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March 30, 1979

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Attention: Dr. R. W. MacDonald

Re: D.S.S. File #07SB.KF833-8-0592  
Title: Development of Meaningful Criteria  
for Ocean Disposal of Dredged or Sedimentary Material

We are pleased to forward our complete report for the above mentioned project.

The results are presented in concise tables on pages 11 - 23 of this report.

I trust you will find our report in good order and if you have any further questions, please don't hesitate to contact the undersigned or Mr. R. Deverall who was the principal analyst for this project.

Yours truly,

CAN TEST LTD.

A. W. Maynard, M.Sc.,  
Assistant General Manager.

AWM/al



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DEVELOPMENT OF MEANINGFUL CRITERIA  
FOR OCEAN DISPOSAL OF DREDGED OR  
SEDIMENTARY MATERIAL

SUMMARY

A series of chemical and physical analyses were carried out to assist the Scientific Authority (Institute of Ocean Sciences) in developing meaningful criteria for ocean disposal of dredged or sedimentary material. Two sediment cores, one from Victoria Harbour and one from False Creek were submitted for these analyses.

This report contains the detailed methodology utilized, with results presented in concise summary tables. The discussion is limited to the suitability of the analysis scheme, as per the terms of reference of our contract.

DEVELOPMENT OF MEANINGFUL CRITERIA  
FOR OCEAN DISPOSAL OF DREDGED OR  
SEDIMENTARY MATERIAL

## 1.0 INTRODUCTION

Can Test Ltd. received a contract on October 2, 1978 from the Institute of Ocean Sciences, to conduct a series of chemical analysis pertinent to the development of meaningful criteria for the ocean disposal of dredged or sedimentary material. The analyses were carried out to assist the Scientific Authority (Institute of Ocean Sciences) to assess how various elements are associated with sediments.

For this study, two sediment cores, one from Victoria Harbour and one from False Creek, were collected by the Scientific Authority, and submitted to Can Test. The sediments were analysed for a number of physical and chemical parameters and then carried through a series of extractions to determine elemental partitioning. Aliquots of the sediments were submitted to Seachem Ltd. for release experiments.

This report contains primarily the detailed methodology used for the analysis, and the results obtained. Our discussion is limited to the suitability of the analytical methodology since it was not within the Terms of Reference of our contract to provide a detailed interpretation of the test results.

## 2.0 APPROACH

Two core samples (one from Victoria Harbour and one from False Creek) were collected by the Scientific Authority and delivered to Can Test Ltd. Each core was split longitudinally under a nitrogen atmosphere. These 2 portions of each core were then subjected to the following analysis.

- 2.1 From one longitudinal half, the top 15 cm and bottom 15 cm were treated to remove the interstitial water. The interstitial water was then analysed for metals and nutrients.
- 2.2 The second longitudinal half was homogenized in a nitrogen atmosphere. An aliquot was removed for a series of chemical and physical tests to characterize the sediment.
- 2.3 An additional aliquot from the second half was removed to carry out a 4:1 elutriate test.
- 2.4 A third aliquot was removed and submitted to Seachem Ltd. for a "Release Experiment".
- 2.5 A fourth aliquot was removed to carry out a series of extractions to determine elemental partitioning.
- 2.6 This extraction series was repeated on the sediments after the release experiment (#2.4).

This analytical scheme is presented as Figure 1 (next page).

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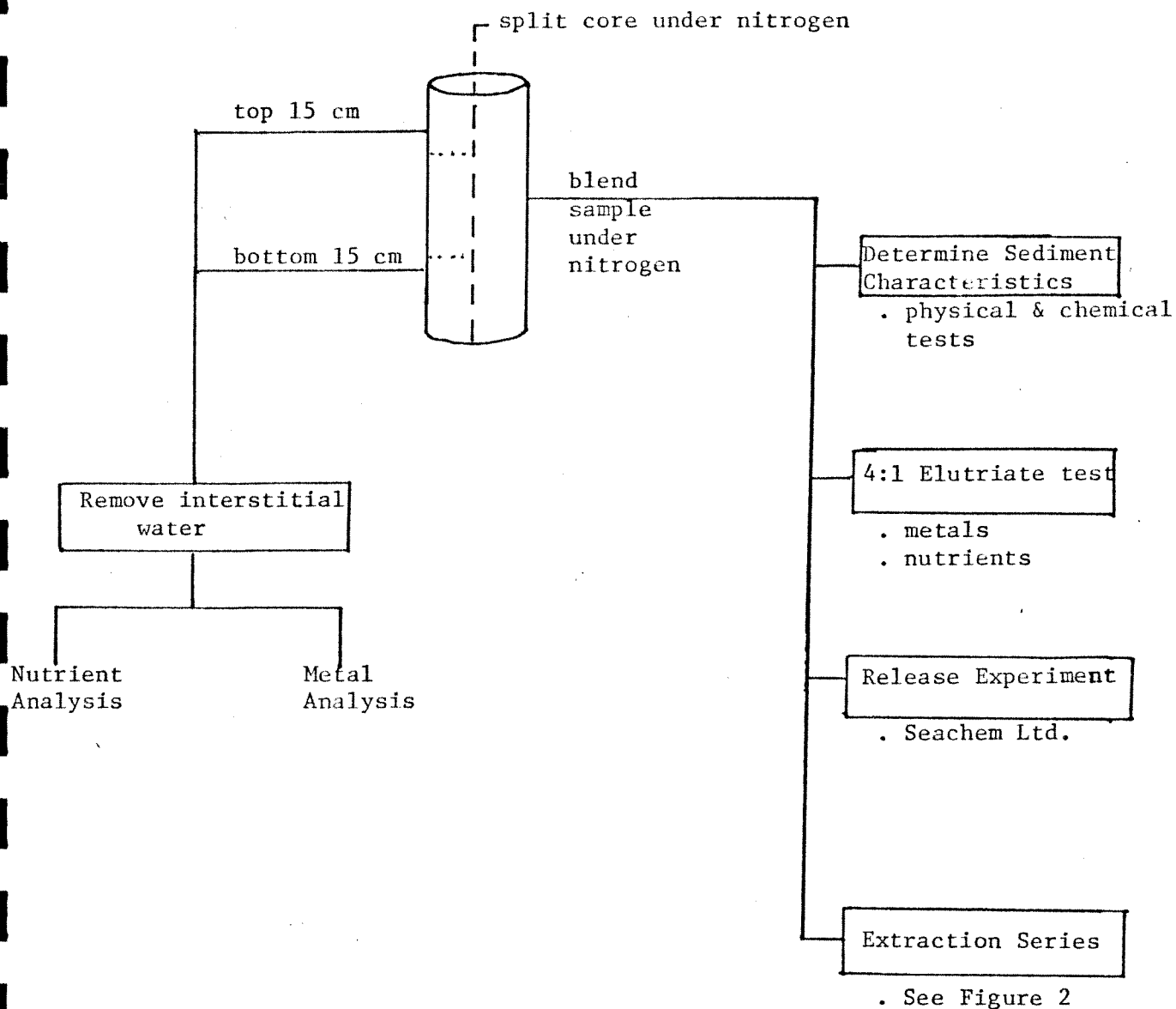


Figure 1 Schematic showing analysis procedures carried out on each of two core samples

### 3.0 METHODOLOGY

The entire procedure of sample preparation was carried out in a glove box under an atmosphere of nitrogen. Aliquots required for various analysis steps were stored in plastic containers, also in a nitrogen atmosphere.

#### 3.1 Interstitial Water

In a glove box under an atmosphere of nitrogen, each core was split longitudinally and the top 15 cm and bottom 15 cm were placed in centrifuge tubes. The centrifuge tubes were capped tightly, to prevent aeration, and centrifuged at 1000 G to separate the interstitial water. The water, being slightly turbid, was then filtered on a 0.45 micron filter. This filtration was carried out in the nitrogen atmosphere glove box.

The four interstitial water samples thus produced were analysed for nutrients and metals using procedures described in Standard Methods (1975) and/or Strickland and Parsons (1968). The nutrient methods used were:

Phosphate	-	Ascorbic Acid Method
Nitrate	-	Cadmium Reduction Method
Nitrite	-	Diazotization
Ammonium	-	Distillation/Nesslerization

The waters were also analysed for the metals Cadmium, Copper, Lead, Iron and Copper using an MLBK/APDC extraction followed by flame atomic absorption spectrophotometry (Perkin Elmer 603). Manganese was determined by an MLBK/DDC extraction followed by direct flame atomic absorption spectrophotometry (Perkin Elmer 603). Arsenic was determined using a MHS-1 (hydride generator) coupled to a Perkin Elmer 603 atomic absorption spectrophotometer. Mercury was determined using a Pharmacia Mercury Monitor (cold vapor atomic absorption spectrophotometer).

#### 3.2 Sediment Characteristics

The second longitudinal half of each core was blended (in nitrogen-glove box) and an aliquot was taken for a series of chemical and physical tests. The tests carried out and a brief description of the procedures used were:

### 3.2 Sediment Characteristics (continued)

- a) Moisture - dried 103°C to a constant weight.
- b) Loss on Ignition - muffle furnace, 450°C.
- c) pH - sediment and distilled water mixed 1:1 by volume and the pH of the slurry determined.
- d) Eh - platinum electrode with saturated calomel reference electrode, inserted into core to a depth of 6 cm (Waldon 1978)
- e) Free sulfide - aliquot of sediment was diluted with 0.1N NaOH, solution filtered on 0.45 u membrane and analysed for sulfide using the methylene blue colorimetric procedure (Standard Methods, 1975).
- f) Total sulfide - aliquot of sediment was acid distilled under nitrogen, distillate was analysed for sulfide using the titrimetric (iodine) method.
- g) Total organic carbon - Leco Induction Furnace
- h) Cation Exchange Capacity - Ammonium saturation method (Chapman, 1965).
- i) Particle Size Distribution - Seiving, hydrometer method (Waldon 1978)
- j) Metals - See Section 3.4

### 3.3 Elutriate Test

A 4:1 elutriate test was carried out as described in Keely and Engler (1974). The sea water used for this experiment was the same as that used for the release experiment. The sea water sample was collected by Seachem Ltd. from the main wharf of the Institute of Ocean Sciences.

The elutriates were analysed for metals and nutrients as discussed in Section 3.1.

### 3.4 Selective Extraction Procedure (See Figure 2) (page 23)

The steps described below were carried out in triplicate for each sediment.

- a) Extraction 1: An aliquot of the second longitudinal half was carried through the selective extraction procedure described in the initial Request for Proposal. Specifically, 50 grams (wet weight) was placed in a 200 ml erlenmeyer flask and 100 ml of deoxygenated 1N ammonium acetate adjusted to the surface pH of the sediment, was added. The mixture was stirred for 1 hour and filtered (0.45 u membrane) and the filtrate collected as EXTRACT 1. All of the above steps to produce EXTRACT 1 were carried out in a glove box under nitrogen atmosphere.
- b) Extraction 2: The remaining sediment from extraction 1 was washed with O<sub>2</sub> free distilled deionized water and the liquid phase was discarded. A 4 g sub-sample was removed and mixed with 100 ml of 0.1M. hydroxylamine hydrochloride in 0.01 M hydrochloric acid. At this stage the oxygen free environment was not required.

The mixture was shaken for 30 minutes and filtered to produce EXTRACT 2.

### 3.4 Selectrive Extraction Procedure (See Figure 2) continued)

- c) Extraction 3: The residue from extraction 2 was washed and the fluid phase was discarded. The washed residue was then digested at 95°C with 30% hydrogen peroxide (acidified to a pH of 2.5 with dilute HNO<sub>3</sub>). After digestion the residue was mixed with 1N ammonium acetate (pH 2.5) for 30 minutes and the slurry was filtered on a 0.45 micron filter. The filtrate was collected as EXTRACT 3.
- d) Extraction 4: The residue was washed, and then extracted with 100 ml of sodium citrate/sodium dithionite (16g/1.67g per 100 ml). This mixture was shaken for 17 hours using a mechanical shaker. The slurry was filtered on a 0.45 micron filter and the filtrate was collected as EXTRACT 4. The residue was washed and the liquid phase was again discarded. The residue was dried, weighted and held for chemical analysis of metals, see 3.4 (f) below.
- e) Analysis of extracts: The extracts were analysed by direct flame atomic absorption spectrophotometry (Perkin Elmer Model 603) with background correction using standards and blanks prepared in the same matrix as the extracts. If an improvement in detection limit was required, a graphite furnace attachment was used. Arsenic and mercury were analysed by flameless atomic absorption spectrophotometry (see section 3.1).
- f) Analysis of sediments before and after extraction: An aliquot of the sediment used for the selective extraction experiments and the residue remaining after extraction 4 were analysed for metals. A one gram (dry) sample was digested with hot aqua regia and the digest analysed for the same metals determined in extracts 1, 2, 3 and 4. The analyses were carried out using atomic absorption spectrophotometry and the results expressed as ug/dry gram of sediment.

### 3.5 Extraction Series - After Release Experiment

The extraction series and analysis described above (3.4 a-f) was repeated on the sediments remaining after the release experiments. The release experiments were carried out in duplicate (by Seachem Ltd.) for each sediment sample. Thus, 4 sediment samples were submitted to Can Test, immediately upon completion of these experiments.

The extraction experiments differed from the previous set with regard to the following:

- a) The samples were small and 50 g (wet) was not available for extraction 1. Therefore, a 10 g (wet) sample was carried through the entire extraction series. The moisture of the starting sediment was determined and the wet weight of sample available for each extraction was also measured.
- b) The extracts were filtered on a Whatman 40 filter followed by a 0.45 micron filter. This shortened the filtration time.
- c) Since the samples had been exposed to oxygenated sea water in the release experiment, none of the steps were carried out in the oxygen free glove box.

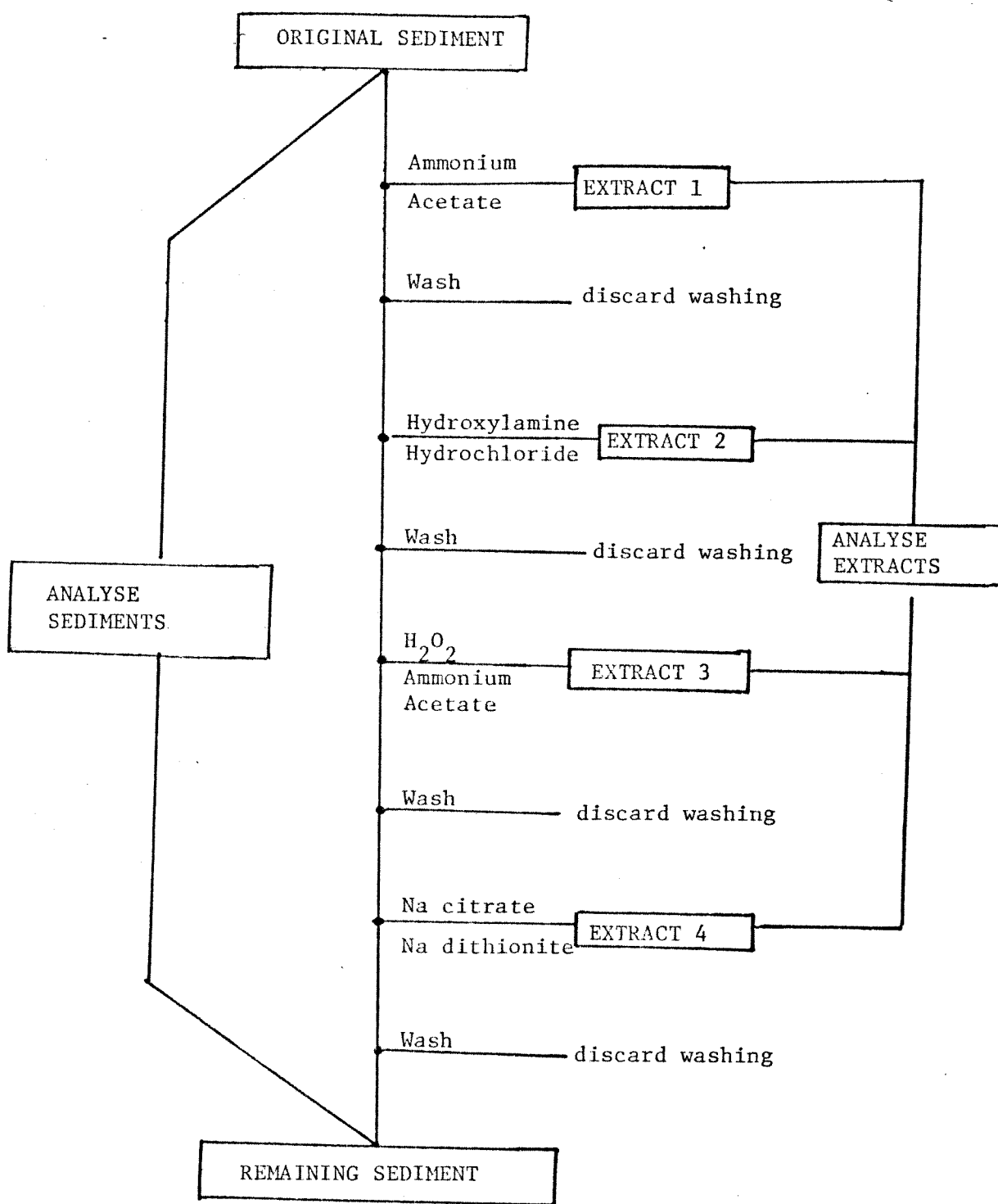


Figure 2 Showing extraction series. This was carried out in triplicate on the original sediment. It was also repeated (in duplicate) on sediment from the release experiment.

## 4.0 RESULTS

### 4.1 Interstitial Water

The interstitial water, removed from the top 15 cm and bottom 15 cm of a longitudinal half of each core, was analysed for metals and nutrients. The results are given in Table 1.

### 4.2 Sediment Characteristics

The results of the chemical and physical analysis of the longitudinal half of each core used in the extraction experiments, are given in Table 2. The metal concentrations of these samples however, are presented in Table 4-c. The sediment analyses carried out on the residues returned after the release experiments are presented in Tables 5-c.

### 4.3 Elutriate Tests

The results of duplicate 4:1 elutriate tests carried out on each sediment are presented in Table 3. The nitrate results obtained were not reported. The initial seawater submitted to us contained a nitrate concentration of 16. mg/L. For this reason we suspected contamination of the collection bottle. The sample was not acidic however, so the presence of nitric acid was not indicated.

### 4.4 Extraction Series

The results of the triplicate extraction experiments are presented in Table 4-a for the Victoria Harbour sample and in Table 4-b for the False Creek sample. The results are given in terms of total microgram of each element in the various extracts. These were calculated simply by multiplying the concentrations (in ug/ml) by 100 ml (i.e. by the total volume of each extract).

It was convenient to present the figures in this manner, to allow comparisons with the results of analysis of sediment fractions before and after the extraction experiments. This data is presented in Table 4-c and 4-d for Victoria Harbour and False Creek respectively. These tables show the concentrations of the sediment, determined in triplicate, before and after extraction (column 1 and 4).

#### 4.4 Extraction Series (continued)

The weight (dry) of sample available for extraction 2 is shown in column 2 of these tables. Here a 50% moisture was assumed on the 4 gram sub-sample used in extraction 2. Column 5 shows the dry weight of residue remaining after the last extraction and the difference between the volume in column 2 (always 2 grams) and the value in column 5, represents the weight of sediment lost during the extraction - either by transfer losses or by dissolution. Column 3 of Tables 4-c and 4-d represent the total weight of the various elements (in micrograms) available for extraction 2 (multiply column 1 by column 2). Column 6 represents the weight (in microgram) remaining after the extraction. The difference between these (column 7) represents the amount of metal (in microgram) lost during the extraction (assuming negligible losses during extraction 1). These amounts should in theory, be equal to, or greater than, the "Sum" figures in Tables 4-a and 4-b.

It was not possible to calculate the results from the extractions in terms of micrograms per gram since the total weight of sample (on a dry basis) used for each extraction, was not known. In order to have obtained such information, a much larger sample should have been carried through extractions 2, 3, and 4 than that specified in the "Request for Proposal" to allow removal of aliquots for moisture determination. The known weights available for the extractions are shown in Table 4-c.

#### 4.5 Extraction Series of Sediments Remaining from Release Experiment

The extraction experiments were repeated using the sediments remaining after the release experiments conducted by Seachem. The release experiments were conducted in duplicate leaving 4 sediments for the extraction experiments. The results are presented in the same format as the original extraction series and are given in Tables 5-a and 5-b for Victoria Harbour and False Creek respectively.

It should be noted however, in viewing these results, that for extraction 1 less sample was available (i.e. in the original extraction series, 50 g wet was taken for extraction 1 whereas in the second extraction series, 10 g wet was taken for extraction 1). For the remaining extractions (2, 3 and 4) more sample was available than for the original extraction series. In the original series a 4 g (wet) sub-sample was taken after extraction 1 whereas in the second series the 10 g sample was taken through all 4 extractions.

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#### 4.5 Extraction Series of Sediments Remaining from Release Experiment (continued)

The results of the sediment analysis are presented in Tables 5-c and 5-d.

In this set of experiments, more information was obtained concerning the weight loss (on a wet basis) after each extraction. These figures are shown in Table 5-c. It was still not possible to determine these results on a dry basis due to the small sample size.

TABLE 1 INTERSTITIAL WATER

Parameter		Victoria Harbour		False Creek	
(all in mg/L)		Top 15 cm	Bottom 15 cm	Top 15 cm	Bottom 15 cm
<u>Metals</u>					
Arsenic	As	0.010	0.009	L 0.005	L 0.005
Cadmium	Cd	L 0.001	L 0.001	L 0.001	L 0.001
Copper	Cu	L 0.001	L 0.001	0.002	0.002
Iron	Fe	0.076	0.063	0.084	0.046
Mercury	Hg	L 0.0005	L 0.0005	L 0.0005	L 0.0005
Manganese	Mn	0.021	0.024	0.020	0.022
Lead	Pb	L 0.001	L 0.001	L 0.001	L 0.001
Zinc	Zn	L 0.001	L 0.001	0.009	0.004
<u>Nutrients</u>					
Phosphate	T-PO <sub>4</sub>	2.94	12.6	4.46	9.18
Nitrate	N	1.19	5.92	3.09	0.51
Nitrite	N	0.034	0.028	0.007	0.028
Ammonia	N	4.0	3.8	5.3	11.0

L = less than

Iw 70 ppb

Iw = 265 ppb

E.T. 45 ppb

610

N.B. 37 ppb

77

TABLE 2 SEDIMENT CHARACTERISTICS

Parameter	Core 1 - Victoria Harbour	Core 2 - False Creek
Total Core Length (cm)	33.5	75.0
Moisture (%)	63.3	49.2
Loss on Ignition (%)	10.0	7.5
pH @ Surface	7.4	7.7
* Eh @ 6 cm (mV)	-91.	-100.
Total Sulfide S (ug/g)	430.	230.
Free Sulfide S (ug/g)	10.	13.
Total Organic Carbon (%)	7.50	2.00
Cation Exchange Capacity (m.eq./100g)	32.	13.
Particle Size Distribution		
% Gravel (> 2mm)	1.1	1.4
% Sand (≤ 2mm, > 63u)	25.7	19.5
% Silt (≤ 63u, > 4u)	43.3	42.8
% Clay (< 4u)	29.9	36.3
Approximate % by volume of wood in:		
"Gravel" Fraction	100.	80.
"Sand" Fraction	80.	50.
Metals ---	See Tables 4c and 4d	
Characteristics after Release Exp.	See Tables 5c and 5d	

\* Analysed prior to splitting the core.

TABLE 3 ELUTRIATE TEST

Parameter (all mg/L)		Receiving Water	Victoria Harbour		False Creek	
			Test 1	Test 2	Test 1	Test 2
<u>Metals</u>						
Arsenic	As	0.0006	0.001	0.001	0.0006	0.0006
Cadmium	Cd	L 0.001	L 0.001	L 0.001	L 0.001	L 0.001
Copper	Cu	0.003	0.008	0.002	0.008	0.007
Iron	Fe	0.006	0.080	0.110	0.61	0.61
Mercury	Hg	L 0.0002	L 0.0002	L 0.0002	L 0.0002	L 0.0002
Manganese	Mn	0.005	0.055	0.052	0.040	0.038
Lead	Pb	L 0.001	L 0.001	L 0.001	L 0.001	L 0.001
Zinc	Zn	L 0.001	0.050	0.040	0.009	0.009
<u>Nutrients</u>						
Phosphate	O-PO <sub>4</sub>	0.43	0.18	0.18	0.12	0.12
Nitrate	N*	-	-	-	-	-
Nitrite	N	0.007	0.060	0.040	0.37	0.38
Ammonia	N	L 0.10	1.5	1.5	1.4	1.5

\* Nitrate not reported - initial receiving water as submitted to this laboratory was extremely high in nitrate and contamination was suspected. (see remarks in report - Section 4.3)

L = Less than

TABLE 4 - a - Extraction of Victoria Harbour Sediment

Metal	Extraction No.				Sum
	1	2	3	4	
Arsenic As	1.0	L 0.5	5.2	L 0.50	6.2
	1.0	L 0.5	5.6	L 0.50	6.6
	1.0	L 0.5	5.5	L 0.50	6.5
Cadmium Cd	L 0.1	L 0.1	2.1	L 1.0	2.1
	L 0.1	L 0.1	2.2	L 1.0	2.2
	L 0.1	L 0.1	3.1	L 1.0	3.1
Copper Cu	0.3	L 0.1	199.	5.0	204.
	0.2	L 0.1	191.	4.0	195.
	0.3	L 0.1	200.	4.0	204.
Iron Fe	85.0	1180.	18,500.	3500.	23,200.
	100.	1270.	17,500.	3200.	22,000.
	89.	1260.	16,800.	3500.	21,600.
Mercury Hg	L 0.05	L 0.05	L 0.05	-	L 0.05
	L 0.05	L 0.05	L 0.05	-	L 0.05
	L 0.05	L 0.05	L 0.05	-	L 0.05
Manganese Mn	5.0	5.0	106.	6.0	122.
	L 5.0	4.0	109.	6.0	119.
	L 5.0	6.0	124.	7.0	137.
Lead Pb	15.0	0.10	247.	L 10.	262.
	15.0	0.20	248.	L 10.	263.
	13.0	L 0.10	246.	L 10.	259.
Zinc Zn	L 1.0	4.0	307.	L 5.0	311.
	L 1.0	3.0	293.	L 5.0	296.
	L 1.0	3.0	300.	L 5.0	303.

All results expressed as micrograms.

Extraction 1 - Ammonium Acetate

2 - Hydroxylamine Hydrochloride

3 - H<sub>2</sub>O<sub>2</sub> Digestion/Ammonium Acetate

4 - Na-citrate/Na dithionite.

Sum - represents total ug of each element found in the extracts.

L = Less than

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 FALSE  
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TABLE 4 - b - Extraction of False Creek Sediment

Metal	Extraction No.				Sum
	1	2	3	4	
Arsenic As	L 0.5	L 0.5	3.7	L 0.5	3.7
	L 0.5	L 0.5	3.9	L 0.5	3.9
	L 0.5	L 0.5	3.5	L 0.5	3.5
Cadmium Cd	L 0.1	L 0.1	3.1	L 1.0	3.1
	L 0.1	L 0.1	2.8	L 1.0	2.8
	L 0.1	L 0.1	2.9	L 1.0	2.9
Copper Cu	0.4	L 0.1	131.	2.0	133.
	0.2	L 0.1	132.	1.0	133.
	0.3	L 0.1	137.	1.0	138.
Iron Fe	28.	955.	19,000.	8300.	28,300.
	32.	1060.	18,200.	7900.	27,200.
	26.	1030.	19,700.	7400.	28,100.
Mercury Hg	L 0.05	L 0.05	L 0.05	-	L 0.05
	L 0.05	L 0.05	L 0.05	-	L 0.05
	L 0.05	L 0.05	L 0.05	-	L 0.05
Manganese Mn	8.0	12.	135.	19.	174.
	6.0	13.	135.	21.	175.
	8.0	12.	141.	20.	181.
Lead Pb	L 10.	L 0.1	70.	L 10.	70.
	L 10.	L 0.1	80.	L 10.	80.
	L 10.	L 0.1	79.	L 10.	79.
Zinc Zn	2.0	L 10.	358.	L 5.	360.
	1.0	10.	354.	L 5.	365.
	2.0	L 10.	362.	L 5.	364.

All results expressed as micrograms.

Extraction 1 - Ammonium Acetate

2 - Hydroxylamine Hydrochloride

3 - H<sub>2</sub>O<sub>2</sub> Digestion/Ammonium Acetate

4 - Na<sub>2</sub>-Citrate/Na dithionite.

Sum - represents total ug of each element found in the extracts.

L = Less than

TABLE 4 - c - Analysis of Victoria Harbour Sediment Before and After Extraction

Element	Before Extraction			After Extraction			
	1) Conc. ug/g	2) Sample Weight(g)	3) Total Metal(ug)	4) Conc. ug/gm	5) Sample Weight(g)	6) Total Metal(ug)	7) Loss of Extr(ug)
Arsenic As	6.0	2.*	12.0	0.8	1.1	0.88	11.1
	6.3	2.	12.6	0.8	1.2	0.96	11.6
	6.5	2.	13.0	0.9	1.0	0.90	12.1
Cadmium Cd	1.1	2.	2.2	L 1.0	1.1	L 1.10	G.1.1
	1.2	2.	2.4	L 1.0	1.2	L 1.20	G.1.2
	1.1	2.	2.2	L 1.0	1.0	L 1.00	G.1.2
Copper Cu	195.	2.	390.	38	1.1	41.8	348.
	194.	2.	388.	39	1.2	46.8	341.
	197.	2.	394.	42	1.0	42.0	352.
Iron Fe	39,100.	2.	78,200.	16,000.	1.1	17,600.	60,600.
	38,600.	2.	77,200.	16,500.	1.2	19,800.	57,400.
	38,800.	2.	77,600.	16,100	1.0	16,100	61,500.
Mercury Hg	3.1	2.	6.20	2.9	1.1	3.20	3.0
	3.2	2.	6.41	2.9	1.2	3.51	2.9
	2.9	2.	5.80	2.8	1.0	2.81	3.0
Manganese Mn	246.	2.	492.	220.	1.1	242.	250.
	259.	2.	518.	233.	1.2	280.	238.
	250.	2.	500.	228.	1.0	228.	272.
Lead Pb	241.	2.	482.	33.	1.1	36.3	446.
	243.	2.	486.	28.	1.2	33.6	452.
	241.	2.	482.	33.	1.0	33.0	449.
Zinc Zn	270.	2.	540.	84.	1.1	92.4	448.
	276.	2.	552.	91.	1.2	109.	443.
	289.	2.	578.	89.	1.0	89.1	489.

Key      Column    1 - Metal concentration of sediment used for extraction series in ug/gm (dry weight basis)  
                      2 - Dry weight (50% moisture assumed) of sediment used for extraction  
                      3 - Total metal (in ug) available for extraction series (multiply column 1 x column 2)  
                      4 - Metal concentration of sediment remaining after extractions  
                      5 - Dry weight of sediment remaining after extractions  
                      6 - Total metal (in ug) remaining after extractions (column 4 x column 5)  
                      7 - Difference between column 3 and column 6, represents total loss of each element after extraction

\* - Aqua-Regia Digestion.

TABLE 4 - d - Analysis of False Creek Sediment Before and After Extraction

Element	Before Extraction			After Extraction			
	1) Conc. ug/g	2) Sample Weight(g)	3) Total Metal(ug)	4) Conc. ug/gm	5) Sample Weight(g)	6) Total Metal(ug)	7) Loss of Extr(ug)
Arsenic As	6.1	2	12.2	1.4	1.2	1.68	10.5
	5.9	2	11.8	1.4	1.2	1.68	10.1
	7.1	2	14.2	1.3	1.1	1.43	12.7
Cadmium Cd	1.0	2	2.0	L 1.0	1.2	L 1.2	G.0.8
	1.1	2	2.2	L 1.0	1.2	L 1.2	G.1.0
	1.0	2	2.0	L 1.0	1.1	L 1.1	G.0.9
Copper Cu	86	2	172	33	1.2	39.6	132.
	90	2	180	36	1.2	43.2	137.
	86	2	172	32	1.1	35.2	137.
Iron Fe	37,300	2	74,600	19,000	1.2	22,800	51,800.
	37,600	2	75,200	19,700	1.2	23,640	51,560.
	36,700	2	73,400	19,100	1.1	21,010.	52,400.
Mercury Hg	0.98	2	1.96	0.95	1.2	1.14	0.82
	0.89	2	1.78	0.85	1.2	1.02	0.76
	0.90	2	1.80	0.85	1.1	0.93	0.87
Manganese Mn	369	2	738	348	1.2	418	320.
	380	2	760	368	1.2	442	318
	375	2	750	361	1.1	397	353
Lead Pb	106	2	212	54	1.2	64.8	147.
	106	2	212	47	1.2	56.4	156.
	118	2	236	50	1.1	55.0	181.
Zinc Zn	225	2	450	100	1.2	132.	318.
	229	2	458	99	1.2	118.	340.
	220	2	440	102	1.1	112.	328.

Key	Column	
	1	- Metal concentration of sediment used for extraction series in ug/gm (dry weight basis)
	2	- Dry weight (50% moisture assumed) of sediment used for extraction
	3	- Total metal (in ug) available for extraction series (multiply column 1 x column 2)
	4	- Metal concentration of sediment remaining after extractions
	5	- Dry weight of sediment remaining after extractions
	6	- Total metal (in ug) remaining after extractions (column 4 x column 5)
	7	- Difference between column 3 and column 6, represents total loss of each element after extraction

TABLE 4 - e(i) - Weights Of Sediment During Extractions Of Victoria Harbour Sediment

Extraction No.	Wet Weight (g)			Dry Weight (g)		
	Test 1 A	1 B	1C	Test 1 A	1B	1C
1	50.	50.	50.	18.4	18.4	18.4
2	---	---	---	---	---	---
3	---	---	---	---	---	---
4	---	---	---	---	---	---
End	---	---	---	1.1	1.2	1.0

TABLE 4 - e(ii) - Weights of Sediment During Extractions Of False Creek Sediment

Extraction No.	Wet Weight (g)			Dry Weight (g)		
	Test 2 A	2 B	2C	Test 2 A	2B	2C
1	50.	50.	50.	25.0	25.0	25.0
2	4.0	4.0	4.0	approx. 2.0	2.0	2.0
3	---	---	---	---	---	---
4	---	---	---	---	---	---
End	---	---	---	1.2	1.2	1.1

TABLE 5 - a - Extraction of Victoria Harbour Sediment After Release Experiment

Metal	Extraction No.				Sum
	1	2	3	4	
Arsenic As	L 0.5 L 0.5	0.70 0.80	7.6 7.8	L 0.50 L 0.50	8.30 8.60
Cadmium Cd	0.80 L 0.50	0.80 0.80	3.9 4.0	L 0.50 L 0.50	5.40 4.80
Copper Cu	2.20 2.20	0.50 0.70	673 680	5.0 5.0	681. 688.
Iron Fe	L 5.0 L 5.0	1075. 1135.	57,300 59,300	8200. 8100.	66,600. 68,600.
Mercury Hg	L 0.05 L 0.05	L 0.05 L 0.05	L 0.05 L 0.05	L 0.05 L 0.05	L 0.05 L 0.05
Manganese Mn	L 5.0 L 5.0	84. 90.	496 519	140. 160.	720. 769.
Lead Pb	L 10.0 L 10.0	1.10 1.0	791 805	23. 25.	815. 831.
Zinc Zn	21.0 23.0	129. 130.	671 620	* 12.0 * 6.0	833. 779.

All results expressed as micrograms.

Extraction 1 - Ammonium Acetate

2 - Hydroxylamine Hydrochloride

3 -  $H_2O_2$  Digestion/Ammonium Acetate

4 - Na-citrate/Na dithionite.

Sum - represents total ug of each element found in the extracts.

L = Less than

FALSE CRK

TABLE 5 - b - Extraction of False Creek Sediment After Release Experiment

Metal		Extraction No.				Sum
		1	2	3	4	
Arsenic	As	L 0.5 L 0.5	L 0.5 L 0.5	6.0 6.1	L 0.5 L 0.5	6.0 6.1
Cadmium	Cd	0.9 0.8	1.0 0.7	3.3 2.8	L 0.5 L 0.5	5.2 4.3
Copper	Cu	11. 10.	0.6 0.5	105 95	L 1.0 L 1.0	117 106
Iron	Fe	L 5. L 5.	255 250	2700 2800	42,500. 37,500.	45,500. 40,600.
Mercury	Hg	L 0.05 L 0.05	L 0.05 L 0.05	L 0.05 L 0.05	L 0.05 L 0.05	L 0.05 L 0.05
Manganese	Mn	7 8	60 53	168 168	116 116	351 345
Lead	Pb	L 10 L 10	0.2 0.1	60 51	L 10 L 10	60.2 51.1
Zinc	Zn	38 36	52 47	220 228	- -	310 311

All results expressed as micrograms.

Extraction 1 - Ammonium Acetate

2 - Hydroxylamine Hydrochloride

3 - H<sub>2</sub>O<sub>2</sub> Digestion/Ammonium Acetate

4 - Na-citrate/Na dithionite.

Sum - represents total ug of each element found in the extracts.

L = Less than

TABLE 5 - c - Analysis of Victoria Harbour Sediment After Release Experiment

Element	Before Extraction			After Extraction			
	1) Conc. ug/g	2) Sample Weight(g)	3) Total Metal(ug)	4) Conc. ug/gm	5) Sample Weight(g)	6) Total Metal(ug)	7) Loss of Extr(ug)
Arsenic As	5.4	4.9	26.5	1.0	2.6	2.60	23.9
	5.5	4.5	24.8	0.9	2.6	2.34	22.4
Cadmium Cd	1.1	4.9	5.39	L 1.0	2.6	L 2.6	G 2.05
	1.3	4.5	5.85	L 1.0	2.6	L 2.6	G 2.27
Copper Cu	185	4.9	906.	42	2.6	109.	797.
	190	4.5	855.	43	2.6	112.	743.
Iron Fe	22,100	4.9	108,000.	13,900	2.6	36,100	71,900.
	24,300	4.5	109,000.	10,500	2.6	27,300	82,000.
Mercury Hg	3.3	4.9	16.2	5.4	2.6	14.0	2.13
	3.5	4.5	15.8	5.4	2.6	14.0	1.71
Manganese Mn	238	4.9	1170.	157	2.6	408.	762.
	249	4.5	1060.	115	2.6	299.	763.
Lead Pb	255	4.9	1250	60.	2.6	156.	1090.
	254	4.5	1140	98.	2.6	255.	888.
Zinc Zn	218	4.9	1068	68.	2.6	177.	891.
	218	4.5	981	60.	2.6	156.	825.

Key	Column	Description
	1	- Metal concentration of sediment used for extraction series in ug/gm (dry weight basis)
	2	- Dry weight (50% moisture assumed) of sediment used for extraction
	3	- Total metal (in ug) available for extraction series (multiply column 1 x column 2)
	4	- Metal concentration of sediment remaining after extractions
	5	- Dry weight of sediment remaining after extractions
	6	- Total metal (in ug) remaining after extractions (column 4 x column 5)
	7	- Difference between column 3 and column 6, represents total loss of each element after extraction

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TABLE 5 - d - Analysis of False Creek Sediment After Release Experiment

Element	Before Extraction			After Extraction			
	1) Conc. ug/g	2) Sample Weight(g)	3) Total Metal(ug)	4) Conc. ug/gm	5) Sample Weight(g)	6) Total Metal(ug)	7) Loss of Extr(ug)
Arsenic As	4.8	6.1	29.3	2.8	4.0	11.2	18.1
	4.6	6.0	27.6	3.3	3.5	11.6	16.0
Cadmium Cd	1.0	6.1	6.1	L 0.5	4.0	L 2.0	G 4.1
	0.8	6.0	4.8	L 0.5	3.5	L 1.75	G 3.0
Copper Cu	70.	6.1	427	68	4.0	272.	155.
	65.	6.0	390	63	3.5	220.	170.
Iron Fe	28,000	6.1	170,800	26,000	4.0	104,000	66,800
	25,900	6.0	155,400	26,700	3.5	93,450	61,950
Mercury Hg	1.1	6.1	6.71	0.90	4.0	3.60	3.11
	1.1	6.0	6.6	0.90	3.5	3.20	3.40
Manganese Mn	345	6.1	2105	366	4.0	1464	641
	324	6.0	1944	345	3.5	1208	736
Lead Pb	84	6.1	512.4	85	4.0	340	172
	70	6.0	420	73	3.5	256	164
Zinc Zn	153	6.1	933.3	118	4.0	472	461.
	125	6.0	750	110	3.5	385	365.

Key      Column    1 - Metal concentration of sediment used for extraction series in ug/gm (dry weight basis)  
                      2 - Dry weight (50% moisture assumed) of sediment used for extraction  
                      3 - Total metal (in ug) available for extraction series  
    (multiply column 1 x column 2)  
                      4 - Metal concentration of sediment remaining after extractions  
                      5 - Dry weight of sediment remaining after extractions  
                      6 - Total metal (in ug) remaining after extractions (column 4 x column 5)  
                      7 - Difference between column 3 and column 6, represents total loss of each element after extraction

TABLE 5 - e - Weights Of Sediment During Extractions Of Sediments Remaining  
After Release Experiments

Extraction No.	Wet Weight (g)				Dry Weight (g)			
	1-A	1-B	2-A	2-B	1-A	1-B	2-A	2-B
1	10.0	10.0	10.0	10.0	4.9	4.5	6.1	6.0
2	8.8	8.8	9.6	9.2				
3	7.1	7.0	8.3	8.0				
4	4.4	4.5	6.4	5.3				
End	-	-	-	-	2.6	2.6	4.0	3.5

1 - A &amp; 1 - B - Victoria Harbour

2 - A &amp; 2 - B - False Creek

## 5.0 DISCUSSION

Standard analytical procedures were utilized in the analysis of the interstitial water (TABLE 1), the sediment used for extraction (TABLE 2) and the elutriates from the 4:1 elutriate test (TABLE 3). No difficulties were encountered with these analyses.

The extraction series however, were unique in terms of analytical approach and a number of points are worthy of consideration for future projects of this nature.

It is apparent from the results presented in Tables 4-a to 4-c and 5-a to 5-e, that considerable losses of sediment did occur during various extraction stages (approximately 50% total loss in each case - see tables 4-e and 5-e).

These weight changes can be attributed to dissolution of various constituents, and transfer losses, most notably from filtration. Because of the weight loss problem, it was difficult to calculate the element concentration obtained from the extracts, in terms of microgram per gram of sediment.

It would have been advantageous to work with larger initial samples than specified in the procedure from the Request for Proposal. This would have allowed the removal of aliquots at each stage for moisture determination. The dry weight of sediment available for each extraction, could then have been determined, thereby allowing a re-calculation of the extract results in ug/gram of sediment.

The information that was obtained concerning sediment weights at various extraction stages, is documented in Table 4-e and 5-e. Considerably more information was obtained for the second experiment (Table 5-e with sediments from the release experiments) than for the first experiment (4-c) since the importance of such information became apparent after the first extraction experiment was complete. It was decided however, during the second extraction experiment, not to remove sub-samples for moisture, because of the small sample size.

These two tables should allow the Scientific Authority to estimate the dry weight concentrations at the various stages and thereby calculate the results in Tables 4-a, 4-b, 5-a, and 5-b in terms of micrograms per gram if this is deemed desirable.

## 5.0 DISCUSSION (continued)

Another factor which must be recognized in interpreting the results, is that the removal of "wet" aliquots from a sediment, represents a sampling problem. The only adequate means, by which a representative sample can be removed from a sediment core, is to dry and then blend or pulverize entire core prior to removal of an aliquot by proper sampling technique (rolling, quartering etc.). This, of course, would not be consistent with the intent of this project, and removal of "wet" aliquots was the only manner in which samples could be removed. This situation was compensated for by carrying out all analyses in triplicate or duplicate.

Some difficulties were encountered, in the analysis of the extracts due to matrix effects with atomic absorption (AA) spectrophotometry. It was absolutely essential to prepare A.A. standards with the various extraction solutions in order to closely match the matrix of the samples, and to use the background correction for all analyses. Some of the solutions could also be analysed by graphite furnace AA where matrix effects are even more serious.

The citrate/dithionite extracts could not be analysed by graphite furnace and these were also the most difficult to handle by flame AA. Firstly, the dithionite contained a high concentration of zinc requiring extensive clean-up which was not completely effective in zinc removal. Secondly the extraction solution was quite viscous and very high in sodium. If an alternate reducing solution could have been employed it may have allowed a better analysis of EXTRACT 4.

The "oxygen free" requirements were difficult to achieve and made the initial preparation steps awkward to carry out. It would have been difficult to handle larger samples with our apparatus. In future we would attempt to carry out this work in a glove box that was about 100 x 200 cm in size at least. Even better would be to work in an "air tight" room filled with nitrogen with breathing air supplied to the analyst.

## 6.0 BIBLIOGRAPHY

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