52	60	8	6	8	7	37
F063	105	2	103	10	9	4	1	17
F064	112	0	112	3	7	11	1	12
F065	105	0	105	8	10	7	0	16
F066	105	0	105	8	13	5	2	18
F084	93	38	55	22	6	1	0	46
F087	109	16	93	30	8	2	1	39
F094	103	0	103	32	18	0	0	40
N041	112	75	37	0	0	15	2	26
N090	100	3	97	8	21	6	2	24
N107	80	0	80	26	36	1	0	56
N158	108	0	108	12	6	3	0	15
N196	64	9	55	6	6	3	21	57
N197	112	22	90	9	3	14	14	35
N200	105	59	46	2	4	3	1	14
N208	112	0	112	0	0	21	16	24

* H and L flags are counted as half of VH and VL flags, respectively.

Less than values and "not detected" results are not included in the calculation of the % Flagged.

Refer to Appendix IV for a complete listing of the individual flags by parameter for each laboratory.

Table 29. Summary of Trace Metal Flags and Biases.

Lab #	No. of Results Reported	Elements not Analyzed	No. of Results Flagged				% Flagged*	Laboratory Bias
			VH	H	L	VL		
F007	(a) 42 (b) 36 (6 "<")	none	1 0	7 2	4 4	1 1	18 11	Cd (insufficient data)
F033	(a) 46 (b) 40 (3 "<" ; 1 flagged)	none	2 0	5 1	4 4	1 1	16 9	-
F040	(a) 48 (b) 42	none	0 0	0 0	1 1	0 0	1 1	-
F057	(a) 47 (b) 41 (1 "<")	none	3 0	4 2	0 0	1 1	13 5	-
F063	(a) 47 (b) 41 (5 "<" ; 4 flagged)	none	1 1	0 0	2 2	10 10	26 29	Zn (biased low); Cd (insufficient data)
F064	(a) 48 (b) 42	none	8 8	1 1	3 3	1 1	23 26	Cu (biased high)
F065	nil	none	** see Appendix II **					DATA SUBMITTED LATE
F066	(a) 47 (b) 41	none	4 4	2 2	1 1	1 1	14 16	Cu and Zn (biased low); Hg (biased high)
F084	(a) 48 (b) 42 (1 "<")	none	15 14	2 0	0 0	6 6	46 48	-
F087	(a) 47 (b) 41 (1 "<")	none	2 2	0 0	4 3	4 4	17 18	Cr (biased low)
F094	(a) 48 (b) 42	none	3 0	10 7	1 1	1 1	20 12	Ni (biased high)
N041	nil	all	-	-	-	-	-	-
N090	(a) 48 (b) 42	none	6 6	3 3	5 0	0 0	21 18	Cr (biased low)
N107	(a) 48 (b) 42	none	6 3	6 4	1 1	0 0	20 13	-
N158	(a) 47 (b) 41 (1 "<")	none	5 5	1 1	5 2	2 2	21 21	Cr and Ni (biased low)
N196	(a) 40 (b) 34 (8 "<")	none	4 4	5 3	3 3	1 1	23 24	Cd and Hg (insufficient data)
N197	(a) 47 (b) 41 (1 "<")	none	11 11	1 1	1 1	1 1	28 32	Cd and Hg (biased high)
N200	(a) 48 (b) 42	none	14 8	5 5	1 1	4 4	44 36	Cr, Cu and Pb (biased high); Cd (biased low)
N208	(a) 48 (b) 42 (2 "<" ; 1 flagged)	none	1 1	0 0	2 2	6 6	17 19	-

* H and L flags are counted as half of VH and VL flags, respectively.

(a) with Cr flags included
(b) without Cr flags

Less than values that were flagged are included in the calculation of the % Flagged.

Table 30. Summary of Overall Laboratory Performance.

Lab #	% Flagged"			PERFORMANCE SCORE**	OVERALL PERFORMANCE RATING***	Comments
	PCBs	PAHs	Metals*			
F007	7	29	11	16	Satisfactory	ampules not analyzed for PAHs; poor PCB precision; some low PAH recoveries; 6 "nd" cadmium results.
F033	21	29	9	20	Moderate	5 PAHs not analyzed in sediments; some PAHs biased high; poor PCB and PAH precision; 3 "nd" cadmium results.
F040	29	53	1	28	Moderate	20 "nd" of 119 organic results; low PCB and PAH recoveries.
F057	43	37	5	28	Moderate	52 "nd" of 119 organic results; out-of-control PCBs; some erratic PAHs; low PAH recoveries from ampules.
F063	0	17	29	15	Satisfactory	5 "nd" cadmium results; zinc biased low.
F064	14	12	26	17	Satisfactory	copper biased high; erratic arsenic.
F065	25	16	-	21	Moderate	low PCB recoveries from ampules; trace metal data submitted late, therefore not included.
F066	43	18	16	26	Moderate	PCBs biased high; copper and zinc biased low; mercury biased high; erratic arsenic.
F084	25	46	48	40	Poor	39 "nd" of 100 organic results; low PCB recoveries from ampules; poor PCB precision; poor PAH resolution; PAHs biased high; some PAHs out-of-control; erratic arsenic and zinc.
F087	0	39	18	19	Satisfactory	16 "nd" of 116 organic results; PAHs biased high; some PAHs out-of-control; chromium biased low; erratic lead and nickel.

continues

Table 30 (continued). Summary of Overall Laboratory Performance.

Lab #	% Flagged			PERFORMANCE SCORE**	OVERALL PERFORMANCE RATING***	Comments
	PCBs	PAHs	Metals*			
F094	7	40	12	20	Moderate	poor PCB precision; PAHs biased high; nickel biased high.
N041	0	26	-	13	Satisfactory	76 "nd" of 119 organic results; PAHs biased low; no data submitted for trace metals.
N090	-	24	18	21	Moderate	no data submitted for PCBs; some PAHs biased high; chromium biased low.
N107	0	56	13	23	Moderate	ampules not analyzed for PAHs; PAHs biased high.
N158	0	15	21	12	Satisfactory	chromium and nickel biased low.
N196	50	57	24	44	Poor	9 "nd" of 68 organic results; very low PCB and PAH recoveries; 4 "nd" cadmium results; 4 "nd" mercury results; no organic data submitted for sediments #3, #4 and #5.
N197	58	35	32	42	Poor	23 "nd" of 119 organic results; poor PCB and PAH precision; out-of-control PCBs; low PAH recoveries; cadmium and mercury biased high.
N200	7	14	36	19	Satisfactory	59 "nd" of 112 organic results; poor PAH recoveries; chromium, copper and lead biased high; cadmium biased low.
N208	21	24	19	21	Moderate	low PCB and PAH recoveries; poor PAH precision; erratic mercury.

* Chromium data has been excluded (see text)

** PERFORMANCE SCORE = $(\sum \% \text{Flagged})/3$
(except for labs F065, N041 and N090 who only submitted
data for two of the three classes in this study)

*** OVERALL PERFORMANCE RATING determined as follows:

Satisfactory:	Performance Score < 20
Moderate:	$20 \leq$ Performance Score < 40
Poor:	Performance Score \geq 40

APPENDIX I

List of Invited Participants

Federal Government:

1. Environment Canada
C&P (EPS) Laboratory Services
River Road Environmental Technology Centre
Ottawa, Ontario
2. Environment Canada
C&P (EPS) Laboratory Services
Wastewater Technology Centre
Burlington, Ontario
3. Environment Canada
C&P (EPS) Laboratory Services
West Vancouver, B.C. **No response**
4. Environment Canada
National Water Quality Laboratory
Burlington, Ontario

Provincial Governments:

5. Ontario Ministry of the Environment
Trace Organics QA/QC Section
Rexdale, Ontario

Private Laboratories:

6. AccuTest Laboratories Ltd.
Nepean, Ontario **Inorganic analyses only;
will forward samples for
organic analyses to
Paracel Labs**
7. Acres Analytical Ltd.
Niagara Falls, Ontario
8. AGRI-SERVICE Laboratory Inc.
Kitchener, Ontario **No response**
9. Altech Environmental Consulting Ltd.
Willowdale, Ontario **No response**
10. Aquatic Sciences Inc.
St. Catharines, Ontario **No response**

(continued)

APPENDIX I (continued)
List of Invited Participants

11. Areco Canada Inc.
Nepean, Ontario
12. Barringer Laboratories
Mississauga, Ontario
13. Beak Consultants Ltd.
Mississauga, Ontario
14. Bondar-Clegg & Co. Ltd.
Ottawa, Ontario
15. C.I.L. Inc. **No response**
Mississauga, Ontario
16. Canviro Analytical Laboratories Ltd.
Kitchener, Ontario
17. Clayton Environmental Consultants Ltd.
Windsor, Ontario
18. Concord Scientific Corporation **Declined**
Downsview, Ontario
19. Dearborn Chemicals Company Ltd. **No response**
Mississauga, Ontario
20. DILLON Environmental Laboratories
Mississauga, Ontario
21. Eco-Recherches
Pointe-Claire, P.Q.
22. Enviroclean
London, Ontario
23. Environmental Applications Group
Markham, Ontario
24. Fine Analysis Laboratories
Hannon, Ontario
25. Mann Testing Laboratories
Mississauga, Ontario
26. Monenco Analytical Labs **No response**
Woodbridge, Ontario

(continued)

APPENDIX I (continued)

List of Invited Participants

- | | |
|---|---|
| 27. Novalab Ltee
Lachine, P.Q. | Declined; Inorganic
analyses not yet on
stream |
| 28. Nucro-Technics Ltd.
Scarborough, Ontario | No response |
| 29. OceanChem Group
Dartmouth, Nova Scotia | |
| 30. ORTECH International
Mississauga, Ontario | No response |
| 31. Paracel Labs
Nepean, Ontario | No response (see
AccuTest Laboratories) |
| 32. Pluritec Laboratoire Ltee
Cap-de-la-Madelaine, Quebec | No response |
| 33. Pollutech Ltd.
Oakville, Ontario | No response |
| 34. Proctor & Redfern Ltd.
Don Mills, Ontario | |
| 35. Sussex Environmental Services Inc.
London, Ontario | No response |
| 36. Technical Services Laboratories
Mississauga, Ontario | |
| 37. Water and Earth Sciences Assoc. Ltd.
Carp, Ontario | No response |
| 38. Wellington Environmental Consultants, Inc.
Guelph, Ontario | No response |
| 39. XRAL Environmental
Don Mills, Ontario | No response |
| 40. Zenon Environmental Inc.
Burlington, Ontario | |

APPENDIX II

The following results were received from laboratory F065 after the data summary was prepared and released for distribution. These data are reproduced below for information only as they are not included or evaluated in this report.

April 1, 1990

Trace Metal Sediment Sample Results for Laboratory F065

Parameter	Sediment Samples (ug/g)					
	10	11	12	13	14	15
Mercury	0.8	0.1	1.3	0.3	0.1	<0.1
Lead	186	62	104	138	64	38
Zinc	840	709	217	969	785	88
Chromium	76	38	37	86	44	36
Copper	98	506	61	63	537	11
Cadmium	5	2	2	3	2	<1.0
Nickel	30	749	48	28	661	40
Arsenic	5.9	33	19	5.0	2.8	8.1

APPENDIX III

Glossary of Terms and Symbols

Legend for Tables 9-25:

*	Chrysene/Triphenylene
**	Peaks not resolved; total value reported
nd	not detected
-	not analyzed
---	data not available
Naph-	Naphthalene
Acen-	Acenaphthylene
Ace-	Acenaphthene
Fl-	Fluorene
Phen-	Phenanthrene
Anth-	Anthracene
Fluor-	Fluoranthene
Pyrene	Pyrene
B(a)A	Benzo(a)anthracene
Chrys-	Chrysene
B(b)F	Benzo(b)fluoranthene
B(k)F	Benzo(k)fluoranthene
B(a)P	Benzo(a)pyrene
I(cd)P	Indeno(1,2,3-c,d)pyrene
D(ah)A	Dibenzo(a,h)anthracene
B(ghi)P	Benzo(g,h,i)perylene

Explanation of Terms for Data Evaluation Techniques:

A set of results is said to be biased when the set exhibits a tendency to be either higher or lower than some standard. The standard which has been used in the analysis of our studies thus far has been the performance of all other participating laboratories. The ranking procedure employed in testing for bias and the rationale for evaluating laboratories' performances by ranking results are described in more detail elsewhere¹⁸⁻²⁰ but a brief synopsis is presented below. In our use of the procedure, there is about one chance in twenty of deeming a set of results biased, when in fact it is not, (i.e. $\alpha=0.05$).

Ranking is a non-parametric statistical technique used for the detection of pronounced systematic error (bias) in interlaboratory studies. According to Youden's procedure¹⁰, rank 1 is given to the laboratory that provided the lowest result, rank 2 to the next lowest. In the case of a tie, the average rank is given to the tied laboratories. Results with a "<" (less than) sign are not ranked. For each parameter, the total rank of each laboratory is the sum of the individual ranks for each sample. In the present case of the trace metals in Study DQC-5, statistically, the permissible score limits for 17 laboratories and six test samples are approximately 20 and 88 (for a full set of data at 5% probability). A laboratory with a score lower than 20 is identified as biased low for that particular set of data. Similarly, a laboratory with a total rank higher than 88 is identified as biased high. In both cases, their results are classified as outliers. In cases where a laboratory did not provide all of the results, or where some of the results were not ranked, the average rank instead of total rank was used for the determination of bias statements.

The more comparable laboratories should have ranks in the middle rather than on the extreme ends. However, laboratories with middle ranks do not necessarily mean that they provide more consistent results since very high results (high ranks) and very low results (low ranks) would average out to yield a total rank close to the median. Therefore, ranking alone is not sufficient to determine the performance of a laboratory.

Flagging (for inorganic analyses): When the true values of constituents in test samples are unknown, individual results can be evaluated by a peer group assessment technique in terms of their absolute differences from the interlaboratory medians. Medians are chosen rather than means since they are not influenced by a moderate number of extreme values. Using this assumption then, that the median values establish the correct target values, it follows that at all concentrations, there must exist a basic acceptable error (BAE) which reflects an allowable deviation from the median. This basic acceptable error is incremented at a concentration level referred to as the lower limit for use of basic acceptable error (LLBAE). The LLBAE for each parameter in a

round-robin study is usually set at the lower end of the range of medians in the test samples. Since it appears that for almost all substances, the absolute variability of the analytical method increases with increasing concentrations, the allowable error must therefore be increased using a concentration error increment (CEI). The amount that is used to augment the BAE for each sample is calculated by multiplying the CEI by the difference between the target value (i.e. the median of that particular sample) and the LLBAE. The values chosen for BAE and CEI are derived from the overall results received from the participants and are selected such that only the most extreme laboratory results are flagged. Further discussion on this evaluation technique has been reported elsewhere¹⁹.

For the inorganic analyses in this study, LLBAE was set at the lower end of the range of medians in the test samples. For most of the metals, a 20-25% error at LLBAE was considered acceptable, and thus, was used as the BAE. However, for cadmium, chromium, lead and mercury, a 30-50% error at LLBAE was used as the BAE since their concentrations in this study were relatively low or, because the participating laboratories' capabilities for the analysis of that particular metal varied widely. (Refer to Table 26 for a list of the reported detection limits for each laboratory in this study). For samples whose medians were at or below LLBAE, the results were evaluated according to the following formulae:

Acceptable:		X	≤	BAE
H or L:	BAE	<	X	≤ 1.5 X BAE
VH or VL:		X	>	1.5 x BAE

where X represents the absolute difference between sample and median results. In this study, the samples whose medians were above the LLBAE used a CEI that was arbitrarily set at 0.1 for all but one of the metals of interest. 0.2 was used as the CEI for nickel because of the wide concentration range between the highest and lowest samples. Sample results were then evaluated by the above formulae except that the augmented BAE was used instead of the BAE.

Therefore, by this flagging technique, all results were graded into the following three groups in the order of decreasing accuracy: (1) results with no flags, (2) results with H (high) or L (low) flags, and (3) results with VH (very high) or VL (very low) flags. Computer printouts which include sample data, individual, total and average ranks, and flagging and biased statements for each metal in this study are given in Tables 17-24. A summary of individual flags and bias statements for each laboratory on their trace metal analyses is given in Table 29.

Flagging (for organic analyses): In order to assess the organic results provided by each laboratory, a modified approach to the technique of flagging was used. To begin, each laboratory's results were recalculated to a "percent recovery" in relation to the interlaboratory medians. Arbitrarily, in this study, recoveries within $\pm 50\%$ of the median were deemed to be satisfactory and any values beyond this range were flagged. Hence, the results were evaluated according to the following rating groups:

Very High (VH)	X	\geq	200%
High (H)	150%	\leq X <	200%
Satisfactory (no flags)	50%	< X <	150%
Low (L)	25%	< X \leq	50%
Very Low (VL)	X	\leq	25%

Thus, as was done for the inorganic data, all organic results were graded into three groups in the order of decreasing accuracy as follows: (1) results with no flags, (2) results with H (high) or L (low) flags, and (3) results with VH (very high) or VL (very low) flags. Appendix IV provides a complete listing of each laboratory's recovery data in relation to the interlaboratory medians and their resultant flags. Appendix IV also provides a listing of each laboratory's "percent recoveries" in relation to the reference or design values of the organic samples in this study. It was found that, because the

medians, for the most part, accurately reflected the reference and design values of the samples in this study, there was not a significant difference in the summary of flags if the latter recovery values were used in assessing each laboratory's results for flags. A summary of individual flags for each laboratory on their organic data is given in Tables 27 and 28, for PCB and PAH analyses, respectively.

Performance Score: To rate the laboratories according to their overall competence in the analysis of all three classes of parameters, a Performance Score was calculated using the percentage of flags obtained in each class:

$$\text{Performance Score} = (\Sigma \% \text{Flagged})/3$$

By calculating an average rather than a sum of the % flagged, those laboratories who had not submitted data for one of the classes did not unfairly receive a better rating in their performance score.

The Overall Performance Rating, for classifying the participating laboratories according to their relative competence in the analysis of more than one class of parameter, was then determined as follows:

Satisfactory: Performance score < 20

Moderate: 20 ≤ Performance score < 40

Poor: Performance Score ≥ 40

Because this assessment is a peer appraisal technique, the limits for each rating were arbitrarily set, such that a few participants would receive the highest rating, and the majority would lie in the moderate range. It is very important to note, however, that some participants may receive a satisfactory rating, despite having provided an incomplete set of results, a large number of 'not detected' values, and/or be biased for one or a number of parameters.

APPENDIX IV

Table IV-1. % Recovery for Laboratory F007 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	-	-
Acenaphthylene	---	---	---	---	---	-	-
Acenaphthene	---	---	---	---	---	-	-
Fluorene	---	---	---	---	---	-	-
Phenanthrene	115	147	126	114	127	-	-
Anthracene	135	167	793	109	134	-	-
Fluoranthene	74	68	58	75	61	-	-
Pyrene	78	70	15	67	67	-	-
Benzo (a) anthracene	87	53	8	79	77	-	-
Chrysene	57	51	9	56	48	-	-
Benzo (b) fluoranthene	91	53	6	17	51	-	-
Benzo (k) fluoranthene	84	56	12	18	83	-	-
Benzo (a) pyrene	88	50	7	15	64	-	-
Indeno (123-cd) pyrene	117	55	5	13	63	-	-
Dibenzo (ah) anthracene	187	95	nd	nd	59	-	-
Benzo (ghi) perylene	149	72	8	19	79	-	-

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	83	130	128	83	72	74	81

Table IV-2. % Recovery for Laboratory F007 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	217 VH	240 VH	220 VH	260 VH	204 VH	-	-
Acenaphthylene	nd	64	30 L	53	70	-	-
Acenaphthene	163 H	141	88	114	118	-	-
Fluorene	128	143	nd	110	114	-	-
Phenanthrene	143	154 H	110	128	132	-	-
Anthracene	136	146	407 VH	105	110	-	-
Fluoranthene	77	110	81	105	82	-	-
Pyrene	111	101	25 VL	101	70	-	-
Benzo (a) anthracene	124	70	12 VL	94	88	-	-
Chrysene	100	83	16 VL	90	75	-	-
Benzo (b) fluoranthene	97	100	8 VL	15 VL	63	-	-
Benzo (k) fluoranthene	129	82	12 VL	22 VL	99	-	-
Benzo (a) pyrene	112	58	7 VL	14 VL	67	-	-
Indeno (123-cd) pyrene	112	68	5 VL	11 VL	72	-	-
Dibenzo (ah) anthracene	187 H	90	nd	nd	48 L	-	-
Benzo (ghi) perylene	160 H	118	9 VL	17 VL	105	-	-

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	91	148	168 H	107	74	85	79

Table IV-3. % Recovery for Laboratory F033 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	-	-
Acenaphthylene	---	---	---	---	---	101	73
Acenaphthene	---	---	---	---	---	123	80
Fluorene	---	---	---	---	---	87	68
Phenanthrene	85	99	114	101	117	107	74
Anthracene	83	123	74	66	146	-	-
Fluoranthene	96	62	60	65	83	56	73
Pyrene	89	79	67	66	97	43	71
Benzo (a) anthracene	119	134	74	73	202	-	-
Chrysene	111	91	112	95	152	-	-
Benzo (b) fluoranthene	114	126	160	113	159	131	35
Benzo (k) fluoranthene	276	188	310	201	353	131	47
Benzo (a) pyrene	200	150	341	161	387	140	45
Indeno (123-cd) pyrene	-	-	-	-	-	66	26
Dibenzo (ah) anthracene	-	-	-	-	-	-	-
Benzo (ghi) perylene	-	-	-	-	-	106	39

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	93	80	149	77	252	99	107

Table IV-4. % Recovery for Laboratory F033 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	65	nd	62	100	105	-	-
Acenaphthylene	-	-	-	-	-	115	95
Acenaphthene	-	-	-	-	-	97	88
Fluorene	208 VH	nd	46 L	280 VH	nd	106	99
Phenanthrene	106	103	100	114	122	124	93
Anthracene	84	107	38 L	64	119	-	-
Fluoranthene	100	100	84	92	112	81	111
Pyrene	127	113	111	99	102	49 L	104
Benzo (a) anthracene	170 H	176 H	107	87	230 VH	-	-
Chrysene	192 H	148	195 H	153 H	240 VH	-	-
Benzo (b) fluoranthene	121	237 VH	197 H	100	197 H	180 H	74
Benzo (k) fluoranthene	425 VH	275 VH	299 VH	249 VH	421 VH	211 VH	85
Benzo (a) pyrene	254 VH	174 H	349 VH	156 H	407 VH	160 H	68
Indeno (123-cd) pyrene	-	-	-	-	-	74	50 L
Dibenzo (ah) anthracene	-	-	-	-	-	-	-
Benzo (ghi) perylene	-	-	-	-	-	103	60

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	102	91	196 H	100	257 VH	113	106

Table IV-5. % Recovery for Laboratory F040 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	40	40
Acenaphthylene	---	---	---	---	---	51	61
Acenaphthene	---	---	---	---	---	nd	64
Fluorene	---	---	---	---	---	37	42
Phenanthrene	21	21	18	18	24	50	80
Anthracene	28	38	155	32	38	103	nd
Fluoranthene	23	22	2	21	22	36	41
Pyrene	21	22	16	18	23	37	41
Benzo (a) anthracene	15	17	16	14	19	17	187
Chrysene	15	18	18	18	19	31	39
Benzo (b) fluoranthene	19	21	28	24	22	47	35
Benzo (k) fluoranthene	31	50	39	42	42	55	55
Benzo (a) pyrene	16	21	23	16	25	52	62
Indeno (123-cd) pyrene	31	18	47	18	29	89	102
Dibenzo (ah) anthracene	18	nd	19	nd	nd	nd	nd
Benzo (ghi) perylene	24	15	39	21	23	64	69

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	38	32	39	37	34	63	78

Table IV-6. % Recovery for Laboratory F040 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	28 L	23 VL	20 VL	29 L	25 VL	51	45 L
Acenaphthylene	nd	nd	17 VL	nd	nd	58	79
Acenaphthene	nd	nd	nd	nd	nd	nd	70
Fluorene	nd	nd	11 VL	nd	nd	45 L	62
Phenanthrene	27 L	22 VL	16 VL	20 VL	25 VL	58	100
Anthracene	28 L	33 L	80	31 L	31 L	108	nd
Fluoranthene	24 VL	35 L	2 VL	29 L	29 L	52	63
Pyrene	29 L	32 L	27 L	27 L	24 VL	43 L	60
Benzo (a) anthracene	21 VL	22 VL	23 VL	17 VL	21 VL	35 L	429 VH
Chrysene	26 L	29 L	32 L	28 L	29 L	41 L	71
Benzo (b) fluoranthene	21 VL	39 L	34 L	21 VL	27 L	65	74
Benzo (k) fluoranthene	48 L	73	38 L	52	50 L	88	100
Benzo (a) pyrene	20 VL	24 VL	23 VL	15 VL	26 L	59	92
Indeno (123-cd) pyrene	30 L	22 VL	47 L	15 VL	33 L	100	192 H
Dibenzo (ah) anthracene	18 VL	nd	18 VL	nd	nd	nd	nd
Benzo (ghi) perylene	26 L	25 VL	46 L	19 VL	31 L	63	105

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	42 L	37 L	52	48 L	35 L	71	77

Table IV-7. % Recovery for Laboratory F057 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	52	44
Acenaphthylene	---	---	---	---	---	36	22
Acenaphthene	---	---	---	---	---	95	nd
Fluorene	---	---	---	---	---	50	17
Phenanthrene	160	nd	32	nd	nd	54	98
Anthracene	455	nd	1221	nd	nd	21	370
Fluoranthene	148	108	75	nd	63	44	53
Pyrene	111	76	47	nd	nd	34	18
Benzo (a) anthracene	54	nd	25	200	nd	26	29
Chrysene	81	nd	50	nd	nd	32	53
Benzo (b) fluoranthene	134	99	102	nd	107	56	42
Benzo (k) fluoranthene	312	nd	nd	nd	nd	44	55
Benzo (a) pyrene	140	nd	70	nd	nd	14	62
Indeno (123-cd) pyrene	184	nd	nd	nd	73	54	31
Dibenzo (ah) anthracene	279	nd	nd	nd	nd	18	0.9
Benzo (ghi) perylene	94	nd	nd	nd	nd	0.005	0.2

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	68	1875	2063	71	1423	117	106

Table IV-8. % Recovery for Laboratory F057 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	nd	1783 VH	nd	nd	nd	65	50 L
Acenaphthylene	nd	nd	nd	nd	nd	42 L	29 L
Acenaphthene	nd	nd	nd	nd	nd	75	nd
Fluorene	nd	nd	127	nd	nd	61	25 VL
Phenanthrene	200 VH	nd	28 L	nd	nd	62	123
Anthracene	459 VH	nd	627 VH	nd	nd	22 VL	360 VH
Fluoranthene	153 H	175 H	105	nd	85	63	81
Pyrene	157 H	109	78	nd	nd	39 L	27 L
Benzo (a) anthracene	78	nd	37 L	238 VH	nd	52	67
Chrysene	141	nd	88	nd	nd	42 L	98
Benzo (b) fluoranthene	142	187 H	126	nd	132	78	89
Benzo (k) fluoranthene	481 VH	nd	nd	nd	nd	71	100
Benzo (a) pyrene	178 H	nd	72	nd	nd	16 VL	92
Indeno (123-cd) pyrene	176 H	nd	nd	nd	84	60	58
Dibenzo (ah) anthracene	278 VH	nd	nd	nd	nd	23 VL	1 VL
Benzo (ghi) perylene	101	nd	nd	nd	nd	0.005 VL	0.2 VL

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	75	2125 VH	2706 VH	92	1453 VH	133	104

Table IV-9. % Recovery for Laboratory F063 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	128	101
Acenaphthylene	---	---	---	---	---	92	81
Acenaphthene	---	---	---	---	---	85	106
Fluorene	---	---	---	---	---	104	85
Phenanthrene	78	84	100	81	93	103	100
Anthracene	158	105	186	121	150	31	nd
Fluoranthene	144	128	125	143	146	99	92
Pyrene	147	140	126	139	161	117	102
Benzo (a) anthracene	74	81	76	86	91	56	42
Chrysene	46	53	50	51	55	112	78
[Benzo (b) fluoranthene + Benzo (k) fluoranthene]	68*	70*	73*	84*	66*	150*	64*
Benzo (a) pyrene	69	86	65	91	81	124	83
Indeno (123-cd) pyrene	54	59	40	70	56	122	41
Dibenzo (ah) anthracene	72	95	56	90	89	192	nd
Benzo (ghi) perylene	51	63	41	71	57	173	92

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	73	61	61	77	85	69	77

Table IV-10. % Recovery for Laboratory F063 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	102	69	95	25 VL	95	161	H 113
Acenaphthylene	205 VH	145	176 H	163 H	240 VH	106	105
Acenaphthene	76	62	40 L	55	74	67	116
Fluorene	95	90	112	100	121	128	124
Phenanthrene	98	88	88	92	97	120	125
Anthracene	159 H	92	95	117	123	32	L nd
Fluoranthene	150 H	208 VH	173 H	202 VH	196 H	142	141
Pyrene	208 VH	201 VH	210 VH	208 VH	169 H	134	149
Benzo (a) anthracene	105	107	110	102	104	113	95
Chrysene	80	86	86	81	86	146	143
[Benzo (b) fluoranthene + Benzo (k) fluoranthene]	82*	121*	83*	83*	81*	216*	VH 128*
Benzo (a) pyrene	88	99	66	88	85	141	123
Indeno (123-cd) pyrene	52	73	40 L	58	65	136	77
Dibenzo (ah) anthracene	72	90	52	83	73	252	VH nd
Benzo (ghi) perylene	55	104	49 L	66	76	168	H 140

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	80	69	80	99	87	78	75

Table IV-11. % Recovery for Laboratory F064 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	111	101
Acenaphthylene	---	---	---	---	---	101	101
Acenaphthene	---	---	---	---	---	148	106
Fluorene	---	---	---	---	---	103	106
Phenanthrene	100	97	139	97	99	106	100
Anthracene	99	97	173	103	93	113	103
Fluoranthene	96	99	128	100	91	100	92
Pyrene	89	102	120	89	94	102	92
Benzo (a) anthracene	55	70	69	61	62	52	42
Chrysene	71	89	146	80	78	83	52
Benzo (b) fluoranthene	72	80	172	88	72	84	53
Benzo (k) fluoranthene	54	80	146	73	67	59	55
Benzo (a) pyrene	53	82	137	73	72	73	41
Indeno (123-cd) pyrene	39	55	155	54	53	70	41
Dibenzo (ah) anthracene	36	74	94	51	54	51	101
Benzo (ghi) perylene	43	61	156	65	51	100	46

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	134	97	98	110	81	54	510

Table IV-12. % Recovery for Laboratory F064 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #		
	1	2	3	4	5	8A	9A	
Naphthalene	65	58	76	74	17	VL	140	113
Acenaphthylene	53	48	L 45	L 60	73		116	132
Acenaphthene	70	50	L 34	L 57	50	L	117	116
Fluorene	100	370	VH 60	98	82		126	155
Phenanthrene	125	101	122	109	103		123	125
Anthracene	100	84	89	99	76		118	100
Fluoranthene	100	161	H 178	H 141	122		144	141
Pyrene	127	147	199	H 134	98		117	134
Benzo (a) anthracene	79	92	100	73	70		105	95
Chrysene	123	144	254	VH 129	122		109	95
Benzo (b) fluoranthene	76	151	H 212	VH 78	89		116	111
Benzo (k) fluoranthene	83	117	141	91	79		96	100
Benzo (a) pyrene	67	95	141	70	75		83	62
Indeno (123-cd) pyrene	38	L 68	154	H 45	L 61		79	77
Dibenzo (ah) anthracene	36	L 70	87	48	L 44	L	66	120
Benzo (ghi) perylene	46	L 100	185	H 60	67		97	70

PCBs	Sample #					Ampule #		
	1	2	3	4	5	6	7	
Total PCBs	147	110	128	143	83	61	502	VH

Table IV-13. % Recovery for Laboratory F065 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	71	86
Acenaphthylene	---	---	---	---	---	119	119
Acenaphthene	---	---	---	---	---	172	136
Fluorene	---	---	---	---	---	58	68
Phenanthrene	72	89	89	89	99	88	96
Anthracene	86	94	103	104	121	87	35
Fluoranthene	61	54	58	71	80	69	60
Pyrene	62	55	56	66	92	76	63
Benzo (a) anthracene	25	105	88	101	118	85	96
Chrysene	18	78	81	69	78	76	102
[Benzo (b) fluoranthene + Benzo (k) fluoranthene]	28*	87*	101*	95*	100*	87*	88*
Benzo (a) pyrene	23	87	71	78	95	88	68
Indeno (123-cd) pyrene	45	167	150	138	153	142	61
Dibenzo (ah) anthracene	41	106	132	108	154	159	187
Benzo (ghi) perylene	55	201	189	160	158	198	62

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	89	170	66	74	182	36	nd

Table IV-14. % Recovery for Laboratory F065 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	113	113	105	55	71	90	96
Acenaphthylene	105	119	98	124	243 VH	136	154 H
Acenaphthene	89	106	67	81	102	136	149
Fluorene	60	95	153 H	83	79	72	100
Phenanthrene	90	94	79	100	104	102	120
Anthracene	87	82	53	100	99	91	34 L
Fluoranthene	63	87	81	100	107	100	91
Pyrene	88	79	93	99	96	87	93
Benzo (a) anthracene	35 L	138	128	120	135	171 H	220 VH
Chrysene	32 L	126	141	111	122	99	185 H
[Benzo (b) fluoranthene + Benzo (k) fluoranthene]	34* L	150* H	114*	94*	122*	126*	176* H
Benzo (a) pyrene	30 L	101	73	76	100	100	101
Indeno (123-cd) pyrene	43 L	207 VH	150 H	115	177 H	159 H	115
Dibenzo (ah) anthracene	41 L	100	122	100	126	208 VH	223 VH
Benzo (ghi) perylene	59	331 VH	224 VH	147	210 VH	192 H	95

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	98	193 H	87	95	186 H	40 L	nd

Table IV-15. % Recovery for Laboratory F066 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	126	165
Acenaphthylene	---	---	---	---	---	101	59
Acenaphthene	---	---	---	---	---	190	91
Fluorene	---	---	---	---	---	114	55
Phenanthrene	82	96	147	70	78	156	62
Anthracene	77	84	195	69	74	185	23
Fluoranthene	57	60	95	54	48	102	55
Pyrene	55	69	101	54	54	119	63
Benzo (a) anthracene	103	118	163	103	105	43	108
Chrysene	66	83	156	74	66	83	55
[Benzo (b) fluoranthene + Benzo (k) fluoranthene]	83*	89*	145*	85*	69*	100*	102*
Benzo (a) pyrene	124	113	358	113	105	289	66
Indeno (123-cd) pyrene	112	119	156	120	104	89	45
Dibenzo (ah) anthracene	121	143	197	108	151	24	25
Benzo (ghi) perylene	124	13	186	136	110	143	71

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	127	136	113	181	139	175	283

Table IV-16. % Recovery for Laboratory F066 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	94	100	175 H	82	84	159 H	185 H
Acenaphthylene	64	109	102	90	100	116	76
Acenaphthene	54	50 L	54	34 L	44 L	150 H	100
Fluorene	92	70	220 VH	58	71	139	81
Phenanthrene	102	100	129	79	82	181 H	78
Anthracene	77	74	100	67	61	194 H	22 VL
Fluoranthene	59	98	132	76	65	148	84
Pyrene	77	99	169 H	82	56	136	93
Benzo (a) anthracene	147	154 H	237 VH	123	120	87	248 VH
Chrysene	115	135	271 VH	119	103	109	100
[Benzo (b) fluoranthene + Benzo (k) fluoranthene]	100*	155* H	163* H	84*	85*	144*	202* VH
Benzo (a) pyrene	157 H	131	367 VH	109	111	329 VH	98
Indeno (123-cd) pyrene	107	148	156 H	100	121	100	85
Dibenzo (ah) anthracene	120	135	183 H	100	124	31 L	30 L
Benzo (ghi) perylene	133	21 VL	221 VH	125	146	139	109

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	140	154 H	148	234 VH	142	199 H	279 VH

Table IV-17. % Recovery for Laboratory F084 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	30	nd
Acenaphthylene	---	---	---	---	---	70	nd
Acenaphthene	---	---	---	---	---	74	nd
Fluorene	---	---	---	---	---	153	nd
Phenanthrene	74	68	405	57	68	174	nd
[Anthracene	nd	1207	885	385	nd	135*	nd
+ Fluoranthene]	131	52	80	37	69		nd
Pyrene	152	150	64	109	193	92	nd
[Benzo (a) anthracene	269*	77*	59*	53*	64*	91*	nd
+ Chrysene]							nd
[Benzo (b) fluoranthene	432*	84*	83*	234*	nd*	135*	nd
+ Benzo (k) fluoranthene]							nd
Benzo (a) pyrene	nd	123	358	869	319	289	nd
[Indeno (123-cd) pyrene	588*	100*	nd*	926*	434*	153*	nd
+ Dibenzo (ah) anthracene]							nd
Benzo (ghi) perylene	580	81	51	nd	nd	186	nd

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	112	96	76	114	158	0.015	nd

Table IV-18. % Recovery for Laboratory F084 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	nd	nd	82	nd	nd	38 L	nd
Acenaphthylene	nd	nd	nd	nd	nd	80	nd
Acenaphthene	nd	382 VH	294 VH	423 VH	nd	58	nd
Fluorene	nd	nd	100	nd	nd	188 H	nd
Phenanthrene	92	71	356 VH	64	71	203 VH	nd
[Anthracene	nd	1051 VH	455 VH	370 VH	nd	193* H	nd
+ Fluoranthene]	136	84	112	52	93		nd
Pyrene	216 VH	216 VH	107	163 H	203 VH	105	nd
[Benzo (a) anthracene	429* VH	114*	95*	74*	87*	137*	nd
+ Chrysene]							nd
[Benzo (b) fluoranthene	518* VH	145*	93*	231* VH	nd*	195* H	nd
+ Benzo (k) fluoranthene]							nd
Benzo (a) pyrene	nd	142	367 VH	841 VH	335 VH	329 VH	nd
[Indeno (123-cd) pyrene	566* VH	117*	nd*	788* VH	465* VH	176* H	nd
+ Dibenzo (ah) anthracene]							nd
Benzo (ghi) perylene	626 VH	133	61	nd	nd	181 H	nd

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	124	109	100	148	161 H	0.017 VL	nd

Table IV-19. % Recovery for Laboratory F087 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	84	82
Acenaphthylene	---	---	---	---	---	82	41
Acenaphthene	---	---	---	---	---	127	42
Fluorene	---	---	---	---	---	91	47
Phenanthrene	31	72	620	53	89	85	48
Anthracene	nd	362	929	nd	586	87	nd
Fluoranthene	102	114	69	105	107	4	63
Pyrene	42	60	39	94	97	85	69
Benzo (a) anthracene	52	93	226	150	127	50	50
Chrysene	30	167	265	334	559	74	52
[Benzo (b) fluoranthene	94		200			96	82
+ Benzo (k) fluoranthene]	221	141*	118	157*	159*	74	69
Benzo (a) pyrene	615	1002	445	620	1069	85	81
Indeno (123-cd) pyrene	77	96	92	154	100	86	77
Dibenzo (ah) anthracene	nd	nd	nd	nd	nd	75	nd
Benzo (ghi) perylene	351	249	260	456	235	114	110

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	129	108	71	106	98	104	125

Table IV-20. % Recovery for Laboratory F087 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #							
	1	2	3	4	5	8A	9A						
Naphthalene	142	258	VH	140	160	H	198	H	106	92			
Acenaphthylene	945	VH	nd	102	867	VH	nd	94	53				
Acenaphthene	370	VH	nd	1779	VH	nd	100	47	L				
Fluorene	1180	VH	1000	VH	496	VH	nd	111	68				
Phenanthrene	39	L	75	545	VH	60	93	99	60				
Anthracene	nd	315	VH	477	VH	nd	479	VH	91	nd			
Fluoranthene	106	185	H	95	148	144	6	VL	97				
Pyrene	60	86	65	141	102	97	101						
Benzo (a) anthracene	73	122	329	VH	178	H	144	100	114				
Chrysene	53	270	VH	460	VH	534	VH	879	VH	97	95		
[Benzo (b) fluoranthene	100	244*	VH	246	VH	155*	H	194*	H	132	174	H	
+ Benzo (k) fluoranthene]	341	VH	113	119	125								
Benzo (a) pyrene	782	VH	1162	VH	456	VH	600	VH	1124	VH	97	120	
Indeno (123-cd) pyrene	74	119	92	128	116	97	146						
Dibenzo (ah) anthracene	nd	nd	nd	nd	nd	98	nd						
Benzo (ghi) perylene	379	VH	410	VH	308	VH	419	VH	314	VH	111	168	H

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	143	123	93	137	100	119	123

Table IV-21. % Recovery for Laboratory F094 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	104	113
Acenaphthylene	---	---	---	---	---	96	89
Acenaphthene	---	---	---	---	---	146	104
Fluorene	---	---	---	---	---	91	104
[Phenanthrene	124	119	142	119	123	117*	137*
+ Anthracene]	107	121	159	125	121		
Fluoranthene	104	100	108	111	102	86	68
Pyrene	100	116	99	107	116	90	83
Benzo (a) anthracene	134	136	160	124	136	65	40
Chrysene	68	57	130	63	64	88	1021
[Benzo (b) fluoranthene	162*	208*	224*	210*	203*	85*	1305*
+ Benzo (k) fluoranthene]							
Benzo (a) pyrene	188	297	364	261	289	125	2312
Indeno (123-cd) pyrene	175	190	199	169	213	102	2303
Dibenzo (ah) anthracene	101	160	122	144	160	78	1520
Benzo (ghi) perylene	168	205	226	187	197	107	3641

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	96	107	84	72	182	95	112

Table IV-22. % Recovery for Laboratory F094 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	291 VH	283 VH	252 VH	405 VH	288 VH	131	126
Acenaphthylene	94	91	107	100	100	111	116
Acenaphthene	120	118	118	127	118	115	114
Fluorene	208 VH	233 VH	226 VH	240 VH	250 VH	111	152 H
[Phenanthrene	154 H	125	125	135	128	136*	164* H
+ Anthracene]	108	105	82	120	99		
Fluoranthene	108	162 H	150 H	157 H	137 H	123	103
Pyrene	142	167 H	164 H	161 H	122 H	103	122
Benzo (a) anthracene	192 H	179 H	233 VH	148	155 H	131	90
Chrysene	118	93	225 VH	100	100	114	1864 VH
[Benzo (b) fluoranthene	195* H	360* VH	251* VH	208* VH	248* VH	122*	2596* VH
+ Benzo (k) fluoranthene]							
Benzo (a) pyrene	239 VH	344 VH	373 VH	252 VH	304 VH	142	3446 VH
Indeno (123-cd) pyrene	167 H	236 VH	199 H	140	246 VH	114	4346 VH
Dibenzo (ah) anthracene	100	151 H	113	133	131	102	1807 VH
Benzo (ghi) perylene	181 H	337 VH	268 VH	172 H	262 VH	103	5579 VH

* Percentage recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	106	121	111	93	186 H	108	110

Table IV-23. % Recovery for Laboratory N041 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	36	nd
Acenaphthylene	---	---	---	---	---	28	nd
Acenaphthene	---	---	---	---	---	71	nd
Fluorene	---	---	---	---	---	48	nd
Phenanthrene	13	nd	119	nd	34	40	nd
Anthracene	nd	103	274	67	69	nd	nd
Fluoranthene	nd	nd	25	20	22	82	nd
Pyrene	27	36	37	31	105	92	nd
Benzo (a) anthracene	nd	22	46	nd	nd	nd	nd
Chrysene	nd	nd	40	nd	nd	78	nd
Benzo (b) fluoranthene	nd	nd	35	nd	nd	68	nd
Benzo (k) fluoranthene	nd	nd	nd	nd	nd	nd	nd
Benzo (a) pyrene	68	29	nd	63	51	nd	nd
Indeno (123-cd) pyrene	nd	nd	21	nd	nd	nd	nd
Dibenzo (ah) anthracene	nd	nd	nd	nd	nd	nd	nd
Benzo (ghi) perylene	nd	nd	33	nd	nd	58	nd

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	107	71	55	107	68	100	nd

Table IV-24. % Recovery for Laboratory N041 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	nd	nd	43 L	nd	nd	46 L	nd
Acenaphthylene	nd	nd	nd	nd	nd	33 L	nd
Acenaphthene	nd	nd	nd	nd	nd	56	nd
Fluorene	nd	nd	40 L	nd	nd	58	nd
Phenanthrene	16 VL	nd	104	nd	36 L	46 L	nd
Anthracene	nd	90	141	65	56	nd	nd
Fluoranthene	nd	nd	35 L	28 L	30 L	118	nd
Pyrene	38 L	52	62	47 L	111	105	nd
Benzo (a) anthracene	nd	28 L	67	nd	nd	nd	nd
Chrysene	nd	nd	69	nd	nd	102	nd
Benzo (b) fluoranthene	nd	nd	43 L	nd	nd	94	nd
Benzo (k) fluoranthene	nd	nd	nd	nd	nd	nd	nd
Benzo (a) pyrene	86	34 L	nd	61	54	nd	nd
Indeno (123-cd) pyrene	nd	nd	21 VL	nd	nd	nd	nd
Dibenzo (ah) anthracene	nd	nd	nd	nd	nd	nd	nd
Benzo (ghi) perylene	nd	nd	40 L	nd	nd	56	nd

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	118	80	72	138	70	114	nd

Table IV-25. % Recovery for Laboratory N090 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	-	-
Acenaphthylene	---	---	---	---	---	127	93
Acenaphthene	---	---	---	---	---	148	80
Fluorene	---	---	---	---	---	36	25
Phenanthrene	107	114	103	109	121	86	62
Anthracene	236	283	395	244	272	267	134
Fluoranthene	120	115	122	117	117	97	75
Pyrene	114	110	101	100	115	90	67
Benzo (a) anthracene	66	74	58	98	88	56	44
Chrysene	105	100	153	108	117	77	57
[Benzo (b) fluoranthene + Benzo (k) fluoranthene]	143*	142*	167*	169*	158*	117	89
Benzo (a) pyrene	109	127	131	120	133	81	60
Indeno (123-cd) pyrene	109	123	112	126	139	105	76
Dibenzo (ah) anthracene	55	168	nd	91	nd	99	75
Benzo (ghi) perylene	56	61	62	74	67	92	71
						81	60

* Percent recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	-	-	-	-	-	-	-

Table IV-26. % Recovery for Laboratory N090 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	-	-	-	-	-	-	-
Acenaphthylene	23 VL	48 L	11 VL	50 L	47 L	145	121
Acenaphthene	100	76	nd	114	106	117	88
Fluorene	34 L	60	75	70	82	44 L	37 L
Phenanthrene	134	119	91	123	126	100	78
Anthracene	238 VH	246 VH	203 VH	235 VH	223 VH	280 VH	130
Fluoranthene	125	187 H	169 H	164 H	157 H	140	114
Pyrene	161 H	158 H	168 H	151 H	121	103	99
Benzo (a) anthracene	94	97	84	116	100	113	100
Chrysene	183 H	163 H	266 VH	173 H	183 H	101	105
[Benzo (b) fluoranthene + Benzo (k) fluoranthene]	171* H	246* VH	188* H	167* H	194* H	162 H	189 H
						131	110
Benzo (a) pyrene	139	147	134	117	140	119	114
Indeno (123-cd) pyrene	104	152 H	112	104	161 H	111	142
Dibenzo (ah) anthracene	55	159 H	nd	84	nd	121	84
Benzo (ghi) perylene	61	100	73	68	90	79	91

* Percent recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	-	-	-	-	-	-	-

Table IV-27. % Recovery for Laboratory N107 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	----	----	----	----	----	-	-
Acenaphthylene	----	----	----	----	----	-	-
Acenaphthene	----	----	----	----	----	-	-
Fluorene	----	----	----	----	----	-	-
Phenanthrene	130	145	159	141	163	-	-
Anthracene	171	183	439	188	217	-	-
Fluoranthene	122	123	121	140	142	-	-
Pyrene	118	132	118	134	148	-	-
Benzo (a) anthracene	119	152	130	162	172	-	-
Chrysene	117	143	167	153	164	-	-
Benzo (b) fluoranthene	181	164	172	211	180	-	-
Benzo (k) fluoranthene	65	137	127	147	145	-	-
Benzo (a) pyrene	162	178	174	196	263	-	-
Indeno (123-cd) pyrene	105	120	114	155	143	-	-
Dibenzo (ah) anthracene	127	150	123	161	176	-	-
Benzo (ghi) perylene	119	133	132	175	151	-	-

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	99	109	96	98	108	91	100

Table IV-28. % Recovery for Laboratory N107 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	246 VH	210 VH	239 VH	300 VH	241 VH	-	-
Acenaphthylene	155 H	279 VH	158 H	290 VH	327 VH	-	-
Acenaphthene	213 VH	174 H	110	125	132	-	-
Fluorene	144	163 H	27 L	176 H	200 VH	-	-
Phenanthrene	163 H	151 H	140	159 H	170 H	-	-
Anthracene	173 H	159 H	225 VH	181 H	177 H	-	-
Fluoranthene	127	200 VH	168 H	197 H	190 H	-	-
Pyrene	168 H	190 H	195 H	201 VH	156 H	-	-
Benzo (a) anthracene	170 H	199 H	189 H	193 H	196 H	-	-
Chrysene	203 VH	232 VH	290 VH	245 VH	257 VH	-	-
Benzo (b) fluoranthene	192 H	310 VH	211 VH	187 H	223 VH	-	-
Benzo (k) fluoranthene	100	199 H	122	182 H	173 H	-	-
Benzo (a) pyrene	206 VH	207 VH	178 H	190 H	276 VH	-	-
Indeno (123-cd) pyrene	100	150 H	114	129	166 H	-	-
Dibenzo (ah) anthracene	126	142	114	149	145	-	-
Benzo (ghi) perylene	129	218 VH	156 H	161 H	202 VH	-	-

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	109	124	126	127	110	103	98

Table IV-29. % Recovery for Laboratory N158 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	74	85
Acenaphthylene	---	---	---	---	---	75	91
Acenaphthene	---	---	---	---	---	141	97
Fluorene	---	---	---	---	---	64	98
Phenanthrene	93	115	33	98	118	86	101
Anthracene	131	115	579	96	122	306	210
Fluoranthene	64	60	85	57	61	48	87
Pyrene	63	67	81	54	67	51	90
Benzo (a) anthracene	75	78	65	73	81	47	65
Chrysene	54	52	58	50	53	51	83
[Benzo (b) fluoranthene	80					73	109
+ Benzo (k) fluoranthene]	139	98*	142*	104*	100*	65	104
Benzo (a) pyrene	65	25	79	59	67	71	102
Indeno (123-cd) pyrene	117	92	108	97	87	95	133
Dibenzo (ah) anthracene	74	69	44	59	70	82	97
Benzo (ghi) perylene	83	61	84	71	64	96	132

* Percent recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	101	71	68	97	75	91	71

Table IV-30. % Recovery for Laboratory N158 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	98	83	140	135	119	94	96
Acenaphthylene	152 H	215 VH	138	224 VH	245 VH	87	118
Acenaphthene	103	94	1818 VH	86	98	111	106
Fluorene	113	115	229 VH	114	143	79	143
Phenanthrene	117	121	29 L	111	123	100	127
Anthracene	132	100	297 VH	92	100	320 VH	204 VH
Fluoranthene	67	98	118	81	82	69	133
Pyrene	89	96	135	80	71	59	132
Benzo (a) anthracene	106	103	95	87	92	94	149
Chrysene	93	84	100	79	83	67	151 H
[Benzo (b) fluoranthene	85					100	231 VH
+ Benzo (k) fluoranthene]	214 VH	169* H	160* H	103*	122*	104	190 H
Benzo (a) pyrene	82	28 L	80	57	70	81	152 H
Indeno (123-cd) pyrene	112	115	108	81	101	106	251 VH
Dibenzo (ah) anthracene	74	65	41 L	55	58	107	116
Benzo (ghi) perylene	90	100	100	65	86	93	203 VH

* Percent recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	112	81	90	126	77	104	69

Table IV-31. % Recovery for Laboratory N196 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8B	9B
Naphthalene	---	---	---	---	---	274	99
Acenaphthylene	---	---	---	---	---	514	118
Acenaphthene	---	---	---	---	---	3249	80
Fluorene	---	---	---	---	---	108	74
Phenanthrene	0.7	0.8	-	-	-	99	156
Anthracene	2	nd	-	-	-	413	339
Fluoranthene	2	5	-	-	-	88	305
Pyrene	1	2	-	-	-	88	207
Benzo (a) anthracene	nd	nd	-	-	-	59	364
Chrysene	3	5	-	-	-	97	183
Benzo (b) fluoranthene	2	1	-	-	-	124	245
Benzo (k) fluoranthene	0.4	nd	-	-	-	96	385
Benzo (a) pyrene	4	5	-	-	-	101	260
Indeno (123-cd) pyrene	0.5	0.5	-	-	-	113	783
Dibenzo (ah) anthracene	nd	nd	-	-	-	227	1368
Benzo (ghi) perylene	2	2	-	-	-	105	218

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	6	3	-	-	-	86	81

Table IV-32. % Recovery for Laboratory N196 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8B	9B
Naphthalene	7 VL	2 VL	-	-	-	197 H	100
Acenaphthylene	nd	nd	-	-	-	180 H	42 L
Acenaphthene	36 L	nd	-	-	-	137	100
Fluorene	4 VL	37 L	-	-	-	111	92
Phenanthrene	0.9 VL	0.8 VL	-	-	-	99	156 H
Anthracene	2 VL	nd	-	-	-	170 H	165 H
Fluoranthene	3 VL	8 VL	-	-	-	88	343 VH
Pyrene	2 VL	3 VL	-	-	-	87	214 VH
Benzo (a) anthracene	nd	nd	-	-	-	100	625 VH
Chrysene	5 VL	8 VL	-	-	-	116	219 VH
Benzo (b) fluoranthene	2 VL	3 VL	-	-	-	98	140
Benzo (k) fluoranthene	0.7 VL	nd	-	-	-	117	467 VH
Benzo (a) pyrene	6 VL	6 VL	-	-	-	100	257 VH
Indeno (123-cd) pyrene	0.5 VL	0.6 VL	-	-	-	111	179 H
Dibenzo (ah) anthracene	nd	nd	-	-	-	100	100
Benzo (ghi) perylene	2 VL	4 VL	-	-	-	92	127

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	6 VL	4 VL	-	-	-	97	80

Table IV-33. % Recovery for Laboratory N197 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8B	9B
Naphthalene	---	---	---	---	---	4	nd
Acenaphthylene	---	---	---	---	---	36	nd
Acenaphthene	---	---	---	---	---	1481	nd
Fluorene	---	---	---	---	---	87	87
Phenanthrene	16	nd	10	5	7	100	94
Anthracene	18	nd	9	nd	nd	71	73
Fluoranthene	63	24	26	43	26	100	89
Pyrene	42	6	37	57	43	102	97
Benzo (a) anthracene	36	46	97	68	71	60	58
Chrysene	34	14	37	57	26	83	83
Benzo (b) fluoranthene	96	36	41	127	38	142	175
Benzo (k) fluoranthene	49	20	28	73	20	82	82
Benzo (a) pyrene	63	25	35	82	22	101	101
Indeno (123-cd) pyrene	173	69	73	256	38	89	92
Dibenzo (ah) anthracene	1910	504	787	2252	168	nd	nd
Benzo (ghi) perylene	183	47	90	266	18	122	124

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	67	21	2377	61	961	35	nd

Table IV-34. % Recovery for Laboratory N197 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8B	9B
Naphthalene	189 H	nd	nd	111	nd	3 VL	nd
Acenaphthylene	nd	nd	nd	nd	nd	13 VL	nd
Acenaphthene	435 VH	294 VH	147	634 VH	nd	63	nd
Fluorene	nd	100	nd	nd	nd	89	108
Phenanthrene	20 VL	nd	9 VL	6 VL	7 VL	100	94
Anthracene	18 VL	nd	5 VL	nd	nd	29 L	36 L
Fluoranthene	65	39 L	36 L	60	35 L	100	100
Pyrene	60	9 VL	62	85	45 L	100	100
Benzo (a) anthracene	51	61	140	81	81	101	100
Chrysene	58	22 VL	64	91	41 L	100	100
Benzo (b) fluoranthene	101	69	50 L	112	47 L	112	100
Benzo (k) fluoranthene	75	29 L	27 L	90	23 VL	100	100
Benzo (a) pyrene	80	28 L	36 L	80	23 VL	100	100
Indeno (123-cd) pyrene	165 H	85	72	213 VH	43 L	88	21 VL
Dibenzo (ah) anthracene	1900 VH	476 VH	731 VH	2083 VH	138	nd	nd
Benzo (ghi) perylene	198 H	77	107	244 VH	24 VL	107	72

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	74	23 VL	3118 VH	79	981 VH	39 L	nd

Table IV-35. % Recovery for Laboratory N200 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8B	9B
Naphthalene	---	---	---	---	---	nd	nd
Acenaphthylene	---	---	---	---	---	285	446
Acenaphthene	---	---	---	---	---	nd	nd
Fluorene	---	---	---	---	---	nd	nd
Phenanthrene	nd	nd	nd	nd	nd	115	100
Anthracene	nd	nd	nd	215	nd	nd	nd
Fluoranthene	102	nd	58	125	91	102	82
Pyrene	nd	nd	38	nd	nd	166	71
Benzo (a) anthracene	59	66	54	55	50	49	42
Chrysene	28	66	55	61	55	49	26
Benzo (b) fluoranthene	nd	nd	nd	nd	nd	127	53
Benzo (k) fluoranthene	57	151	104	131	121	79	55
Benzo (a) pyrene	102	115	98	92	91	84	62
[Indeno (123-cd) pyrene	nd*	nd*	nd*	nd*	nd*	nd	nd
+ {Dibenzo (ah) anthracene}	nd	nd	nd	nd	nd	85*	75*
+ Benzo (ghi) perylene}	nd	nd	nd	nd	nd		

* Percent recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	36	51	48	66	71	90	103

Table IV-36. % Recovery for Laboratory N200 Calculated from the Interlab Median.

PAHs	Sample #					Ampule #		
	1	2	3	4	5	8B	9B	
Naphthalene	nd	nd	nd	nd	nd	nd	nd	
Acenaphthylene	nd	nd	nd	nd	nd	100	158	H
Acenaphthene	nd	nd	nd	nd	nd	nd	nd	
Fluorene	nd	nd	nd	nd	nd	nd	nd	
Phenanthrene	nd	nd	nd	nd	nd	115	100	
Anthracene	nd	nd	nd	207 VH	nd	nd	nd	
Fluoranthene	106	nd	81	176 H	122	103	92	
Pyrene	nd	nd	63	nd	nd	163	74	H
Benzo (a) anthracene	85	87	78	65	56	84	71	
Chrysene	49 L	107	95	98	86	59	31	L
Benzo (b) fluoranthene	nd	nd	nd	nd	nd	100	30	L
Benzo (k) fluoranthene	88	220 VH	100	162 H	144	96	67	
Benzo (a) pyrene	130	134	100	89	95	83	61	
[Indeno (123-cd) pyrene	nd*	nd*	nd*	nd*	nd*	nd	nd	
+ {Dibenzo (ah) anthracene}	nd	nd	nd	nd	nd	62*	19*	VL
+ Benzo (ghi) perylene)								

* Percent recovery based on a total value reported for these parameters

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	40 L	58	62	86	72	103	102

Table IV-37. % Recovery for Laboratory N208 Calculated from the Reference Value.

PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	---	---	---	---	---	93	93
Acenaphthylene	---	---	---	---	---	51	49
Acenaphthene	---	---	---	---	---	81	72
Fluorene	---	---	---	---	---	77	70
Phenanthrene	44	25	29	45	51	56	60
Anthracene	30	10	34	33	36	44	41
Fluoranthene	61	28	28	55	48	54	42
Pyrene	59	28	28	53	49	55	45
Benzo (a) anthracene	84	37	41	82	74	34	23
Chrysene	59	33	35	63	53	52	34
Benzo (b) fluoranthene	111	52	60	122	90	41	37
Benzo (k) fluoranthene	65	40	51	88	85	51	30
Benzo (a) pyrene	62	17	35	103	91	51	33
Indeno (123-cd) pyrene	101	60	54	114	85	50	29
Dibenzo (ah) anthracene	101	63	55	108	92	44	51
Benzo (ghi) perylene	92	53	51	109	75	56	34

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	54	32	30	61	36	65	88

Table IV-38. % Recovery for Laboratory N208 Calculated from the Interlab Median.

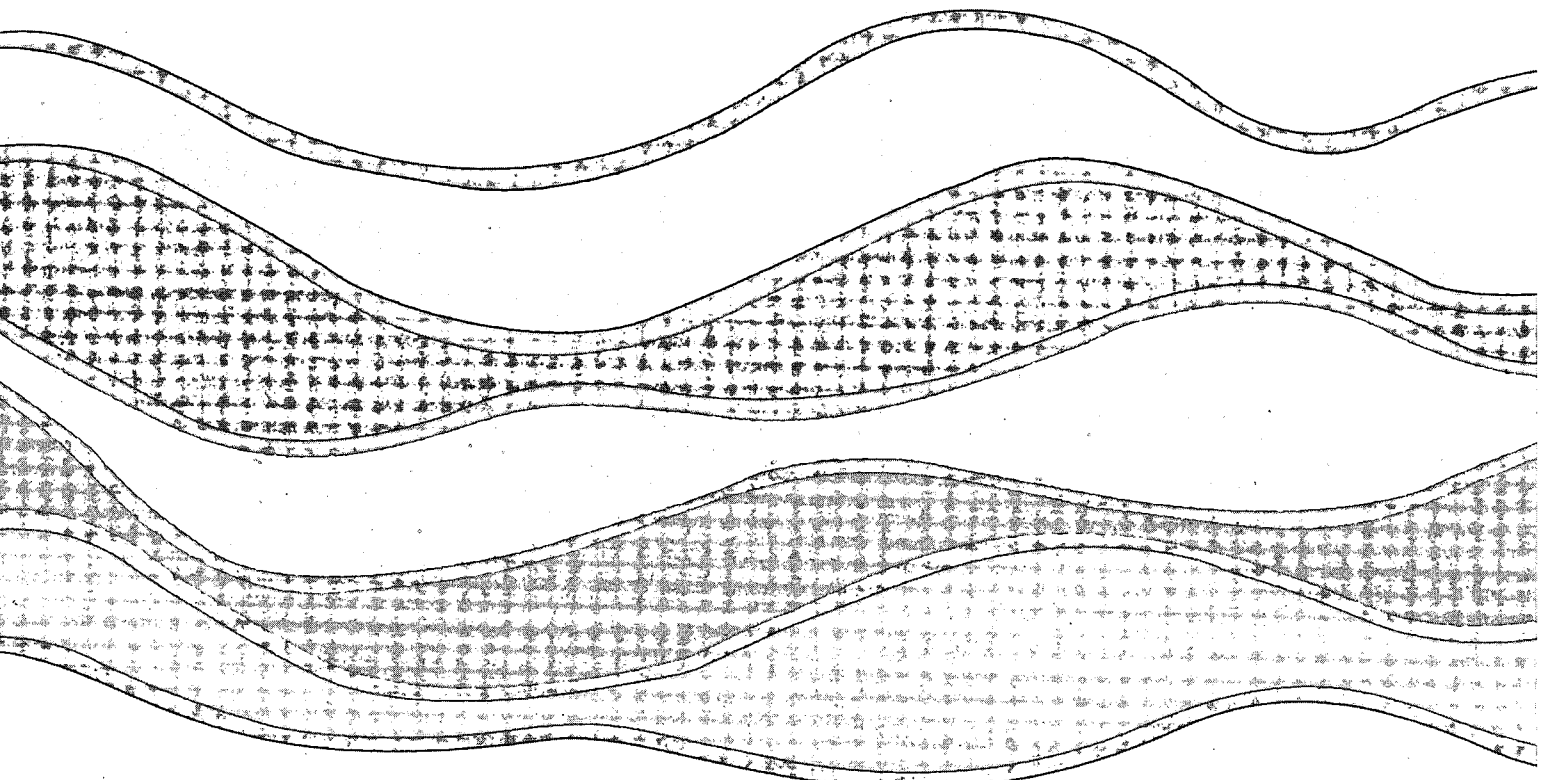
PAHs	Sample #					Ampule #	
	1	2	3	4	5	8A	9A
Naphthalene	46 L	4 VL	16 VL	9 VL	46 L	117	104
Acenaphthylene	18 VL	12 VL	6 VL	20 VL	27 L	58	63
Acenaphthene	22 VL	9 VL	6 VL	17 VL	26 L	64	79
Fluorene	38 L	17 VL	2 VL	40 L	50 L	94	102
Phenanthrene	55	26 L	26 L	51	53	66	75
Anthracene	31 L	9 VL	17 VL	31 L	30 L	46 L	40 L
Fluoranthene	63	45 L	40 L	78	65	78	64
Pyrene	84	40 L	46 L	80	52	63	66
Benzo (a) anthracene	120	49 L	59	97	85	69	52
Chrysene	102	53	61	100	83	68	62
Benzo (b) fluoranthene	118	98	74	108	111	57	78
Benzo (k) fluoranthene	100	59	50 L	109	101	82	55
Benzo (a) pyrene	79	20 VL	36 L	100	95	59	49
Indeno (123-cd) pyrene	96	75	54	95	99	56	54
Dibenzo (ah) anthracene	100	60	51	100	76	58	60
Benzo (ghi) perylene	99	87	61	100	100	54	53

PCBs	Sample #					Ampule #	
	1	2	3	4	5	6	7
Total PCBs	60	36 L	39 L	79	37 L	74	87

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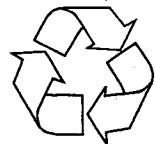


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