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**FINAL REPORT**

**BENCH SCALE STUDIES  
HAMILTON HARBOUR SEDIMENTS**

**CONTRACT NO. 1-6021**

**for**

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## **I. INTRODUCTION**

### **1.0 BACKGROUND**

Concern about Great Lakes water quality and the sources of pollution affecting the water quality emerged in the late 1960's. In 1972, Canada and the United States signed the first Great Lakes Water Quality Agreement to initiate a joint effort to address the pollution problems evident in the Great Lakes basin.

Forty-two Areas of Concern (AOC's) have been identified and preparation of Remedial Action Plans (RAP) for all the areas has been initiated. Contaminated sediments are one of the pollution sources under consideration. Forty-one of the forty-two AOC's are confirmed to contain contaminated sediments.

Canada's commitment to the cleanup of the AOC's is being coordinated under the Canada/Ontario Remedial Action Plan. As Environment Canada's contribution, the Great Lakes Cleanup Fund was established to provide federal assistance in the development and interpretation of cleanup options. The Contaminated Sediments Treatment Technology Program, an initiative of the Great Lakes Cleanup Fund is committed to financial support of bench, pilot and full scale demonstration projects of promising technology for treatment of contaminated sediments in five selected AOC's. The Wastewater Technology Centre was selected to administer the Program.

The Alberta Research Council and United States Electric Power Research Institute have, over the past few years, developed a novel process for the clean-up of soils contaminated with hydrocarbons. The potential of the ARC/EPRI Clean Soil Process has been tested on a number of contaminated soils. Particular emphasis was placed on applying the process to remediate old Manufactured Gas Plant sites by removing the tarry contaminant and the coal (coke, char, etc.) from the soil matrix. During the development of the process, broader applications were revealed including remediation of soils contaminated with oils and other types of hydrocarbons.

However, no experience was acquired in treatment of extremely fine solids such as is the case of sediments. Therefore, in order to evaluate the ability of the ARC/EPRI process to clean sediments, ARC responded to the WTC, RFP-0002. As a result, the Contract No. 1-6021 on "Bench Scale Studies - Welland River or Hamilton Harbour Sediments" was awarded. The

sample of Hamilton Harbour Sediment was received on October 25, 1991. This report presents the results obtained for sample characterization and treatment.

## **2.0 ARC/EPRI CLEAN SOIL PROCESS**

The Clean Soil Process is an outgrowth of an advanced coal cleaning technology based on the principle of coal agglomeration. In coal agglomeration, the coal is selectively wetted by oil which is added as a bridging liquid, and the mineral matter, that is often associated with coal, is wetted by water. The result is an increase in the size of coal particles and a separation of the minerals from the coal.

When the principle of coal agglomeration is applied to the cleanup of soils contaminated with oil/tar, the contaminants in the soil assume the role of a bridging liquid. The mechanism of contaminant transfer is based on abrasion mass transfer of contaminants from mineral matter to coal particles. The process performance is determined by the ease with which the soil releases the contaminants, the contaminant affinity towards coal, and the adsorption capacity of the coal. The end-products are clean soil that would be returned to the site, and coal/contaminant agglomerates that would be utilized as a fuel in, for example, coal-fired power plants.

The Clean Soil Process has been developed through an extensive batch experimental program followed by verification in a 250 kg/hr continuous testing facility. A simplified diagram of the Clean Soil Process and the block diagram of the 250 kg/hr pilot plant, are shown in Figures 1 and 2, respectively.

The process, which is a modified version of Agloflotation, consists of two steps: mixing/cleaning and flotation (separation). In the first step, ground coal (particle size less than 0.6 mm) is mixed with the contaminated soil and water in a rotary drum. Solids concentration is typically on the order of 60%. During mixing/cleaning, the transfer of oily/tarry contaminants from soil to coal takes place. The material discharged from the drum is screened at 1 mm. The -1 mm material passes to the second step in which the contaminant-loaded coal microagglomerates are separated by flotation from the fine clean soil and tarry/oily fines. The latter are separated from the fine clean soil by decantation or hydrocycloning. The +1 mm oversized solids are separated by gravity separation (e.g., jigging) into two fractions: coarse clean solids (inorganic solids, e.g. rocks, stones, pebbles) and coarse organic solids (largely organic impurities, e.g. coke, char,

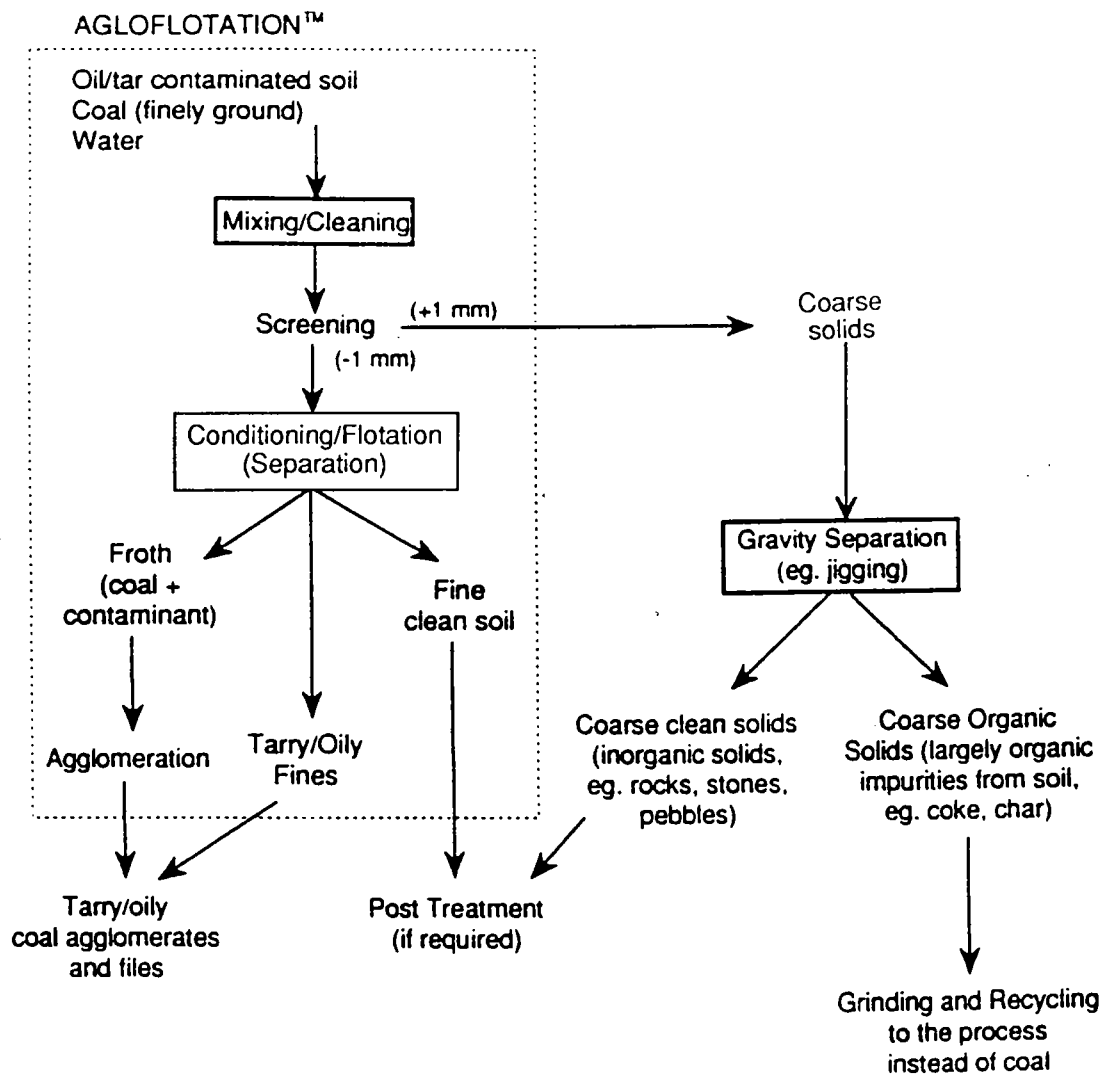


Figure 1. Scheme of Clean Soil Process

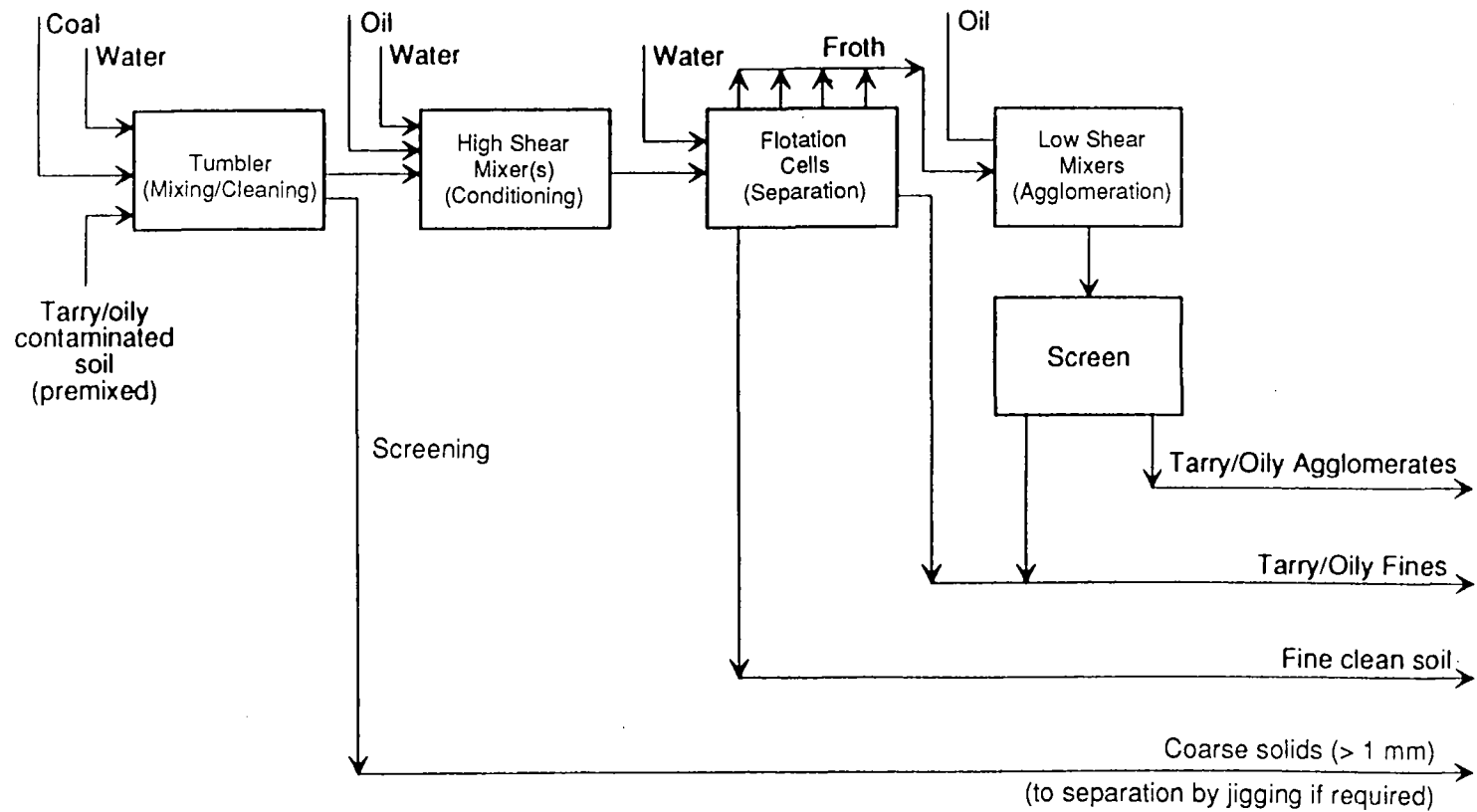


Figure 2. The Block Diagram of the 250 kg Per Hour Pilot Plant

slag). After grinding, the organic fraction could be recycled into the process and used instead of coal. Water used in the process can also be recycled. The process does not produce contaminated water. Coal used in the process acts in a manner similar to charcoal and active carbon which are commonly employed in water treatment for removing organic pollutants.

In cases when the residual contaminant concentration in the fine clean soil and/or the coarse clean solids is too high to comply with local regulations, the fine and/or coarse clean soil/solids could be subjected to post-treatment using other suitable processes (e.g., thermal desorption).

The parameters that determine the process performance are slurry concentration, mixing intensity, residence time, cleaning temperature, coal addition and froth collector addition. The latter three parameters are closely associated with the composition of the contaminated material, and therefore have to be optimized in each case.

Testing of a number of contaminated soils has shown that regardless of the initial contaminant concentration, the Clean Soil Process is capable of removing up to 99% of oily and/or tarry waste from the mineral matrix. Overall, the process can result in considerable volume reduction of contaminated material due to the much greater adsorptive capacity of coal as opposed to mineral matter in soil.

Selected results, in terms of clean soil recovery and residual contaminant concentration in some of the processed samples are presented in Table 1.

**Table 1. Clean Soil Process: Batch Testing - Results**

Sample	wt %	
	Clean Soil Recovery	Residual Oil/Tar Content in Clean Soil
MGP-1	41.5	0.15
HO-1	96.3	0.04
OS-1	82.0	0.04
OS-2	77.5	0.08
OS-3	50.7	0.04
OS-4	97.5	0.06
OS-5	81.9	0.05

MGP - manufactured gas plant waste  
HO - bitumen contaminated soil  
OS - oil spills



## II. EXPERIMENTAL DESIGN AND RESULTS

### 1.0 SAMPLE HANDLING

A sample of the Hamilton Harbour Sediment (HHS) was received in two 10 litre pails. Visual inspection showed that the sample consisted of two layers:

- upper layer - muddy water
- bottom layer - solids

Following the WTC analytical protocol, the sample was allowed to settle for two days. The water layer was then separated from solids by decantation. In order to obtain uniform distribution, the solids were then thoroughly blended and transferred into 1 litre and 2 litre jars. The jars filled to the brim were tightly closed and stored at room temperature for future use. The sample preparation scheme is presented in Figure 3. Table 1-1 gives the sample weight.

**Table 1-1. HHS Sample Weight**

Sample	Grams	%
HHS as received	36,400	100.0
HHS solids	31,990	87.9
HHS water	4,410	12.1

It should be noted that decantation is of no benefit to our method of processing. To the contrary, the inherent water in sediment reduces the amount of make-up water needed in processing.

### 2.0 SAMPLE CHARACTERIZATION

Sediment sample characterization included mandatory determinations such as sample pH, concentrations of water, solids, organics, polynuclear aromatic hydrocarbons and metals.

In addition, the bulk of organic contaminants was characterized further using steam distillation and gel permeation chromatography methods. These procedures are used in our lab to estimate the

volatility and molecular weight range of organic components. Both properties are pertinent to our process, because they affect process temperature, process efficiency and also safety measures.

The solids were analyzed for particle size. The particle size distribution and concentration of organic contaminants as a function of particle size allows for presetting the process configuration, e.g. type of mixing, use of screens, flotation conditions.

Ashing of toluene extracted solids was used as an indirect method for determining the amounts of non-extractable organic carbon. If present, this has to be taken into account in preparing the process conditions.

The sample characterization scheme is shown in Figure 3. The HHS solids and water were analysed separately.

## 2.1 Hamilton Harbour Sediment (HHS) Solids

### 2.1.1 Overall Composition

The composition of the HHS sample in terms of wt % distribution of organic matter, inorganic solids and water was determined by azeotropic distillation with toluene using Dean-Stark Soxhlet extraction (75-100 g sample/200 ml toluene). The average composition is given in Table 2-1.

**Table 2-1. Overall HHS Solids Composition**

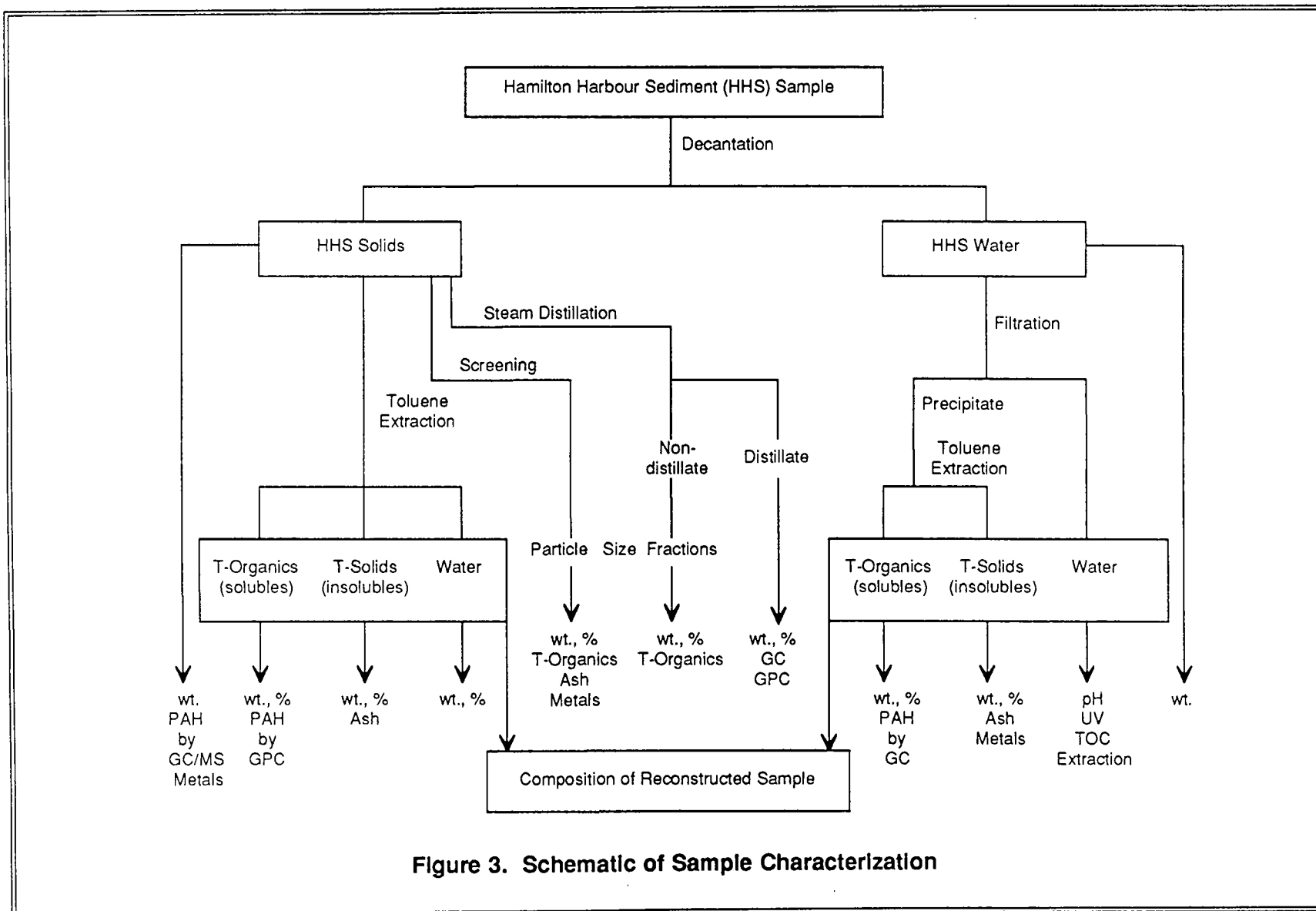
	wt%		
	Mean <sup>a</sup>	Minimum	Maximum
Water	49.32		
T-Solids <sup>b</sup>	48.11		
T-Organics <sup>c</sup>	1.77	45.92	50.10
Recovery	99.20	46.08	51.60
T-Organics <sup>d</sup>	3.55	1.60	1.90
Ash in T-Solids <sup>d</sup>	75.6		
Nitrogen <sup>d</sup>	0.45		
Sulfur <sup>d</sup>	0.63		

<sup>a</sup>of 10 extractions - sampling from various jars, Appendix 1, Table A1-1

<sup>b</sup>toluene insolubles

<sup>c</sup>toluene solubles

<sup>d</sup>dry basis



T-Organics include all hydrocarbon contaminants soluble in toluene. The concentration of organics as obtained by extraction with toluene may be somewhat lower than the actual value due to the losses of volatile compounds that occur during removal of toluene from the extract by distillation. For instance, the gas chromatographic analysis of toluene extract before and after solvent removal indicated that about 15% of naphthalene present in the sediment was lost upon toluene distillation. No other losses were detected.

### 2.1.2 Particle Size, Organics and Ash Distribution

The HHS solids were wet screened, and each fraction was analyzed for organics content by extraction with toluene and ash content after extraction by ashing, Table 2-2.

About 80% of sediment solids have a particle size below 0.1 mm. The portion of sediment with a particle size above 0.15 mm has higher toluene soluble organics concentration and lower ash content as compared to the portion of sediment with particle size below 0.15 mm. Low ash values indicate the presence of non-extractable organic carbon such as in coke and/or coal, etc., which in turn explains a high accumulation of organic contaminants in this portion.

**Table 2-2. HHS Solids Particle Size, Organics and Ash Distribution**

Particle Size mm	%		
	Yield	T-Organics	Ash <sup>a</sup>
0.000 - 0.106	79.0	3.00	82.0
0.106 - 0.150	6.8	2.49	65.9
0.150 - 0.250	7.3	7.71	43.8
0.250 - 1.000	6.9	9.91	39.9

<sup>a</sup>determined in T-Organics-free solids

### 2.1.3 Steam Distillation

The HHS solids were subjected to steam distillation. Table 2-3 shows that about 1/3 of toluene soluble organics distilled with steam. The solids before and after steam distillation were divided

by wet screening into +/- 0.15 mm particle size fractions, and the toluene soluble organics were determined in each fraction, Table 2-4. The results show that the steam distillable components of T-Organics account for about 64% of organics present in the +0.15 mm fraction and 22% of organics present in the -0.15 mm fraction.

**Table 2-3. HHS Solids Steam Distillation<sup>a</sup>**

T-Organics	wt% of HHS Solids (Dry Basis) Experiment No.			
	1	2	3	Mean
Distillable	1.18	1.09	1.21	1.16
Non-distillable <sup>b</sup>	2.18	2.32	2.49	2.33

<sup>a</sup>residence time, 15 min.; overhead temperature, 98°C; <sup>b</sup>determined in solids after distillation

**Table 2-4. T-Organics Distribution in Sediment Before and After Steam Distillation**

Fraction	Solids %	T-Organics, % in Solids	
		As Received	Steam Distillate
-0.15 mm	85.8	2.96	2.30
+0.15 mm	14.2	8.78	3.17

#### 2.1.4 Gel Permeation Chromatography (GPC)

The GPC using Biobeads SX-8 in benzene is routinely used in our laboratory to gain information pertinent to the ARC Clean Soil Process regarding molecular weight and associated volatility of T-Organics. The weight distribution of T-Organics and steam distillate as obtained by GPC separation is given in Table 2-5.

Gas chromatographic analysis indicated that naphthalene was the main component of the distillate. In the distillate, 94% of the lowest molecular weight fraction (No. 4) consists of naphthalene.

**Table 2-5. GPC Separation**

Fraction No.	Elution vol., ml	Molecular Weight Range	Yield, wt %	
			T-Organics	Steam Distillable <sup>a</sup>
1	50	high ↓ ↓ ↓ low	32.4	5.2
2	25		16.6	8.2
3	25		12.0	8.8
4	250		39.0	77.8

<sup>a</sup>steam distillate constitutes 33% of T-Organics

#### 2.1.5 Determination of Polynuclear Aromatic Hydrocarbons (PAH)

Concentration of polynuclear aromatic hydrocarbons were determined by (1) gas chromatography using internal standard and, (2) by GC/mass spectrometry according to EPA 8270 method, Table 2-6.

Naphthalene appeared to be the most prominent aromatic hydrocarbon contaminating the sediment.

There were some discrepancies in the PAH's determination by the two methods arising most likely from using different calibration procedures. The direct gas chromatographic analysis should be considered here as a diagnostic measure only, without putting emphasis on the correctness of the actual values.

The sample of Hamilton Harbour sediment contained PAH's on a level which, according to Ontario tentative guidelines, exceeded by far the Lowest Effect Level of 2 ppm and was approximately half of the Severe Effect Level of 11,000 ppm (see Appendix 2, Table A2-1).

**Table 2-6. Concentration of PAH's**

	Method	
	GC Analysis	EPA 8270
T-Organics	36789	
PAH (total)	8413	4009
Acenaphthene	64.8	73.6
Acenaphthylene	32.1	31.2
Anthracene	106.8	172.0
Benzo(a)anthracene	92.2	107.4
Benzo(b)fluoranthene	NA	
Benzo(k)fluoranthene	NA	221.5*
Benzo(ghi)perylene	45.9	51.5
Benzo(a)pyrene	78.6	118.9
Chrysene	93.8	105.2
Dibenzo(ah)anthracene	ND	Trace
Fluoranthene	240.0	435.6
Fluorene	110.0	159.1
Indeno(1,2,3-c,d)pyrene	NA	48.9
Naphthalene	7042.5	1724.1
Phenanthrene	293.3	492.5
Pyrene	212.7	267.2

Units: ppm in solids (dry basis)

NA denotes Not Analyzed

ND denotes Not Detected

Trace: above detection limit, but below quantitation limit

\*a sum of Benzo(b) and Benzo(k) fluoranthene

#### 2.1.6 Determination of Metals by ICP

Besides organic contaminants, the Great Lakes sediments are also contaminated with metals. Although the Clean Soil Process is primarily designed to deal with an organic type of contamination, treatment of metals may be an added benefit to this process.

Comparison of the criteria set out in the Ontario draft guidelines indicates that the metal concentration in the sample represents the Severe Effect Level, (Appendix 2, Table 2A-1).

**Table 2-7. Metals (ppm in Dry Solids)**

	HHS Solids		
	Total	+0.15 mm Fraction	-0.15 mm Fraction
Aluminum	12586	4990	12928
Antimony	60	37	75
Arsenic	54	24	59
Barium	83	53	114
Beryllium	9	5	8
Cadmium	10	4	12
Calcium	10349	12569	4909
Chromium	135	128	139
Cobalt	28	21	30
Copper	108	80	124
Iron	173494	87116	195068
Lead	871	324	1072
Lithium	23	19	24
Magnesium	3855	6877	6578
Manganese	2841	2354	3156
Molybdenum	16	15	18
Nickel	83	60	94
Potassium	7823	3129	9131
Phosphorus	1837	2607	1929
Selenium	215	104	229
Silver	-	-	-
Sodium	3984	1553	4179
Strontium	93	90	90
Titanium	1644	808	1911
Vanadium	98	68	108
Zinc	5693	2046	7094
Zirconium	98	54	107

## 2.2 Hamilton Harbour Sediment Water

Water decanted from the HHS feed sample contained some particulates. The particulates were removed from the water by filtration and characterized for overall composition, PAH's content by GC and metal content.

In the filtrate, the pH, UV absorbance, total organic carbon (TOC) and extractability with organic solvent were measured. The results are compiled in Table 2-8.



Table 2-8. HHS Water Characteristics

Composition	Water	
	Particulates	Filtrate
Concentration, wt %	1.01	98.99
T-Organics, % <sup>a</sup>	9.15	
Ash, % <sup>b</sup>	74.2	
Nitrogen, % <sup>b</sup>	0.82	
Sulfur, % <sup>b</sup>	1.93	
pH		7.08
TOC, ppm		5.7
UV-A, 256 nm		1.573
Water Soluble Organics, ppm		50
PAH (total), ppm <sup>b,c</sup>	10943	
Acenaphthene	78	
Acenaphthylene	47	
Anthracene	156	
Benzo(a)anthracene	193	
Benzo(ghi)perylene	ND	
Benzo(a)pyrene	203	
Chrysene	193	
Dibenzo(ah)anthracene	ND	
Fluoranthene	483	
Fluorene	100	
Naphthalene	8887	
Phenanthrene	434	
Pyrene	169	
Metals, ppm <sup>b</sup>		
Aluminum	19258	
Antimony	63	
Arsenic	65	
Beryllium	11	
Cadmium	26	
Chromium	219	
Copper	231	
Iron	191996	
Lead	1321	
Magnesium	4708	
Manganese	3628	
Nickel	117	
Selenium	186	
Zinc	21224	

<sup>a</sup>% of particulates; <sup>b</sup>% of T-Solids on dry basis; <sup>c</sup>GC analysis only; ND denotes Not Detected

### 2.3 Composition of the Hamilton Harbour Sediment Sample As Received

The composition of the bulk sediment as received was determined indirectly using the results obtained for HHS solids and HHS water, Table 2-9.

**Table 2-9. Composition of Hamilton Harbour Sediment**

Composition	wt%	ppm <sup>a</sup>
Water	55.66	36955
T-Solids	42.64	
T-Organics	1.70	
Ash	75.60 <sup>a</sup>	
Nitrogen	0.45 <sup>a</sup>	
Sulfur	0.63 <sup>a</sup>	
PAH (total) <sup>b</sup>		8419
Acenaphthene		64.8
Acenaphthylene		32.1
Anthracene		107.0
Benzo(a)anthracene		92.5
Benzo(ghi)perylene		45.8
Benzo(a)pyrene		78.9
Chrysene		94.0
Dibenzo(ah)anthracene		ND
Fluoranthene		240.6
Fluorene		110.0
Naphthalene		7047.3
Phenanthrene		293.7
Pyrene		212.6
Metals		
Aluminum		12605
Antimony		60
Arsenic		54
Beryllium		9
Cadmium		10
Chromium		135
Copper		108
Iron		173548
Lead		872
Magnesium		6422
Manganese		2843
Nickel		83
Selenium		215
Zinc		5738

<sup>a</sup>dry basis; <sup>b</sup>based on GC analysis only

### 3.0 PROCESSING OF THE HHS SOLIDS

The main thrust of the bench testing was the separation of the hydrocarbon contaminants, especially polynuclear aromatic hydrocarbons, from the sediment.

The ARC/EPRI Clean Soil Process utilizes coal as a hydrocarbon contaminant adsorbent. The end result of the treatment is a solid matrix free of contaminant and a combustible (coal + contaminant) by-product.

Depending on the composition of a contaminated material, the source of coal may be either external or internal. Previous work on contaminated soils that contained appreciable amounts of indigenous coke/coal/char indicated that the inherent absorbent could reduce or eliminate the quantity of fresh coal used in the process.

From ash analysis of the solids, it appeared that the HHS sample contained about 25% of non-extractable organic carbon. Since this carbon is usually associated with the presence of coal, the concept of utilizing this material in the processing scheme was also tested.

The fine nature of the sediment allowed the tumbler and pre-screening step, which are integral parts of the original process, to be by-passed. Mixing and conditioning were performed directly in the flotation cell. A schematic of the bench scale processing is shown in Figure 4.

The products collected were froth, middlings and tailings. Froth refers to combustible product that consists of coal and adsorbed organic contaminants; solids that collect at the bottom of the flotation cell are defined as tailings; and middlings represent particulates that remain suspended in water on completion of flotation. For each experiment the mass distribution of products and the concentration of toluene soluble organic contaminants were determined.

The process performance was determined by the recovery of soil in the form of tailings and the residual concentration of contaminants in the tailings.

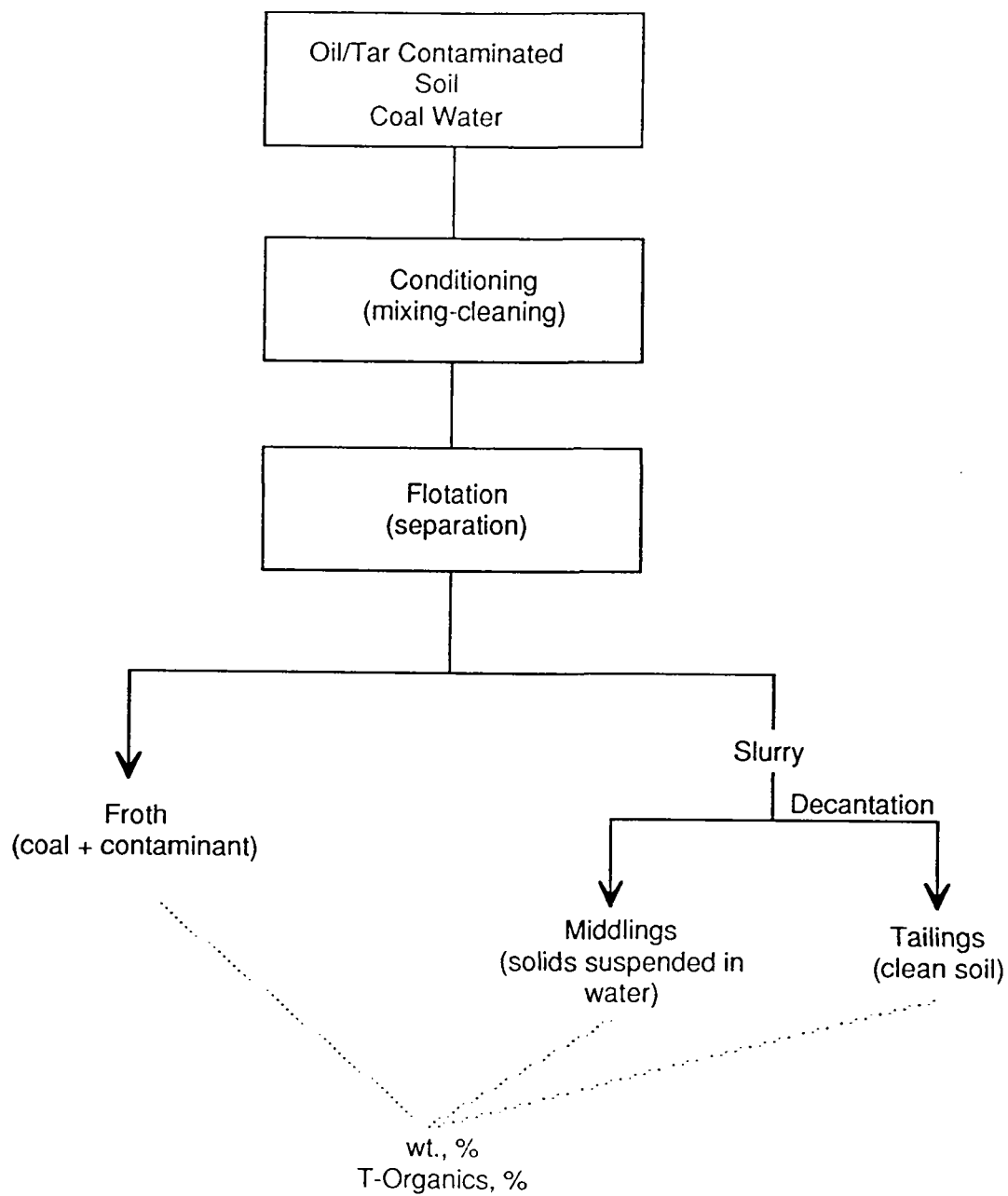


Figure 4. Schematic of Bench Scale Testing

### 3.1 Processing with Addition of Coal

Four experiments were performed using bituminous coal ground to top size of 0.6 mm. Process variables investigated were:

- amount of coal (contaminant/coal ratio 1:14 and 1:3.5)
- addition of frother (MIBC)
- addition of froth collector (bitumen/diesel)

The amount of coal is critical in the mixing/cleaning step of the Clean Soil Process. The amount required depends on the concentration of organic contaminants in the material to be processed and also on the compatibility of coal and contaminant. Frothers and froth collectors play an important role in separation by flotation by facilitating bubble-particle attachment. Methyl isobutyl carbinol (MIBC) is a frother commonly used to increase combustible recovery in coal flotation. A mixture of bitumen and diesel proved to be beneficial in enhancing the hydrophobicity of coal, which is relevant to flotation.

The nominal HHS sample charge was 400 g and the process conditions were as follow:

Conditioning	-	solids concentration, 32% temperature, 70°C residence time, 10 min. agitation, 1800 rpm, and
Flotation	-	solids concentration, 12% temperature, 40°C residence time, 4.5 min. agitation, 1400 rpm.

The best results, by a narrow margin, were obtained when MIBC frother was added at the contaminant/coal ratio of 1:14, Table 3-1 (series 1) and Appendix 1, Table A1-2. However, even in this case flotation of combustible product was not complete, as indicated by low ash values of middlings and tailings. In addition, the treatment yielded significant amounts of middlings. Overall, about 33% of sediment was recovered in the form of tailings, with an organic contaminant concentration of 0.4%. That corresponded to about 4% of the total organics present in the sediment.

**Table 3-1. HHS Solids Processing**

	Experiment Series No.		
	1 <sup>b</sup>		2 <sup>c</sup>
Conditioning Temperature, °C	70		20
Flotation Temperature, °C	40		20
Additions			
Coal, % of feed (db)	32		0
MIBC, % of feed (db)	0.008		0
Product Distribution, % (db)			
Froth	36	6 <sup>a</sup>	28
Middlings	42	62 <sup>a</sup>	51
Tailings	22	32 <sup>a</sup>	21
T-Organics content, %			
Froth	2.33		4.71
Middlings	2.65		2.60
Tailings	0.40		0.36
Ash content, %			
Froth	29.2		52.1
Middlings	67.6		84.7
Tailings	76.6		89.3

<sup>a</sup>coal free distribution; product recovery (db): <sup>b</sup>97.6%, <sup>c</sup>99.0%

### 3.2 Processing without Addition of Coal

In this series of tests, the non-extractable carbonaceous material present in the sediment were expected to play the role of coal in the cleanup process.

Five experiments were conducted under conditions listed in Section 3.1, and Table 3-1 (series 2) and Appendix 1, Table A1-3.

Process variables investigated were:

- conditioning and flotation temperature
- addition of MIBC frother (0.006%/sample)
- addition of bitumen/diesel (0.5%/sample)

None of these variables had any definitive effect on the results. The process could be carried out at ambient temperature. As compared to the results obtained in the experiments with addition

of coal, the yield of froth obtained in this series was higher and the yields of middlings and tailings were lower (Table 3-1).

Both series of tests with coal addition and without coal addition indicated a similar trend. The trend can be described as separation of the sediment based on mineral content as expressed by ash%. The ash content was increasing in the order being Froth < Middlings < Tailings, with the organic contaminants concentration decreasing in the same order.

The experiments show that there is no need to add coal for processing of HHS solids.

The presence of indigenous coal in the sediment and other highly hydrophobic species such as metal oxides, and especially iron oxides, in view of the high iron content of ~17%, which exhibit a profound affinity towards organic compounds, explains the negligible effect of using additional coal in the process.

### **3.3 Processing without Addition of Coal - Final Approach**

An undesirable behaviour of the sediment during the processing tests was an extensive formation of contaminated middlings. Moreover, there appeared to be no clear end to flotation (froth collection was arbitrarily terminated). This behaviour was attributed to the extremely fine nature of the sediment and/or to the presence of iron oxides. Therefore, an effort was made to improve flotation by employing specific conditioners/promoters. The purpose of adding conditioners/promoters was to facilitate optimal floating characteristics by modifying the surface properties of the materials to be floated.

In the sediment, the organic contaminants are deposited on coal and hydrophobic metal surfaces. Therefore, two types of conditioners/promoters were used.

- The first polyglycol ethers variety was aimed at improving the floatability of organic matter (Froth 1).
- The second anionic petroleum sulphonates group was to aid metal ore flotation (Froth 2).

Both combinations are commercially available and applied in industry. A modified version of processing and product collection is given in Figure 5.

Three experiments were performed to check the reproducibility in terms of product and toluene soluble organics distribution, Table 3-2 (Experiment Nos. 1, 2 and 3) and Appendix 1, Table A1-4. They were followed by five identical runs that produced representative samples for split samples and samples to be analysed for PAH's and metals (Table 3-2, Experiment No. 4). No process optimization was attempted.

Table 3-3 shows the organic contaminant distribution in the product streams.

**Table 3-2. HHS Solids Processing - Final Approach**

Experiment No.	wt % (Dry Basis)			
	1 <sup>b</sup>	2	3	4 <sup>a,c</sup>
Mass Distribution				
Froth 1	39.2	45.7	41.1	43.6
Froth 2	8.3	4.6	7.6	6.4
Tailings 1	52.5	49.7	51.3	50.0
T-Organics Content				
Froth 1	5.70	5.63	5.53	5.56
Froth 2	3.66	3.88	3.90	4.05
Tailings 1	1.47	1.38	1.49	1.45
Ash Content				
Froth 1	50.9	-	-	54.5
Froth 2	80.4	-	-	81.2
Tailings 1	90.5	-	-	89.9

<sup>a</sup>split samples and PAH's and metal analyses; product recovery (db): <sup>b</sup>98.8%, <sup>c</sup>99.2%

**Table 3-3. T-Organics and Ash Distribution, wt% (Experiment No. 4)**

Stream	T-Organics	Ash
Feed	100.0	100.0
Froth 1	66.9	31.0
Froth 2	7.3	7.0
Tailings 1	20.8	62.0
Loss (calculated)	5.0	



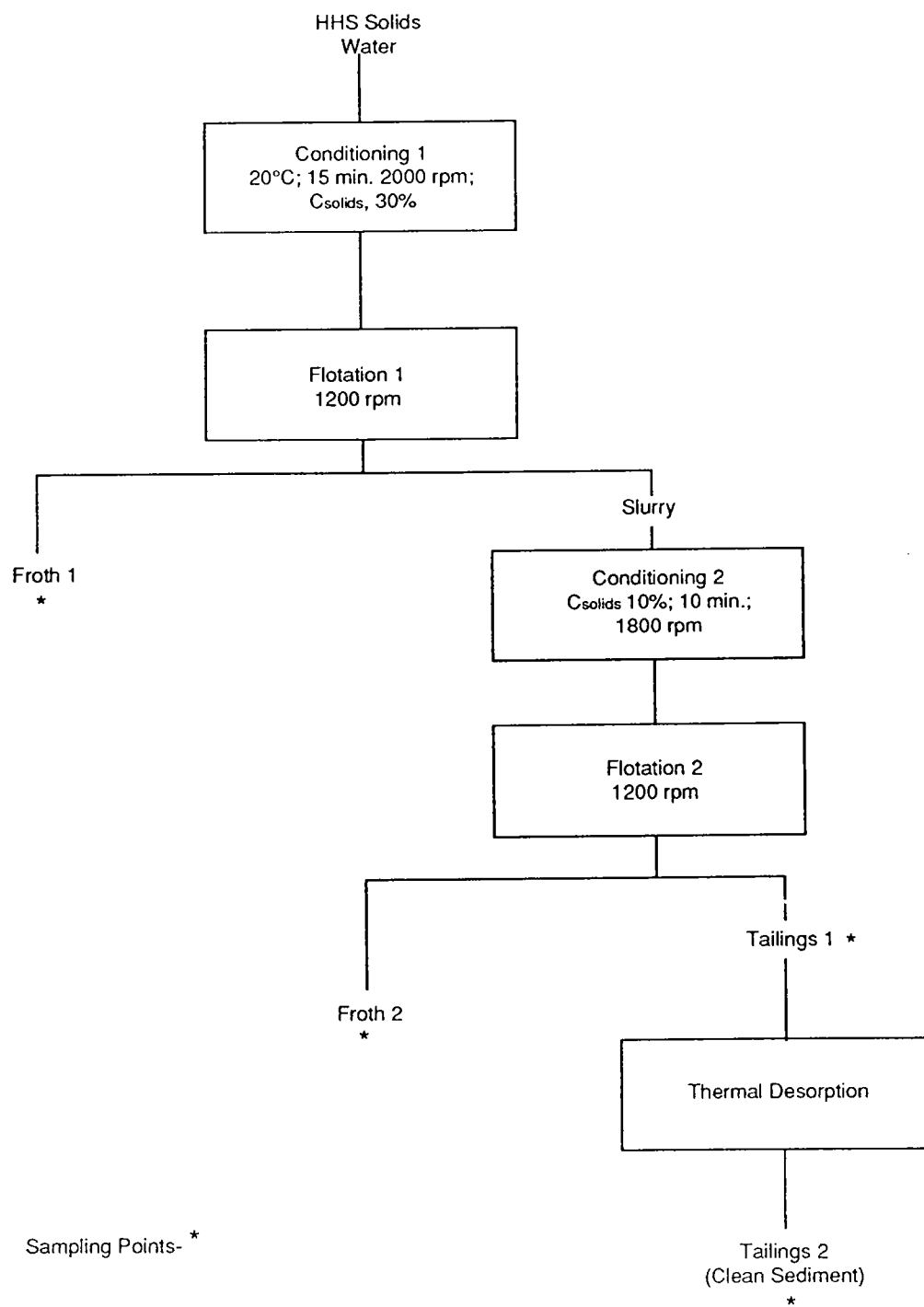


Figure 5. Modified Version of Processing

Application of the additives to the processing of sediment resulted in achieving a clear division between froth and tailings during flotation. The problem of middlings formation was eliminated. Over 60% of inorganic matter present in the sediment was recovered in tailings; retention of organic contaminant in the tailings remained unacceptably high. However, a low concentration of non-extractable organics made the tailings amenable to secondary treatment using thermal desorption. Non-extractable organics such as coal and/or chars may decompose on thermal treatment and produce additional PAH's, defeating the usefulness of thermal desorption.

### **3.4 Post Treatment of Tailings by Thermal Desorption**

The thermal desorption of Tailings 1 obtained in Experiment No. 4 (Table 3-2), was conducted in a Fisher ISOTEMP Programmable Ashing Furnace, in a fixed bed using 30-50g sample as received (25% moisture). Three experiments were conducted at temperatures from 200-300°C, Table 3-4.

A temperature of 250°C appeared to be sufficient for treatment.

**Table 3-4. Thermal Desorption of Tailings 1**

Temperature, °C	T-Organics, %
200	0.555
250	0.014
300	0.016

Heating rate, 5°C/min.

Nitrogen, 5L/min.

Residence time, 10 min.

## **4.0 PRODUCT SAMPLING AND PRODUCT ANALYSIS**

Products for analysis (see page 21) were collected according to scheme given in Figure 5. The excess water from product streams was drained by filtration. The products were distributed to jars; the jars were tightly closed and kept at room temperature. The sample size varied from 10 to 400 g depending on the type of analysis to be performed. The size of jars was selected to

reduce headspace to minimum. Samples were subjected to analysis as indicated in Table 4-1.

Almost all analyses were performed at the Alberta Research Council. The exception was the analysis of the PAH's by Method 8270 which was done by CHEMEX Laboratories Alberta, Inc.

The QA/QC activities were focussed on experimental repeatability and analytical quality. Experiments were repeated to determine overall repeatability of mass balances and organic contaminant concentration and distribution. To ensure the quality of analytical work, analytical equipment was calibrated daily, appropriate standards were used for calibration, reference samples were analysed on a regular basis.

The results obtained are listed in Table 4-2 to 4-6.

Split samples were sent to the Wastewater Technology Centre for comparative analysis. The results are included in Appendix 3. The results from the various labs compare fairly well with the exception of the Naphthalene value.

**Table 4-1. Analytical Procedures**

No.	Analysis	Product Streams			
		Froth 1	Froth 2	Tailings 1	Tailings 2*
1.	Azeotropic extraction with toluene	+	+	+	+
2.	Proximate - ash (ASTM D5142)	+	+	+	+
3.	Elemental - Nitrogen, Carbon, Sulfur (ASTM D4239)	+	+	+	+
4.	Selected PAH's by GC (internal standard)	+	+	+	+
5.	Requested PAH's (EPA Method 8270)	+	ND	+	+
6.	Metals (microwave digestion with aqua followed by combination of ICP and atomic absorption)	+	+	+	+

\*Tailings 1 after thermal desorption treatment  
ND denotes Not Determined

**Table 4-2. Azeotropic Extraction with Toluene<sup>a</sup> (wt %)**

Stream	T-Organics (Solubles)	Solids (Insolubles)	Water	Recovery
Froth 1	3.45	58.62	36.96	99.03
Froth 2	2.90	68.68	28.12	99.70
Tailings 1	1.08	73.55	25.00	99.63
Tailings 2	0.01	99.69	0.20	99.90

<sup>a</sup>Dean-Stark Soxhlet extraction.

**Table 4-3. Proximate and Elemental Analysis**

Stream	wt % (Dry Basis)			
	C	N	S	Ash
Froth 1	45.2	0.61	1.06	54.5
Froth 2	12.2	0.12	1.02	81.2
Tailings 1	5.0	0.00	0.13	89.9
Tailings 2	5.0	0.04	0.33	90.0

**Table 4-4. Selected PAH's by GC**

	Froth 1	Froth 2	Tailings 1	Tailings 2
T-Organics	58854	42225	14684	140
PAH (total)	11969	3606	2043	1.8
Acenaphthene	79.8	50.9	28.1	ND
Acenaphthylene	46.6	20.4	10.4	ND
Anthracene	140.2	84.0	43.0	ND
Benzo(a)anthracene	162.6	200.3	32.8	0.1
Benzo(ghi)perylene	121.0	50.7	ND	0.5
Benzo(a)pyrene	180.4	89.5	24.6	0.3
Chrysene	165.3	97.3	34.1	0.2
Dibenzo(ah)anthracene	ND	ND	ND	ND
Fluoranthene	327.0	236.0	103.5	0.2
Fluorene	97.9	65.6	37.3	ND
Naphthalene	9997	2236	1504	0.2
Phenanthrene	368.2	261.1	126.6	0.2
Pyrene	282.7	213.9	98.5	0.1

Units: ppm in solids (dry basis)

ND denotes Not Detected

**Table 4-5. Concentration of PAH's as Obtained by EPA 8270 Method**

	Froth 1	Tailings 1	Tailings 2
PAH (Total)	5335	819	1.9
Acenaphthene	118.2	22.3	ND
Acenaphthylene	86.6	4.5	ND
Anthracene	392.3	58.2	ND
Benzo(a)anthracene	233.7	34.5	Trace
Benzo(b)fluoranthene			
Benzo(k)fluoranthene	430.7*	56.0*	1.0
Benzo(ghi)perylene	99.8	8.8	Trace
Benzo(a)pyrene	215.1	25.7	ND
Chrysene	207.1	45.7	Trace
Dibenzo(ah)anthracene	19.6	Trace	Trace
Fluoranthene	565.5	133.8	ND
Fluorene	220.9	49.8	ND
Indeno(1,2,3-c,d)pyrene	110.4	18.5	Trace
Naphthalene	1470.3	121.5	0.9
Phenanthrene	747.3	154.9	Trace
Pyrene	417.9	85.2	Trace

Units: ppm in solids (dry basis); ND denotes Not Detected; \*a sum of Benzo(b) and Benzo(k) fluoranthene

**Table 4-6. Metal Concentration in Product Streams**

Metals	Froth 1	Froth 2	Tailings 1	Tailings 2
Aluminum	11327	10625	14925	14830
Antimony	34	37	67	49
Arsenic	34	40	39	39
Beryllium	8	12	11	13
Cadmium	14	17	8	10
Chromium	119	187	156	160
Copper	140	154	80	93
Iron	163006	220983	239870	230027
Lead	1198	1479	584	853
Magnesium	6474	10662	5215	6074
Manganese	2960	4579	3980	4071
Nickel	89	127	95	101
Selenium	118	142	197	168
Zinc	9074	11710	5010	6554

Units: ppm in solids (dry basis)

### III. DISCUSSION

The ARC/EPRI Clean Soil Process utilizes coal for clean-up of hydrocarbons contaminated soil. The amount of coal needed for treatment depends on the hydrocarbons concentration and their affinity towards coal. The process efficiency in terms of clean soil recovery and concentration of the residual organic contaminant in soil depends on the texture and composition of soil.

There was no benefit in using additional coal in the processing of the Hamilton Harbour Sediment. The sediment was found to contain sufficient amount of non-extractable combustibles, such as coal, to theoretically adsorb all hydrocarbon contaminants (organics/coal ratio, 1:7). The analysis showed that this coal had already a higher accumulation of contaminants per unit mass than the rest of the sediment. However, its cleaning potential could not be further utilized likely because of the presence of highly hydrophobic metal oxides such as iron of which the sediment contained about 20%. Some organic matter is known to strongly adhere to iron oxides through complexation and as such, can not be dislodged by the physical process of which transfer of organic contaminants from mineral matter to coal is an example.

In view of these results, it was decided to concentrate more on the flotation step in the process.

Evaluation of flotation for sediment treatment was initially carried out under standard conditions established for the Clean Soil Process. Also, in what was called the final approach, selected conditioners/promoters were applied to improve the selectivity and floatability of organic matter and metal oxides. However, because of contract constraints, comprehensive optimization tests could not be completed.

The results obtained from the final approach can be summarized as follows:

- Satisfactory separation of coal was obtained. Coal product (Froth 1) contained about 76% of non-extractable organic carbon and 70% of toluene soluble organics.
- Froth 1 and Froth 2 retained almost all of the sulfur and nitrogen that were present in the sediment.
- Due to removal of coal by flotation, about 50% of the sediment was left in the form of tailings which were amenable to thermal desorption.

- Post-secondary treatment of the sediment by thermal desorption brought the total PAH's concentration to a level below 2 ppm.
- No sharp separation of metals in the product fractions was observed.
- Further optimization tests would have to be done to improve the separation.

Advantages of this approach are:

- Flotation, which is an integral part of the Clean Soil Process, is one of the least expensive separation procedures that has been commercially applied in both coal and mineral industries for a long period of time.
- When applied to the treatment of this sediment, flotation results in organic contaminant volume minimization and improves handleability of the product streams.
- Post-treatment of sediment tailing using thermal desorption is made practical due to the separation of non-extractable organic solids with this processing method. Thermal desorption of the Hamilton Harbour Sediment as received would be both environmentally and economically a much more difficult task. During thermal treatment, coal present in the sediment may generate additional tar and PAH's that might be partially retained in solid matrix. Also, sulfur and nitrogen present in coal may be partially volatilized creating additional emission hazards.

#### IV. CONCLUSION

Based on the information gathered in these bench scale studies, the approach suggested for treatment of the Hamilton Harbour Sediment is as follows:

- removal of the carbonaceous materials that include most of the PAH's as well as sulfur and nitrogen bearing compounds by flotation, and then
- depending on the residual content of PAH's, thermal desorption of the remaining sediment.

## APPENDIX 1



**Table A1-1. Dean-Stark Soxhlet Extraction of HHS Solids with Toluene<sup>a</sup>**

Experiment No.	wt %		
	Toluene Solubles	Toluene Insolubles	Water
1	1.78	46.08	50.05
2	1.60	51.60	45.92
3	1.76	46.40	50.10
4	1.87	50.11	47.50
5	1.88	49.04	48.88
6	1.72	47.38	49.96
7	1.90	46.30	50.10
8	1.68	47.82	50.00
9	1.71	48.81	49.18
10	1.84	47.56	50.10

<sup>a</sup>Sample size, 60-80 grams; Toluene, 200 ml; Extraction time, 12 hours

**Table A1-2. Processing with Addition of Coal**

	Experiment No.			
	1	2	3	4
Charge, gms				
HHS Solids	400	400	400	400
Coal	100	100	100	25
Water	400	400	400	450
Process Temperature °C <sup>a</sup>	70	70	70	22
Additives				
MIBC, 0.5%, ml	0	5	0	3
Bitumen/Diesel (1:1) gm	0	0	2	0
Products (db), gms				
Froth	308.2	305.7	308.5	224.9
Middlings	95.6	109.0	187.8	72.2
Tailings	173.6	128.9	78.0	69.5
Tailings	39.0	67.8	42.7	83.2
Mass Distribution, %				
Froth	31	36	61	32
Middlings	56	42	25	31
Tailings	13	22	14	37
Toluene Solids Concentration, % (db)				
Froth	2.66	2.33	3.13	4.08
Middlings	2.10	2.65	2.52	2.26
Tailings	0.34	0.40	0.36	0.91

<sup>a</sup>Mixing/conditioning

**Table A1-3. Processing without Addition of Coal<sup>a</sup>**

	Experiment No.				
	1	2	3	4	5
Temperature °C					
Conditioning	71	70	21	21	20
Flotation	42	41	21	22	20
Additives					
MIBC, 0.5%, ml	5	0	5	0	0
Bitumen/Diesel (1:1) gm	0	2	0	2	0
Products (db), gms	207.7	211.3	213.2	210.3	208.2
Froth	59.9	59.6	64.2	41.3	58.3
Middlings	96.7	104.2	115.5	149.2	106.2
Tailings	51.0	47.5	33.5	19.8	43.7
Mass Distribution, %					
Froth	29	28	30	20	28
Middlings	46	49	54	71	51
Tailings	25	22	16	9	21
Toluene Solids Concentration, % (db)					
Froth	4.37	4.45	5.47	6.36	4.71
Middlings	3.00	3.34	1.89	2.50	2.60
Tailings	0.46	0.39	0.25	0.31	0.36

<sup>a</sup>Charge: 400 g sample (HHS solids)/400 ml water

**Table A1-4. Final Approach - Experimental Matrix<sup>a</sup>**

Particular	Unit	Amount
Charge	gms	
HHS solids sample as received		400
Water		500
Solids Concentration	wt %	
Conditioning 1		30
Conditioning 2		10
Flotation 1		10
Flotation 2		~6
Additives	ppm (wt)	
Conditioning 1		
Octyl Dodecyl Dimethyl Ammonium Chloride		0.04
BETZ M150		100.00
Conditioning 2		
AEROPROMOTER 801		250.00
AEROPROMOTER 825		250.00

<sup>a</sup>Temperatures and mixing intensity given in Figure 5

## APPENDIX 2

Table 2A-1

TABLE 1 - IN-PLACE SEDIMENT CRITERIA COMPARISONS (Part 1)									
PARAMETER (µg/g dry weight)	ONTARIO (Draft)		U.S. EPA			WISCONSIN		GREAT LAKES BACKGROUND CONCENTRATIONS	
	Lowest Effect Level	Severe Effect Level	Non- Polluted	Moderately Polluted	Heavily Polluted	MAC Lake Michigan	MAC Lake Superior	Present	Past
Antimony (Al)	6	33	< 3	3-8	> 8	10	10	1.1-10.5	--
Arsenic (As)									
Beryllium (Be)	0.6	10	--	--	> 6	1.0	1.0	0.9-2.5	0.6-1.3
Cadmium (Cd)	26	110	< 25	25-75	> 75	75	100	32.0-163.0	36.0-62.0
Chromium (Cr)	16	110	< 25	25-50	> 50	50	100	22.0-62.0	21.0-62.0
Copper (Cu)	20000	40000	< 17000	17000-25000	> 25000				
Iron (Fe)	31	250	< 40	40-60	> 60	50	50		
Lead (Pb)	0.2	2	--	--	≥ 1	0.1	0.1		
Mercury (Hg)	16	75	< 20	20-50	> 50	50	100	24.0-95.0	36.0-57.0
Nickel (Ni)						1.0	1.0	0.6-1.2	1.8
Selenium (Se)									
Silver (Ag)									
Thallium (Tl)									
Zinc (Zn)	120	820	< 90	90-200	> 200	100	100	62.0-192.0	74.0-106.0
Total Organic Carbon %	1	10							
Total Sulfur									
Acid Volatile Sulfide									
Total Phenols			< 1000 <sup>2</sup>	1000-2000	> 2000	1000			
Oil and Grease									
pH									
Acenaphthene									
Acenaphthalene									
Anthracene									
Benzo(a)anthracene									
Benzo(b)fluoranthene									
Benzo(k)fluoranthene									
Benzo(ghi)perylene									
Benzo(a)pyrene									
Chrysene									
Dibenzo(ah)anthracene									
Fluoranthene									
Fluorene									
Indeno(1,2,3-cd)pyrene									
Naphthalene									
Phenanthrene									
Pyrene									
Radionuclides									
Total PAHs	(2)	(11000)	-- <sup>3</sup>	-- <sup>3</sup>	≥ 10	.05	.05		
Total PCBs	0.07 <sup>1</sup>	530							

## NOTES:

\* All units are in µg/g (ppm) dry weight unless otherwise noted

( ) Denotes tentative guidelines

Lowest Effect Level (LEL) and Severe Effect Level (SEL) are based on the 5<sup>th</sup> and 95<sup>th</sup> percentiles respectively of the Screening Level Concentration (SLC)

-- Denotes that values have not been established

1. No Effect Level given as 0.01

2. Hexane Solubles

3. Pollutational classification of sediments with total [PCB] between 1.0 and 10.0 mg/kg dry weight determined on a case-by-case basis

Table 2A-2

TABLE 1 - IN-PLACE SEDIMENT CRITERIA COMPARISONS (Part 2)						
PARAMETER (µg/g dry weight)	NETHERLANDS <sup>4</sup>			GERMANY		
	Quality Objective 2000	Provisional Test Value	Provisional Warning Value	Clay Soil Standard	Arable Land Standard	Limit for Improvement Investigations
Antimony (At)						
Arsenic (As)	85	85	150	(9)	(20)	50
Beryllium (Be)						
Cadmium (Cd)	2	7.5	30	0.3	3	20
Chromium (Cr)	480	480	1000	90	100	800
Copper (Cu)	35	90	400	45	100	500
Iron (Fe)						
Lead (Pb)	530	530	1000	20	100	600
Mercury (Hg)	0.5	1.6	15			
Nickel (Ni)	35	45	200	.. <sup>6</sup>	(50)	500
Selenium (Se)						
Silver (Ag)						
Thallium (Tl)						
Zinc (Zn)	480	480	1000	95	300	3000
Total Organic Carbon %						
Total Sulfur						
Acid Volatile Sulfide						
Total Phenols						
Oil and Grease						
pH						
Acenaphthene						
Acenaphthalene						
Anthracene	0.05	0.8	3			
Benzo(a)anthracene	0.05	0.8	3			
Benzo(b)fluoranthene	0.2	0.8	3			
Benzo(k)fluoranthene	0.2	0.8	3			
Benzo(ghi)perylene	0.05	0.8	3			
Benzo(a)pyrene	0.05	0.8	3			
Chrysene	0.05	0.8	3			
Dibenzo(ah)anthracene	0.05	0.8	3			
Fluoranthene	0.3	2.0	7			
Fluorene						
Indeno(1,2,3-cd)pyrene	0.05	0.8	3			
Naphthalene						
Phenanthrene	0.05	0.8	3			
Pyrene	0.05	0.8	3			
Radionuclides						
Total PAHs						
Total PCBs		0.2 <sup>5</sup>	0.4 <sup>5</sup>			

## NOTES:

- \* All units are in µg/g (ppm) dry weight unless otherwise noted
- ( ) Denotes tentative guidelines
- Denotes that values have not been established
- 4. Criteria are for standard sediment (organic content - 10%, lutum - 25%)
- 5. Sum of 7 PCBs, IUPAC numbers 28, 52, 101, 118, 138, 153, 180
- 6. Under development

Factors for other, non-standard, sediments are:

i.e. Criteria non-standard sediment = f(L,H) \* Reference / f(L=25, H=10)

Arsenic	15 + 0.4 (L + H)	H = Weight percentage of organic matter basis in the soil
Cadmium	0.4 + 0.007 (L + 3H)	H = 10 for 'standard sediment'
Chromium	50 + 2L	L = Weight percentage of the clay fraction
Copper	15 + 0.8 (L + H)	(particles smaller than 2µm) in the soil
Lead	50 + H + L	L = 25 for 'standard sediment'
Mercury	0.2 + 0.0017 (2L + H)	
Nickel	10 + L	
Zinc	50 + 1.5 (2L + H)	

Organics H / 10

Table 2A-3

TABLE 6 - DREDGED MATERIAL GUIDELINES			
PARAMETER (mg/kg dry weight)	ONTARIO MINISTRY OF THE ENVIRONMENT DREDGED MATERIAL		
	Open Water Disposal	Unrestricted Land Use	Restricted Land Use
Antimony (At)			
Arsenic (As)	8.0	14.0	20.0
Beryllium (Be)			
Cadmium (Cd)	1.0	1.6	4.0
Chromium (Cr)	25.0	120.0	120.0
Copper (Cu)	25.0		
Iron (Fe)	1.0 %	35.0	35.0
Lead (Pb)	50.0	60.0	500.0
Mercury (Hg)	0.3	0.5	0.5
Nickel (Ni)	25.0	32.0	60.0
Selenium (Se)	--	1.6	2.0
Silver (Ag)	0.5	--	--
Thallium (Tl)			
Zinc (Zn)	100	220.0	500.0
Total Organic Carbon (%)			
Total Sulfur			
Acid Volatile Sulfide			
Total Phenols			
Oil and Grease	1500		
pH			
Acenaphthene			
Acenaphthalene			
Anthracene			
Benzo(a)anthracene			
Benzo(b)fluoranthene			
Benzo(k)fluoranthene			
Benzo(ghi)perylene			
Benzo(a)pyrene			
Chrysene			
Dibenzo(ah)anthracene			
Fluoranthene			
Fluorene			
Indeno(1,2,3-cd)pyrene			
Naphthalene			
Phenanthrene			
Pyrene			
Radionuclides			
Total PAHs			
Total PCBs	0.05	2.0	2.0

NOTES:

-- Values not established

**APPENDIX 3**

WASTEWATER TECHNOLOGY CENTRE  
REPORT OF ANALYSIS

PROJECT 896 - Attn: Paul Bucens  
JOB NO. 920113-7

SAMPLE I.D. PARAMETER	ARC-FROTH 1	ARC-FROTH 2	ARC-TAILINGS 1	ARC-TAILINGS 2
<b>METALS</b>				
Al	5173	13546	15155	35658
Sb	<20	<20	<20	<20
As	8.89	18.5	10.4	11.9
Be	<1.2	<1.2	<1.2	<1.2
Cd	<2	<2	<2	<2
Cr	69	197	173	325
Cu	37	83	41	54
Fe	5.4%	13.6%	13.4%	20.1%
Pb	464	1072	520	616
Mg	2245	6290	6351	9634
Mn	776	2313	2235	3349
Ni	6.7	19.5	4.4	6.9
Se	<.001	3.52	0.87	0.89
Ag	2.2	4.0	2.1	3.4
Ti	<2	<2	<2	<2
Zn	2160	5090	2644	3354
TOC %C	24.6	7.06	2.01	3.41
O & G %	1.9	0.7	0.3	0.6
S %	0.612	0.770	0.421	0.517

Note: all results in ug/g, unless stated otherwise.

RESULTS RELEASED:

April 2/92

ANALYZED BY:

AUTHORIZED BY:



WASTEWATER TECHNOLOGY CENTRE  
REPORT OF ANALYSIS

PROJECT 896 – Attn: Paul Bucens  
JOB NO. 920207

PARAMETER	SAMPLE I.D.	CONCENTRATION UG/G			
		FROTH 1	FROTH 2	TRAILINGS 1	TRAILINGS 2
PAH's					
Naphthalene		782	186	44.1	2.95
Acenaphthylene		21.7	7.73	2.41	
Acenaphthene		34.1	44.2	2.10	
Fluorene		76.3	117	7.62	
Phenanthrene		175	233	11.5	
Anthracene		70.8	54.0	2.49	
Fluoranthene		319	234	59.5	
Pyrene		225	165	53.4	
Benzo(a)anthracene		121	81.5	33.8	
Chrysene		137	75.3	15.8	
Benzo(b)fluoranthene		204	96.7	32.9	
Benzo(k)fluoranthene		64.9	31.3	9.23	
Benzo(a)pyrene		128	56.4	20.2	
Indeno(1,2,3-c,d)pyrene		127	52.6	17.1	
Dibenzo(a,h)anthracene		57.4	23.6	8.44	
Benzo(g,h,i)perylene		100	41.7	13.7	
SURROGATES					
% Recoveries					
Naphthalene-d8		*	103	86	99
Acenaphthene-d10		*	101	90	81
Fluorene-d10		*	100	97	90
Phenanthrene-d10		*	102	89	101
Pyrene-d10		*	100	93	91
Chrysene-d12		*	100	201	97

NOTE: \*SURROGATES DATA UNAVAILABLE.

WASTEWATER TECHNOLOGY CENTRE  
REPORT OF ANALYSIS

PROJECT 896 - Attn: Paul Bucens  
JOB NO. 920207

PARAMETER	SAMPLE I.D.	CONCENTRATION UG/G	
		SOIL REF. MAT. (14.II.92)	BLANK (14.II.92)
PAH's			
Naphthalene		46.7	
Acenaphthylene		1.28	
Acenaphthene		0.93	
Fluorene		25.9	
Phenanthrene		14.8	
Anthracene		0.82	
Fluoranthene		27.3	
Pyrene		19.8	
Benzo(a)anthracene		13.7	
Chrysene		15.1	
Benzo(b)fluoranthene		18.8	
Benzo(k)fluoranthene		4.40	
Benzo(a)pyrene		4.74	
Indeno(1,2,3-c,d)pyrene		10.2	
Dibenzo(a,h)anthracene		5.09	
Benzo(g,h,i)perylene		7.36	
SURROGATES			
% Recoveries			
Naphthalene-d8		84	64
Acenaphthene-d10		86	33
Fluorene-d10		81	35
Phenanthrene-d10		102	69
Pyrene-d10		101	60
Chrysene-d12		128	98

RESULTS RELEASED:

27 Feb/92

ANALYZED BY:

*[Signature]*

AUTHORIZED BY:

*[Signature]*

WASTEWATER TECHNOLOGY CENTRE  
REPORT OF ANALYSIS

PROJECT 896 - Attn: Paul Bucens  
JOB NO. 920207-2

SAMPLE I.D.	Wet wt (g)	Dry %	Dry wt (g)	TOTAL PCB'S (ng/g)
Soxhlet BLK	-	-	-	ND
Florisil BLK	-	-	-	ND
Florisil SPK	-	-	-	88 %
FROTH 1	7.3165	61.66	4.5113	5375
FROTH 2	5.4766	71.10	3.8936	4637
TRAILINGS 1	8.0656	75.36	6.0782	1000
TRAILINGS 2	5.3031	99.54	5.2787	108
TRAILINGS 2 DU	5.3885	99.54	5.3637	114
TRAILINGS 2 SP	5.3019	99.54	5.2775	96 %

Florisil SPK - spiked with 625 ng Total PCB's.

Tailings 2 SPK - spiked with 3125 ng Total PCB's

RESULTS RELEASED:	ANALYZED BY:	AUTHORIZED BY:
March 24 / 92	Lewina Subbala	R. Hong / 91