

## FINAL REPORT

**WASTE TREATABILITY STUDY OF  
NORTHERN WOOD PRESERVERS SOIL AND  
THUNDER BAY HARBOUR SEDIMENT  
THUNDER BAY, ONTARIO**

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*prepared for:*

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Environment Canada's Great Lakes Cleanup Fund

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## 1

## INTRODUCTION

ELI Eco Logic International, Inc. (herein referred to as ECO LOGIC) was formed by Dr. Douglas Hallett in 1986. It was formed specifically to address the need for a clean-up technology for one of the most difficult environmental problems, that of severely contaminated aqueous wastes such as harbour sediments, landfill leachates, and lagoon sludges. The goal was to develop a technology that could deal with these watery wastes and also process stored wastes such as contaminated soils, solvents and oils, industrial wastes, obsolete pesticides, and obsolete chemical warfare agents. Other companies and agencies at that time were focusing primarily on incineration as a method for destroying hazardous waste, and were investigating a variety of pre-destruction cleaning or dewatering processes to deal with the problem of aqueous wastes. The process chemistry and equipment designs developed by ECO LOGIC were based on a different set of criteria than other technologies, to allow complete elimination of aqueous and stored wastes in a more timely, cost-effective, and efficient manner.

Development of the ECO LOGIC Process began in 1987, and by 1988, a lab-scale version of the hazardous waste destruction system had been built. A pilot-scale field demonstration unit was built in 1990 with the help of a grant from the Canadian Department of National Defence (DND). It is fully transportable, being mounted on two flatbed trailers. While possessing a much greater capacity than the lab-scale version, it is still four times smaller than a typical commercial-scale system. In 1991, ECO LOGIC completed the first successful demonstration of the pilot-scale system by processing coal-tar-contaminated harbour sediment from Hamilton Harbour, Ontario. Support for that project came from Environment Canada (the Canadian federal environment regulatory body) and the Ontario Ministry of Energy and the Environment (MOEE). A second demonstration of the pilot-scale unit was completed in 1992 in Bay City, Michigan for the US Environmental Protection Agency (USEPA) Superfund Innovative Technology Evaluation (SITE) program. In that demonstration, the waste processed included polychlorinated biphenyl (PCB) oil, and PCB-contaminated landfill soil and groundwater. Support for that project came from DND, MOEE, Environment Canada and the USEPA. ECO LOGIC has recently received preliminary test results from the USEPA, which show that destruction removal efficiencies (DREs) of 99.9999% were achieved.

The lab-scale process unit is located at ECO LOGIC's facility in Rockwood, Ontario. It is capable of processing small quantities of real waste, which makes it useful for determining waste treatability. Since 1992, it has been used extensively for this purpose. This report details tests conducted at lab-scale which were aimed at evaluating the treatability of two hazardous wastes from Thunder Bay.

The report on this study is being submitted to the Contaminated Sediments Treatment Technology Program (COSTTeP), Great Lakes Cleanup Fund for work performed under contract (#3-6017) to the Wastewater Technology Centre (WTC) in Burlington, Ontario. COSTTeP was created in 1991 and is administered by WTC. It is charged with facilitating the development of new technologies capable of safely and cost-effectively removing and/or treating contaminated sediments.

The objective of this study was to quantitatively determine the ability of the ECO LOGIC Process and Thermal Desorption Unit (TDU) to remove and destroy organic contaminants in two PCB wastes from Thunder Bay, Ontario. The first waste is soil from the Northern Wood Preservers' (NWP) site adjacent to Thunder Bay Harbour and the second is Thunder Bay Harbour (TBH) sediment. Samples of both wastes were processed by the lab-scale unit. The processed material from each test was analysed in ECO LOGIC's laboratory to determine the extent of decontamination. All test details and results are presented and discussed in the report. The application of the ECO LOGIC Process to full-scale waste remediation is also discussed.

## 2 TECHNOLOGY DESCRIPTION

### 2.1 The ECO LOGIC Process

Since 1986, ECO LOGIC has conducted research with the aim of developing a new technology for destroying aqueous organic wastes, such as contaminated harbour sediments, landfill soil and leachates, and lagoon sludges. The goal was a commercially-viable chemical process that could deal with these watery wastes and also process stored wastes (e.g. contaminated soils, solvents, oils, industrial wastes, pesticides and chemical warfare agents). Other companies and agencies at that time were focusing their efforts primarily on incineration, and were investigating a variety of pre-destruction cleaning or dewatering processes to deal with the problem of aqueous wastes. The ECO LOGIC Process was developed with a view to avoiding the expense and technical drawbacks of incinerators, while still providing high destruction efficiencies and waste volume capabilities.

A lab-scale process unit was constructed in 1988 and tested extensively. Based on the results of these tests, it was decided to construct a mobile pilot-scale unit that could be used for further testing and ultimately for small commercial waste processing operations. The pilot-scale plant was completed and commissioned in 1991. It was taken through a preliminary round of tests at Hamilton Harbour, Ontario, where the waste processed was coal-tar-contaminated harbour sediment. In 1992, the same unit was taken through a second round of tests as part of the US Environmental Protection Agency (USEPA) Superfund Innovation Technology Evaluation (SITE) program in Bay City, Michigan. This demonstration was partially funded by the DESRT program, the Ontario Environmental Technologies Program and the Defence Industrial Research Program. In the second round of tests, the pilot-scale unit processed PCBs in aqueous, organic and soil matrices. This section describes the process reactions and the pilot-scale process unit, and presents the results of pilot-scale testing thus far. A full-scale process unit is currently being designed and is expected to be constructed and ready for operation by the fall of 1994.

### 2.1.1 Process Chemistry

The process involves the gas-phase reduction of organic compounds by hydrogen at temperatures of 850°C or higher. Chlorinated hydrocarbons, such as PCBs and polychlorinated dibenzo-p-dioxins (PCDDs), are chemically reduced to methane and hydrogen chloride (HCl), while non-chlorinated organic contaminants, such as polyaromatic hydrocarbons (PAHs), are reduced to methane and ethylene. The system product gas consists essentially of hydrogen, methane, ethylene, carbon monoxide, and carbon dioxide. The HCl produced is scrubbed out in a caustic scrubber downstream of the process reactor.

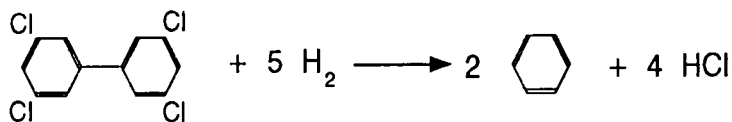
Figure 1 shows some of the reduction reactions, including intermediate steps, for the destruction of a variety of contaminants using the ECO LOGIC Process. Unlike oxidation reactions, the efficiency of these reduction reactions is enhanced by the presence of water, which acts as a reducing agent and a source of hydrogen. The water shift reaction shown produces carbon monoxide and hydrogen from methane and water. Some carbon dioxide is also produced, along with more hydrogen, when carbon monoxide and water react.

A benefit of using an actively reducing hydrogen atmosphere for the destruction of chlorinated organic compounds, such as PCBs, is that no formation of dioxins or furans occurs. Any dioxins or furans in the waste are also destroyed effectively. The reducing hydrogen atmosphere is maintained at more than 50% hydrogen (dry basis) to prevent formation of PAHs. This makes the scrubbed recirculation gas suitable for continuous monitoring using an on-line chemical ionization mass spectrometer (CIMS). By measuring the concentrations of intermediate reduction products, the CIMS produces a continuous indication of destruction efficiency.

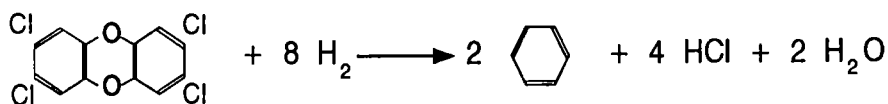
### 2.1.2 The Full-Scale Commercial Process Unit

ECO LOGIC's pilot-scale unit is currently available for small commercial contracts. However, ECO LOGIC has now also designed a full-scale commercial destruction unit. The first such unit is under construction, and will be going into service in the fall of 1994. Construction of additional units will begin as soon as the first unit enters service.

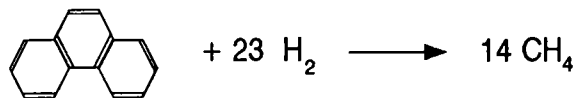
Figure 1 ECO LOGIC PROCESS REACTIONS



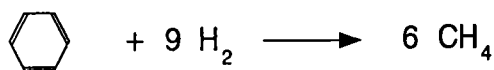
PCB molecule & hydrogen react to produce benzene & hydrogen chloride



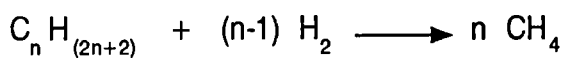
Dioxin molecule & hydrogen react to produce benzene, hydrogen chloride & water



PAH molecule & hydrogen react to produce methane

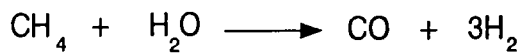


Benzene & hydrogen react to produce methane

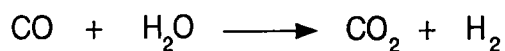


Hydrocarbons & hydrogen react to produce methane

#### WATER SHIFT REACTIONS



Methane & water react to produce carbon monoxide and hydrogen



Carbon monoxide & water react to produce carbon dioxide and hydrogen



The full-scale unit differs from the pilot-scale unit in more than just size. Its waste handling capability is approximately 5 times greater (a nominal capacity of 100 tonnes per day of contaminated soil or sediment). The process control and instrumentation is more advanced. The process gas scrubber has been expanded to clean the gas more completely and permit recovery of the generated hydrogen chloride. A steam reformer has been added that will permit the process to be self-sufficient in hydrogen once waste processing has begun. This section describes the new full-scale destructor unit.

Figure 2 is a schematic of the reactor where the destruction of the waste takes place. The various input streams are injected through several ports mounted tangentially near the top of the reactor. Special nozzles are used to atomize liquid wastes, in order to accelerate liquid vaporization. The gas mixture swirls around a central ceramic-coated steel tube, and is heated by 18 vertical electric heating elements. By the time it reaches the bottom of the reactor, the gas mixture has reached a temperature of at least 850°C. Some particulate initially present in the waste drops out of the reactor bottom and is collected in a grit box. Finer particulate entrained in the gas stream flows up the ceramic tube, into the exit elbow and through the retention zone. The process reactions take place from the bottom of the ceramic tube onwards, and take less than one second to complete.

Figure 3 is a process schematic of the entire full-scale unit, including the reactor. Most of the components of the unit are mounted on standard drop-deck highway trailers. Nitrogen ( $N_2$ ) is used to purge the entire assemblage prior to waste processing and following the discontinuation of processing. Hydrogen ( $H_2$ ) is introduced into the recirculation product gas stream and enters either the recirculation gas heater or the steam reformer. The recirculation gas flowrate varies with the waste type and concentration, up to a maximum of 95% of the product gas. Both the recirculation gas heater and steam reformer preheat the gas stream, but the steam reformer also contains a catalyst to enhance the water shift reaction (see Figure 1). This reaction converts the methane portion of the recirculation gas to CO (and some  $CO_2$ ) and  $H_2$ , the net effect being to recover and reuse the  $H_2$  consumed in the reactor. The gas flow distribution between the recirculation gas heater and steam reformer is a controlled function of the methane fraction in the recirculation gas.

Figure 2 SE25 FULL-SCALE PROCESS REACTOR

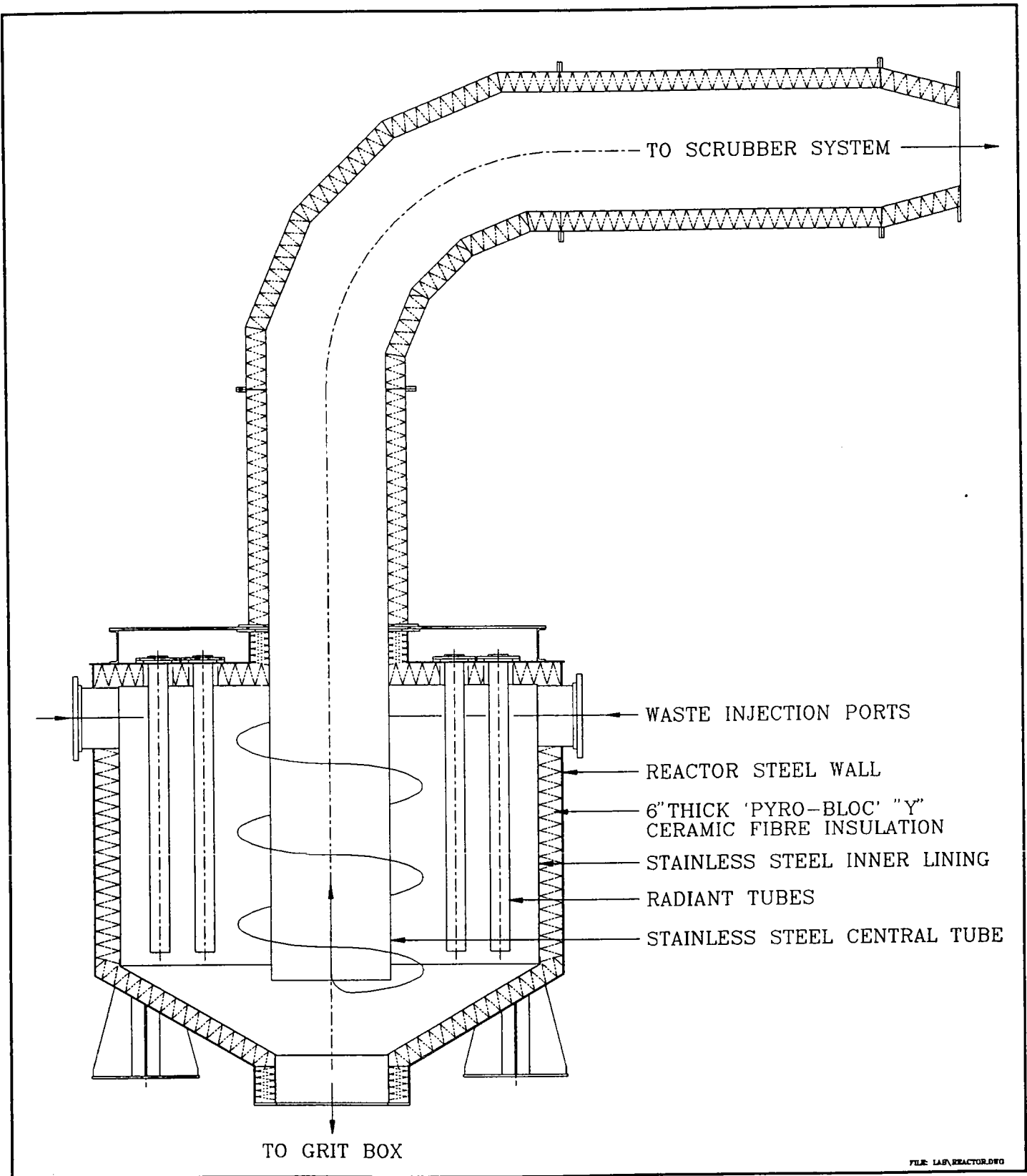
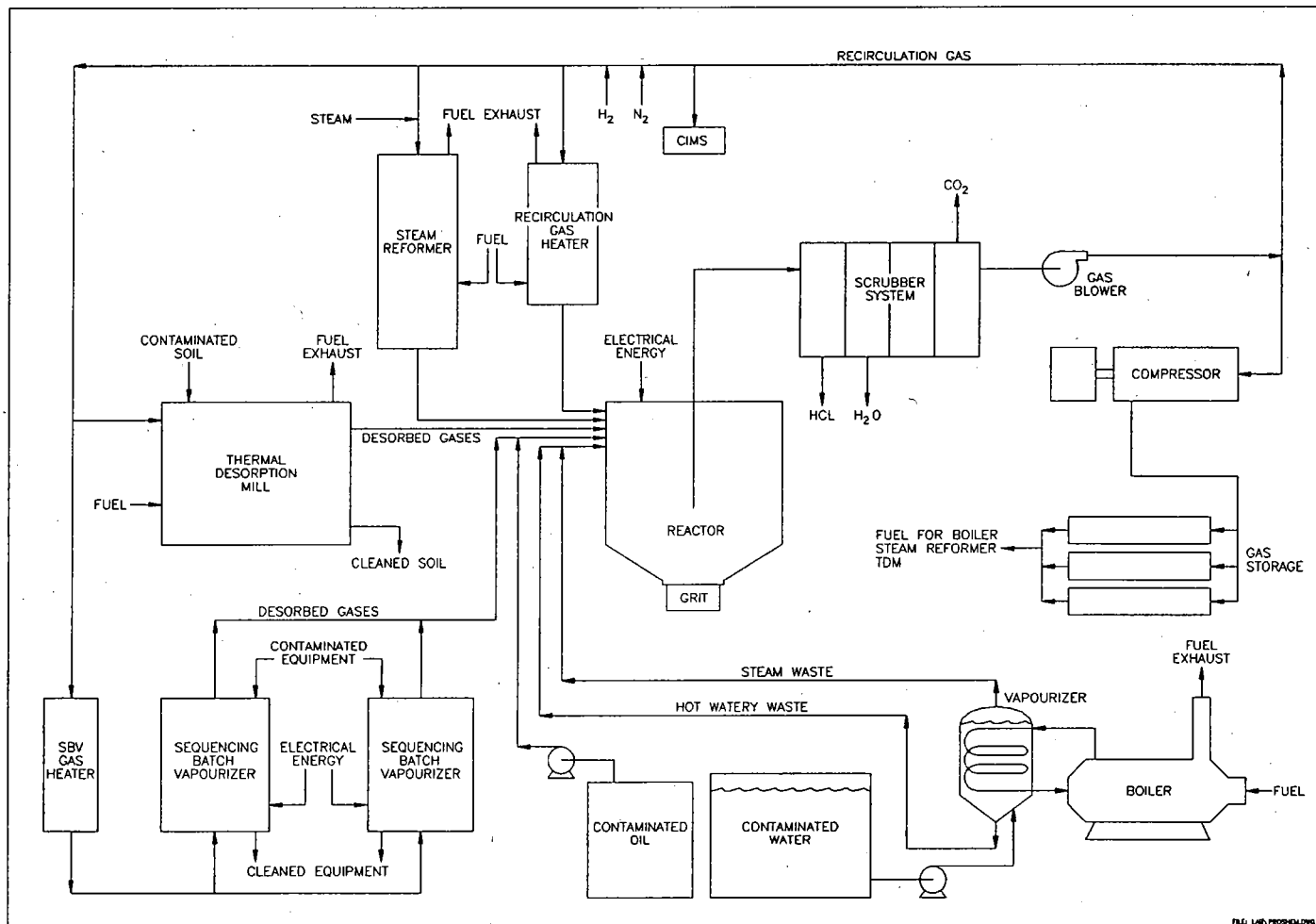


Figure 3 FULL-SCALE PROCESS UNIT SCHEMATIC



Several feed systems are available for various types of wastes, depending on whether watery waste, oil waste, or solid waste is being processed. Watery waste is preheated in a vaporizer using steam from a boiler. The contaminated steam from the vaporizer is metered into the reactor at a rate determined by the process control system. Hot contaminated liquid exits the bottom of the vaporizer at a controlled flowrate and enters the reactor through an atomizing nozzle. Oil waste can be metered directly from drums into the same line using a peristaltic pump.

Solid wastes such as soil or decanted sediment are decontaminated in a thermal desorption unit (TDU). The design of the TDU has been substantially modified, such that it is now referred to as a thermal desorption mill (TDM). The internal workings of the TDM are designed to vaporize all water and organic contaminants in the waste soil/sediment while mechanically grinding the solids. The water vapour and organic contaminants are swept into the reactor by a sidestream of scrubbed recirculation gas. The processed solids are recovered in a water quench tank.

Large contaminated solid objects, such as transformers and electrical equipment, can be thoroughly decontaminated using the sequencing batch vaporizer (SBV) chambers. These chambers take advantage of the reheated recirculation gas stream to heat the equipment and carry contaminants into the reactor. The hydrogen atmosphere is non-reactive with most metals, and there are none of the problems with metal oxide formation associated with rotary kilns.

The SBV can also be used for vaporization of drummed solid chemical wastes, such as hexachlorobenzene. Significant stockpiles of "hex wastes" exist and are still being generated as byproducts of chlorinated solvent production. Advantages of vaporizing hex wastes directly from the drum include decreases in worker exposures and fugitive emissions from drum transfer operations, cleaning of the drums in place, and segregation of inorganic contaminants into the existing drums. The SBV has been tested at lab-scale with hex waste samples and PCB-contaminated electrical equipment.

The product gas leaving the reactor is treated in a multi-leg scrubber system. The first leg is the acid leg, where a series of water sprays quench the hot gas stream exiting the reactor.

The water is collected and recirculated to the sprayers via a series of filters and heat exchangers. The net effect of the acid leg of the scrubber system is to remove water, heat, fine particulates and HCl from the gas stream. The heat exchangers are connected to evaporative coolers for heat rejection. Clean concentrated HCl solution can be recovered for third party use. The collected particulates are removed from the filters periodically, and if necessary fed into the TDM for final processing. The gas exiting the acid leg goes through a weak acid caustic leg. Here another series of water sprays removes residual HCl. The resulting weak acid solution is recovered in a hydraulic seal tank, filtered and recirculated to the sprayers. Another heat exchanger and evaporative cooler removes residual heat, such that by the end of the weak acid caustic leg, the product gas temperature has been cooled to approximately 35°C. The hydraulic seal tank is connected to a surge tank for emergency pressure relief. Just before the process gas exits the weak acid caustic leg, it moves through a packed media bed wetted by a caustic solution spray. A solution of 50% sodium hydroxide (NaOH) is added at a controlled rate to the spray solution to ensure complete removal of HCl from the process gas.

The de-acidified process gas then enters a third scrubber leg where benzene, naphthalene and any other products of incomplete reduction in the gas are scrubbed out. Using a series of heat exchangers, pumps and a stripper vessel, these hydrocarbons are recirculated back to the reactor for complete destruction. A fourth scrubber leg removes from the gas all CO<sub>2</sub>, which has no beneficial effect on the process and occupies useful space in the system. The removed CO<sub>2</sub> is vented to the atmosphere via the boiler stack.

A gas booster fan draws the cooled and scrubbed product gas out of the scrubber system. The gas is now a clean dry mixture of hydrogen, methane and carbon monoxide. As earlier indicated, most of the gas is steam reformed or reheated, and then recirculated back to the reactor. This maintains a high concentration of hydrogen in the reactor. A sidestream is drawn off for on-line sampling. Sidestreams can also go to the TDM and/or SBV as a sweep gas, or to a compressor for storage. Storage of the product gas under pressure permits the analysis of large batches of gas prior to using the gas as fuel and allows the operation of the system in a "stackless" mode. The stored product gas can then be used as fuel, recirculation gas, or sweep gas for the TDM or SBV.

Throughout waste processing operations, the product gas is sampled continuously using the CIMS. This analyser is capable of accurately monitoring up to 10 organic compounds every few seconds at concentrations ranging from percent levels down to ppb levels. It is used as part of the ECO LOGIC Process to monitor the concentrations of certain compounds indicative of the process destruction efficiency. The compounds selected for monitoring depend on the waste being processed. For example, during PCB processing, monochlorobenzene is typically monitored as an indicator of destruction efficiency. Low levels of this volatile compound indicate that destruction of the PCBs is proceeding to completion. An increase in the monochlorobenzene concentration triggers an alarm in the process control system, and the exceedance of a preset threshold is used to automatically curtail waste input. The CIMS also provides a continuous record of the quality of the product gas being compressed and stored.

### 2.1.3 Demonstration Testing of the Pilot-Scale Unit

The pilot-scale process plant was tested for the first time at Hamilton Harbour, Ontario in 1991. The waste processed during those tests was harbour sediment contaminated with coal-tar at concentrations of up to 300 g/kg (dry weight basis). Destruction removal efficiencies (DREs) of 99.9999% were calculated (see Table 1), based on the total organic input and the PAHs analysed in the stack emissions. During one test, the liquid waste input was spiked with PCBs in the air emissions, liquid effluent and processed solids were below the detection limits for each, respectively. Based on the detection limits for the stack sampling trains, a PCB DRE of at least 99.9999% was achieved.

A second round of tests of the pilot-scale unit was conducted in 1992 in Bay City, Michigan as part of the USEPA's SITE program. The wastes processed included oily PCB-contaminated water, high-strength PCB oil, and PCB-contaminated soil. Triplicate test runs were planned for each waste type. The results for the test program, confirmed by the USEPA, are shown in Table 2. The SITE Program Project Bulletins and Technical Evaluation Report for this demonstration are now available.

The waste oil was obtained from beneath the Bay City landfill and was analysed by ECO LOGIC to contain 40% PCBs and percent levels of other chlorinated solvents. The contaminated

**Table 1** HAMILTON HARBOUR PERFORMANCE TEST RESULTS

Run	Target Analytes	Conc.in Waste (mg/kg)	Decant Water Conc. (µg/kg)	Grit Conc. (mg/kg)	Sludge Conc. (mg/kg)	Stack Gas Conc. (µg/m <sup>3</sup> )	DRE (%)
P1	PAHs	21,000	483	1.67	32.8	0.27	99.9999
P2	PAHs	30,000	680	7.76	56.1	0.23	99.9999
P3	PAHs	30,000	423	0.37	4.3	0.14	99.9999
P3	PCBs	500	ND	ND	ND	ND	99.9999

DRE = (Total Input - Stack Emissions) / (Total Input)

ND = Non-Detect

soil was obtained from installation of the sump wells used to collect the oil, and the contaminated water was groundwater from the landfill. The test matrix called for three water/oil tests, three oil tests, and three soil tests.

The water/oil tests were to be nominally 4000 mg/kg PCBs, based on injecting the water and oil in a 100:1 ratio through the atomizing nozzle. As well, perchloroethene was added as a tracer compound. The oil tests were designed to process the high-strength oil at higher throughputs while demonstrating the ability to compress and store the product gas generated. Steam was added through a separate port, but liquid water was not co-injected with the PCB oil. Again, perchloroethene was added as a tracer compound. After oil waste processing, the stored gas was directed to the boiler for about 24 hours, and stack testing by the USEPA sub-contractor was conducted. The target DRE for the PCBs was 99.9999%, and this was achieved for all six tests. The target destruction efficiency (DE) for the perchloroethene was 99.99% and this was also achieved for all six tests. The SITE program analytical results for the input concentrations of the water/oil mixture and the high-strength oil are shown in Table 2.

Soils with various contamination levels were mixed to produce a relatively homogeneous quantity of soil with a nominal 1000 mg/kg PCB concentration. The soil test runs were delayed until construction and commissioning of the new TDU was completed. During the first TDU

Table 2 USEPA SITE PROGRAM RESULTS

<i>Water/Oil and High-Strength Oil Tests</i>					
Run	Waste Type	Contaminant	Concentration (mg/kg)	Target DRE/DE	Achieved
1	Water/Oil	PCBs	4,800	99.9999	Yes
	Tracer	Perchloroethene	4,670	99.99	Yes
2	Water/Oil	PCBs	2,450	99.9999	Yes
	Tracer	Perchloroethene	2,360	99.99	Yes
3	Water/Oil	PCBs	5,950	99.9999	Yes
	Tracer	Perchloroethene	6,100	99.99	Yes
4	Oil	PCBs	254,000	99.9999	Yes
	Tracer	Perchloroethene	33,000	99.99	Yes
5	Oil	PCBs	254,000	99.9999	Yes
	Tracer	Perchloroethene	26,000	99.99	Yes
6	Oil	PCBs	254,000	99.9999	Yes
	Tracer	Perchloroethene	34,000	99.99	Yes
<i>Soil Tests</i>					
Run	Waste Type	Contaminant	Concentration (mg/kg)	Desorption Efficiency (%)	
1	Soil	PCBs	538	94	
	Tracer	HCB	12,400	72	
	Tracer	OCDD	0.744	40	
2	Soil	PCBs	718	99	
	Tracer	HCB	24,800	99.99	
	Tracer	OCDD	1.49	99.8	

test, contaminated soil was processed with a desorption efficiency of 94%, resulting in a processed soil PCB concentration of 30 mg/kg. This result was encouraging for a first run, but the desorbed soil was still above disposal guidelines. The waste soil residence time inside the TDU was increased for the second run, and a desorption removal efficiency of 99% was achieved according to SITE program results. A duplicate sample analysed on site by ECO LOGIC showed a PCB concentration of 0.6 mg/kg, which would correspond to a 99.9% desorption efficiency. The tracer compound used for the soil tests was hexachlorobenzene (HCB), which was spiked at significantly higher concentrations than the PCBs. The hexachlorobenzene was also contaminated with significant levels of octachlorodibenzo-p-dioxin



(OCDD). The desorption efficiencies achieved for the HCB and OCDD for Test 2 were 99.99% and 99.8%, respectively. Due to TSCA permit restrictions, only two runs were performed for the third test condition. It should be noted that the performance of the TDU is independent of the destruction process. The reactor destruction efficiencies for the desorbed contaminants were high for both TDU runs.

An additional component of the test program was a 72-hour endurance test aimed at demonstrating the continuous operation capabilities of the ECO LOGIC Process. The equipment operated perfectly and the 72-hour test was concluded successfully.

#### **2.1.4 Current Status**

The ECO LOGIC Process has been demonstrated to be a high-efficiency alternative to incineration for the destruction of PCB wastes. High water-content wastes and high-strength oils can both be processed with destruction removal efficiencies of at least 99.9999%. The ability to compress and store the product gases generated during processing means that no uncontrolled air emissions occur.

As previously indicated, the full-scale destructor currently under construction will have a nominal throughput capacity of 100 tonnes per day for soils at a cost of approximately \$400 per tonne. This unit will enter service in the fall of 1994 in Perth, Western Australia. A second unit will be under construction by late summer 1994. ECO LOGIC has made proposals to major corporations and government agencies in Canada and the U.S. for the clean-up of contaminated sites.

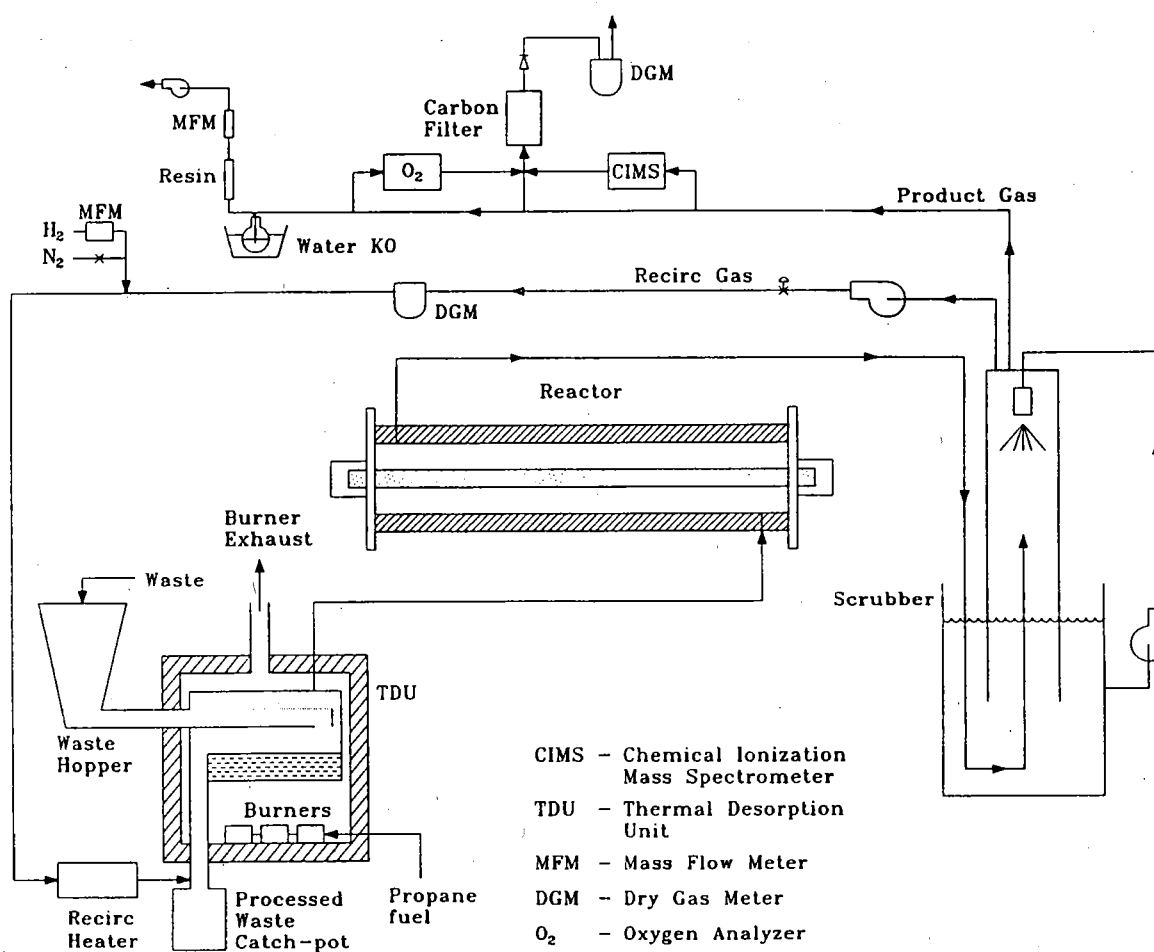
Treatability studies using ECO LOGIC's lab-scale destruction system are continuing. The lab-scale equipment includes a TDM sized for processing 1 - 2 kilograms of soil or sediment, and an SBV suitable for processing samples of chemical wastes or contaminated electrical equipment. Clients find that treatability studies are a cost-effective method for determining the applicability and effectiveness of the ECO LOGIC Process to their waste problems.

## 2.2 Lab-Scale Process Unit

The lab-scale destruction system is designed to mimic the operation of the pilot-scale unit, processing real waste samples to yield the information required to calculate the destruction efficiency of the ECO LOGIC Process. Originally, the pilot-scale destruction system was designed to process watery harbour sediments at a low rate (nominally 1 kg/min). This material was injected directly into the chemical reduction reactor for desorption of the organic contaminants. The decontaminated solids were removed at the end of each test. However, during preliminary testing of ECO LOGIC's pilot-scale unit in 1991, material handling problems were encountered when watery harbour sediment was injected directly into the reactor. The destruction system was subsequently redesigned to include the concept of a thermal desorption unit (TDU), which would remove organic contaminants from the sediment, and send only the vaporized contaminants to the reactor. The TDU allows contaminated soil or sediment waste to be processed at a much higher rate. Two TDUs have been constructed, one each for the lab-scale and pilot-scale destruction units. Figure 4 shows a schematic of the TDU-equipped lab-scale system. The TDU is a sealed vessel containing an atmosphere of hot hydrogen and a bath of molten tin. Contaminated soil or sediment is fed into the TDU by an double-screw mechanism, which exposes the watery waste to the hot hydrogen. As water and volatile organics in the waste are vaporized, the mechanical action of the screw mechanism breaks the dried solids into small chunks and dumps them onto the surface of the tin, which is heated to approximately 600°C. A second screw mechanism slowly pushes the solids across the tin, which heats the solids and vaporizes the semi-volatile organic contaminants. The organic-free solids are then removed from the TDU to a collection vessel for analysis and subsequent disposal. The volatilized organic contaminants are swept by recirculation process gas to the gas-phase chemical reduction reactor.

The reactor contains an atmosphere of hydrogen and is electrically heated to maintain a temperature of 900°C. At this temperature, chlorine is stripped from the chlorinated molecules and the remaining organic molecules are reduced to methane and ethylene. Past experience has proven that the chemical reduction reactions are at least 99.9999% efficient, given a residence time of one second or more.

Figure 4 LAB-SCALE DESTRUCTION SYSTEM AND THERMAL DESORPTION UNIT



In the pilot-scale unit, the process gas exits the reactor and goes to a scrubber where heat, HCl, and water are removed. The methane-rich hydrogen gas exiting the scrubber is then split, with 95% of the process gas recirculated back to the reactor and the other 5% sent to a boiler, where it is burned as a supplementary fuel to produce steam. This system is modelled in the lab-scale process unit. A vertically-mounted plexiglass tube with a single downward-spraying nozzle simulates the pilot-scale scrubber. The tube is partially submerged in a small tank of water to maintain a seal between the process gas inside and the outside environment, and to allow for relief of any over-pressure. The scrubber water is maintained at a temperature of 35°C. It is pumped through the spray nozzle and recirculated to contain any possible contaminants. A small amount of sodium hydroxide is periodically added to the scrubber water to neutralize the HCl. Approximately 50% of the process gas leaving the scrubber is recirculated back to the reactor, and the remaining 50% is vented to atmosphere with a small side-stream drawn through the process gas sampling system. The sampling system includes a water knock-out bottle, and if process gas sampling for PCBs is required, an XAD-2 resin absorption column and a mass flow meter. Another side-stream is drawn into the chemical ionization mass spectrometer (CIMS), which can simultaneously measure the concentrations of up to 10 organic compounds on a real-time basis. The compounds monitored are selected for their ability to indicate the destruction system's performance.

### 3 SAMPLE CHARACTERIZATION

The NWP site soil sample had a medium brown colour and was coarse and sandy. The soil was relatively dry, with a moisture content of 28.4%. The TBH sediment was a blackish silt, possibly containing some clay and possessing a moisture content of 48.3%. It was slippery to the touch, with a consistency like that of a thick paste. Single samples of each waste were collected and analysed for PCBs, EPA 8270 SVOCs, and CPs in ECO LOGIC's laboratory. The results are shown in Table 3. Other than PCBs, the most prominent contaminant is pentachlorophenol (PCP). Both wastes were insignificantly contaminated with a few other CPs and semi-volatile organic compounds (SVOCs). Surrogate recoveries for CPs and SVOCs were generally lower than desirable, suggesting that the levels of CPs and SVOCs may be somewhat higher than indicated. The concentration of PCP in the NWP soil, for example, could possibly be as high as 8.1  $\mu\text{g/g}$  (dry weight).

Table 3 CONTAMINATION LEVELS IN WASTE SAMPLES

(µg/g Dry Weight)	NWP Soil (µg/g)	TBH Sediment (µg/g)	(µg/g Dry Weight)	NWP Soil (µg/g)	TBH Sediment (µg/g)
<b>PCBs</b>			<b>SVOCs (Continued)</b>		
Mono	0.0017	ND(0.005)	Nitrobenzene	ND(0.05)	ND(0.05)
Di	0.019	ND(0.0005)	Isophorone	ND(0.04)	ND(0.04)
Tri	0.35	ND(0.0003)	2-Nitrophenol	ND(0.02)	ND(0.02)
Tetra	1.3	ND(0.005)	2,4-Dimethylphenol	ND(0.04)	ND(0.04)
Penta	0.58	0.0038	Bis(2-chloroethoxy)methane	ND(0.1)	ND(0.1)
Hexa	0.28	0.0039	1,2,4-Trichlorobenzene	0.014	ND(0.004)
Hepta	0.12	ND(0.0004)	Naphthalene	0.32	9.1
Octa	0.043	ND(0.001)	Hexachlorobutadiene	ND(0.008)	ND(0.008)
Nona	ND(0.003)	ND(0.005)	4-Chloro-3-methylphenol	ND(0.02)	ND(0.02)
Deca	ND(0.002)	ND(0.0007)	Hexachlorocyclopentadiene	ND(0.01)	ND(0.01)
Total	2.7	0.0077	2-Chloronaphthalene	ND(0.006)	ND(0.006)
<b>Surrogate Recovery %</b>			Acenaphthylene	0.07	0.15
PCB 14	50	106	Dimethylphthalate	ND(0.08)	ND(0.08)
PCB 65	73	105	2,6-Dinitrotoluene	ND(0.03)	ND(0.03)
PCB 166	1	68	Acenaphthene	0.04	5.8
PCB 204	125	117	2,4-Dinitrophenol	ND(0.03)	ND(0.03)
			2,4-Dinitrotoluene	ND(0.09)	ND(0.09)
<b>CPs</b>			4-Nitrophenol	ND(0.07)	ND(0.07)
2-Chlorophenol	ND(0.0003)	ND(0.0005)	Fluorene	0.05	5.2
2,6-Dichlorophenol	ND(0.0003)	ND(0.0005)	4-chlorophenyl phenyl ether	ND(0.01)	ND(0.01)
2,5-Dichlorophenol	ND(0.0003)	ND(0.0005)	Diethylphthalate	ND(0.008)	ND(0.008)
2,3-Dichlorophenol	0.003	0.0062	4,6-Dinitro-2-methylphenol	ND(0.05)	ND(0.05)
3,5-Dichlorophenol	ND(0.0003)	ND(0.0005)	N-Nitrosodiphenylamine	ND(0.009)	ND(0.009)
3,4-Dichlorophenol	ND(0.0003)	ND(0.0005)	4-bromophenyl phenyl ether	ND(0.01)	ND(0.01)
2,4,6-Trichlorophenol	0.001	0.012	Hexachlorobenzene	ND(0.009)	ND(0.009)
2,3,6-Trichlorophenol	0.0038	ND(0.0005)	Phenanthrene	0.65	22
2,4,5-Trichlorophenol	0.0053	0.011	Anthracene	0.16	3.0
2,3,5-Trichlorophenol	0.0058	0.0054	Di-n-Butylphthalate	ND(0.09)	ND(0.09)
2,3,4-Trichlorophenol	0.0046	ND(0.0005)	Fluoranthene	0.69	17
3,4,5-Trichlorophenol	0.057	0.043	Benzidine	ND(1)	ND(1)
2,3,5,6-Tetrachlorophenol	0.023	0.033	Pyrene	0.68	13
2,3,4,5-Tetrachlorophenol	0.11	0.015	Butylbenzylphthalate	0.14	ND(0.04)
Pentachlorophenol	2.2	0.45	Benzo[a]anthracene	0.32	4.4
<b>Surrogate Recovery %</b>			3,3'-Dichlorobenzidine	ND(0.06)	ND(0.06)
2,4,6-Tribromophenol	27	75	Chrysene	0.55	3.9
			Bis(2-ethylhexyl)phthalate	0.51	0.54
<b>SVOCs</b>			Di-n-octylphthalate	0.14	0.14
Phenol	ND(0.01)	ND(0.01)	Benzo[b]fluoranthene	0.60	4.8
Bis(2-chloroethyl)ether	ND(0.006)	ND(0.006)	Benzo[k]fluoranthene	0.55	4.4
1,3-Dichlorobenzene	ND(0.003)	ND(0.003)	Benzo[a]pyrene	0.48	4.5
1,4-dichlorobenzene	0.004	0.007	Indeno(1,2,3-c,d)pyrene	0.48	2.6
1,2-dichlorobenzene	ND(0.003)	ND(0.003)	Dibenzo[a,h]anthracene	ND(0.006)	0.42
Bis(2-chloroisopropyl)ether	ND(0.002)	ND(0.002)	Benzo[g,h,i]perylene	0.48	2.4
Hexachloroethane	ND(0.1)	ND(0.1)	<b>Surrogate Recovery (%)</b>		
N-Nitrosodi-n-propyl amine	ND(0.2)	ND(0.2)	Phenol-d6	21	18
			2,4,6-Tribromophenol	29	38
			Nitrobenzene-d5	30	30
			2-Fluorobiphenyl	47	48
			Terphenyl-d14	50	59

I = INTERFERENCE; ND = NOT DETECTED (METHOD DETECTION LIMIT IN BRACKETS)

## 4 SCOPE OF WORK

### 4.1 Waste Preparation

Each waste sample was prepared for processing by mechanically sifting it through 1/4" screen to remove stones, clamshells, and other solid objects that could disrupt operation of the material handling mechanisms inside the TDU. The sifted waste was then added directly to the TDU's hopper in preparation for processing.

### 4.2 Waste Processing

The process unit took approximately two hours to reach steady-state at the desired conditions. Waste processing was not initiated until the molten tin temperature in the TDU had reached 600°C and the reactor temperature was in the range 850-900°C. Using thermocouples, the temperatures at several locations within the system were monitored. Thermocouple locations included the TDU tin, inner TDU atmosphere, reactor inlet, reactor outlet, scrubber water, and recirculation gas heater. The reactor pressure was also monitored. The O<sub>2</sub> volume fraction of the system gas was kept below 0.4%, which is well outside H<sub>2</sub>-O<sub>2</sub> combustion limits. All these parameters were recorded every half hour. Test #1 took 90 minutes, with about 4 kg of NWP soil waste processed during that time. Test #2 took 120 minutes with about 8 kg of Thunder Bay Harbour sediment processed during that time.

Single samples of the processed material and scrubber water were collected in amber jars with teflon-lined lids. Samples of each raw waste were also collected. The process gas leaving the scrubber was not sampled in this study. All samples were analysed for PCBs, CPs, and SVOCs. Concentrations of the target compounds were estimated by taking weighed portions of each collected sample through soxhlet extractions and clean-ups, and analysing the extracted samples in two gas chromatographs, one equipped with a mass selective detector (GC-MSD), the other with an electron capture detector (GC-ECD). Section 5 details the sampling and analysis methodologies employed. Appendix A details quality assurance methodologies employed. The percent desorption of PCBs and CPs was calculated from the mass of waste

processed, the mass of processed material recovered, and the contaminant concentrations in each.

Subsequent to the completion of Tests #1 and #2, it was decided to modify the TDU design. The intermeshed screws that drive the waste solids across the tin bath would be replaced by a ball mill containing steel grinding balls. The mill would rotate and float on the bath. The waste feed mechanism would dump waste directly inside one end of the mill. The processed solids would exit the other end of the mill and be recovered in a quench tank or catch-pot. In this way, the waste solids would be ground into fine material, thereby maximizing particle surface area, and contact between molten tin and waste would be prevented.

The lab-scale TDU was modified to reflect the desired design changes, with one exception. The waste feed mechanism was removed and the corresponding port for waste entry was sealed up. The geometry of the existing lab-scale TDU forced this change. Full-scale ball mills normally have an aspect ratio (length:diameter) of 5 or higher, to prevent raw input material from rapidly working its way down the length of the mill. However, in order to fit the largest possible ball mill inside the existing lab-scale TDU, the mill had to have an aspect ratio of approximately 1. (A smaller mill would have been impractical, given the scale of the TDU.) For such a mill, some input waste could travel rapidly to the output end of the mill and be inadequately desorbed.

It was decided therefore to have the new TDU work in batch mode, rather than continuous feed mode. The waste sample to be test-processed is loaded in a sealable plastic bag and wrapped in a layer of aluminum foil to prevent spillage and placed inside the ball mill beforehand. The processed solids are recovered from the mill following each test. The name of the entire unit has been changed to Thermal Desorption Mill (TDM), to reflect the change in design.

To investigate the effect of these modifications on treatability of the Thunder Bay wastes, five test runs with the TDM were conducted. These runs were conducted at ECO LOGIC's expense. The test parameters were as shown in Table 4.



Each of the test runs listed in Table 4 were similar to the two runs conducted with the old TDU design, with two notable exceptions. The wastes in Tests #3 to #7 was spiked with PCB oil to a nominal level of 1000  $\mu\text{g/g}$ , whereas the wastes in Tests #1 and #2 possessed PCB concentrations of 2.7  $\mu\text{g/g}$  and 0.0077  $\mu\text{g/g}$ , respectively. The PCB desorption load was therefore much greater in Tests #3 to #7. The tests were also shorter in duration. Once the temperature of the tin bath reached 600°C, ball mill operation was begun, and continued for the interval indicated in Table 4. At the end of this interval, ball mill rotation was discontinued and the test was considered terminated.

**Table 4** TEST PARAMETERS

Test	Waste Source	Mass of Waste Charge (kg)	Ball Mill Run Time (min.)	Ball Mill Speed (RPM)	Tin Bath Temp. (°C)
#3	NWP soil	0.45	10	12	600
#4	NWP soil	0.45	20	12	600
#5	TBH sediment	0.45	10	12	600
#6	TBH sediment	1.0	20	12	600
#7	TBH sediment	0.9	20	12	500

## 5 SAMPLING/ANALYTICAL METHODOLOGIES

ECO LOGIC will conduct all of the organic laboratory analyses. Metals analyses will be sub-contracted to an accredited inorganic laboratory. This section describes the standard analytical methods which will be used during the test program for the measurement of interest. Only USEPA and ASTM methodology will be followed in this program.

### 5.1 Proofing and Preparing

#### 5.1.1 Glassware

All glassware and utensils coming into contact with the samples are washed thoroughly with an alkaline cleaning solution and water, rinsed first with water and then with deionized reverse osmosis (organic free) water. They are then solvent rinsed with acetone to remove water and any organics, then rinsed with the extracting solvent (hexane, dichloromethane) to remove additional organics. This process is also done with sampling containers and lids that will be used in the field. All lids must be Teflon-coated. Autosampler vials are rinsed prior to extract introduction. Bottle caps and hypo vial discs undergo the same solvent rinsing technique.

#### 5.1.2 Reagents

Reagents such as silica-gel, florisil, alumina and sodium sulphate undergo heating in an oven or muffle furnace to activate and to remove organics. All solvents used for extractions are of distilled in glass purity. Acids and bases used for pH adjustment of liquid samples are extracted with solvent to remove impurities before use. All reagents undergo solvent extraction and instrumental analysis to prove they are free of contamination before use.

#### 5.1.3 Sample Extraction - Organics

Sample extraction follows the methodology outlined in EPA methods 3510 and 3540 for liquids and solids respectively. Extractions are outlined in greater detail below.

#### 5.1.4 Solid Waste Extraction

Approximately 20 g of the ground and homogenized solid waste sample will be accurately weighed, spiked with the appropriate surrogates for analysis, as listed at the bottom of Table 6 mixed with anhydrous sodium sulphate (10 g) and placed in a soxhlet thimble. The soil will then be soxhlet extracted overnight (16 hours) with 300 mL of 50:50 acetone/hexane. A portion of the sample will be subsampled for dry weight determination according to Section 5.5. The solvent extracts will be combined and dried over powdered sodium sulphate, then concentrated to 10 mL using a Kuderna-Danish evaporator. Exactly 5 mL of the 10 mL concentrate was removed, diluted to 300 mL with R.O. water, and acetylated according to the procedure in Section 5.1.5.2 for chlorophenols analysis. The remaining 5 mL was concentrated to one mL, one half mL portion of which was used for semivolatiles analysis without further clean-up. The other 1/2 mL portion was cleaned-up by acid silica gel chromatography for analysis for PCBs. Refer to EPA Method 3540 for greater detail.

#### 5.1.5 Aqueous Samples

##### 5.1.5.1 Semivolatile Organics

The volume of the one litre aqueous sample was accurately determined and the sample placed in a two litre separatory funnel. The sample was then spiked with acid, base-neutral, and PCB surrogates, and the pH adjusted to  $> 11$  with sodium hydroxide solution (10 M). The sample was extracted three times with 100 mL DCM, the pH adjusted to  $< 2$  with sulphuric acid (9 M), and again extracted three times with DCM. The extracts were combined, dried over sodium sulphate, and concentrated to 1 mL using a Kuderna-Danish evaporator. Exactly half of the concentrate was analysed for semivolatiles (EPA Method 8270), and the remaining extract was cleaned-up by acid silica gel chromatography for analysis for PCBs.

##### 5.1.5.2 Chlorophenols

100 mL of aqueous sample was diluted to 300 mL, placed in a two litre separatory funnel, and spiked with chlorophenol surrogate. To this was added 7.8 mL of potassium

carbonate solution (4.34 M) with mixing. In situ acetylation was carried out with the addition of 9 mL of triple distilled acetic anhydride. The sample was shaken for five minutes and allowed to stand for an additional five minutes. The derivitized chlorophenols were extracted from the aqueous solution with 3 aliquots of 40 mL hexane and the extracts combined. The combined extracts were then dried over sodium sulphate, concentrated to 1/2 mL and analysed by GC/MS.

## 5.2 Organics Fractionation

The extracts prepared as described above will be subjected to open column chromatographic clean-up prior to instrumental analysis by GC/MS. These clean-ups are necessary to ensure that the sample can be sufficiently concentrated to achieve the desired detection limits and reduce the level of background interferences.

### 5.2.1 Acid Silica Gel Chromatography

Samples for PCB analysis were cleaned up on a column made up as follows: A plug of silanized glass wool was placed in a large volume pipette and the column rinsed with hexane. The column was then packed with 2 cm of 5% deactivated florisil, 4 cm of acid silica gel, and 1 cm of sodium sulphate. The sample was added to the top of the column and eluted with 10 mL of hexane. The eluent was concentrated to 100  $\mu$ L and analysed for PCBs by GC/MS.

## 5.3 Instrumental Analysis

The designated extracts were analysed for PCBs, semivolatile organics, and chlorophenols using a Hewlett Packard 5890 Series II Gas Chromatograph (GC) equipped with a model 5971A Mass Selective Detector (MSD). The GC is equipped with a 30 m DB-5 (J&W Scientific) column having a 0.25 mm ID and 0.25  $\mu$ m film thickness. For the EPA Method 8270 semivolatiles, a full scan monitoring of ions with mass-to-charge ratio of 35 to 500 amu was employed. A 2  $\mu$ L sample was injected through a splitless injector with a purge delay of 1 min. For chlorophenols, the same injection system was used. The MSD was operated in the selective ion monitoring (SIM) mode which monitors only for the ions characteristic of acetylated

chlorophenols. PCBs were analysed by a 1  $\mu$ L cool on-column injection with selected ion monitoring operation of the MSD. Refer to Table 5 reference for GC/MSD conditions.

**Table 5** GAS CHROMATOGRAPH CONDITIONS

	Chlorophenols	Semivolatiles	PCBs
Injector Temp.	280°C	280°C	90°C
Detector Temp.	260°C	260°C	310°C
Initial Oven Temp.	35°C	35°C	90°C
Initial Hold	2.5 min.	2.5 min.	2 min.
Ramp A	5°C/min	5°C/min	20°C/min
Final Temp. A	60°C	60°C	150°C
Hold A	0 min.	0 min.	2 min.
Ramp B	5°C/min	5°C/min	10°C
Final temp. B	200°C	200°C	310°C
Hold B	0 min.	0 min.	2 min.
Ramp C	10/min°	10/min°	-
Final Temp. C	270°C	270°C	-
Hold C	17.5 min	17.5 min	-
Total Time	60 min.	60 min.	25 min.

## 5.4 Identification and Calculation

### 5.4.1 Peak Identification and Qualification

Data files generated from instrumental analysis are integrated and quantitated using the provided software. The experience of the analyst is also essential, and in many instances, manual integration and quantitation is used to override the computer in the estimation of maximal peak response. In order for a peak to qualify as a positive detection of target analyte, the following criteria must be met.

- a) The peak response of the quantitation and confirmation ion must be greater than 3 times the background noise.
- b) The peak area ratio of the confirmation to quantitation ion must be within  $\pm 20\%$  (absolute) of the ratios observed within the calibration standards and elute simultaneously  $\pm 0.02$  minutes.
- c) The observed retention times within the samples should not vary more than  $\pm 0.05$  minutes relative to the standards.
- d) The peak response of all internal standards must be observed.

#### 5.4.2 Determination of Response Factors

The response factors (RF) for each target compound are determined by tabulating the area response of the characteristics ions against concentration and the concentration of the internal standard. The internal standard selected for the calculation of the RF for a compound is the internal standard that has a retention time closest to the compound being measured. The response factor is calculated according to equation (1).

$$RF = \frac{(A_x C_{is})}{(A_{is} C_x)} \quad (1)$$

where:

- $A_x$  = Area of the characteristic ion for the compound being measured.
- $A_{is}$  = Area of the characteristic ion for the specific internal standard.
- $C_{is}$  = Concentration of the specific internal standard.
- $C_x$  = Concentration of the compound being measured.

#### 5.4.3 Calculation for Analyte Concentration

When a compound has been identified, the quantification of that compound is based on the integrated abundance determined from the ion chromatogram of the primary characteristic

ion. The internal standard used is the one nearest in retention time to the target analyte. The concentration of each identified analyte in the sample is calculated from Equation (2).

$$\text{Concentration (ng/g)} = \frac{(A_x) (I_s) (V_t)}{(A_{is}) (RF) (V_i) (W_s)} \quad (2)$$

where:

- $A_x$  = Area of characteristic ion for compound being measured.
- $I_s$  = Amount of internal standard injected (ng).
- $V_t$  = Volume of total extract ( $\mu\text{L}$ ).
- $A_{is}$  = Area of characteristic ion for the internal standard.
- $RF$  = Response factor for the compound being measured (see Section 5.4.2).
- $V_i$  = Volume of extract injected ( $\mu\text{L}$ ).
- $W_s$  = Weight or volume of sample extracted or purged (wet weight, g).

## 5.5 Dry Weight Determinations

A portion of each solid waste sample will be placed in a pre-weighed weighing dish and dried in an oven at  $130^\circ\text{C}$  overnight. The moisture content is then calculated as a percentage of whole sample mass according to equation (3).

$$\% \text{ moisture} = \frac{(W_w - W_d)}{W_w} \times 100 \quad (3)$$

where:

- $W_w$  = Wet weight of subsample
- $W_d$  = Dry weight of subsample

## 6 RESULTS

Appendix B contains the results of all lab analyses conducted for Tests #1 and #2. These results include concentration data for blanks as well as the collected samples. Appendix B also contains the system start-up and shut-down logs, as well as the operator logs recorded during testing.

Both Tests #1 and #2 ran smoothly. In the first run, the NWP soil, being relatively dry and sandy, tended to pack easily. Once loaded into the TDU hopper, the soil had to be manually compressed and prodded repeatedly or else the waste feed screw mechanism could not feed it into the TDU. The Harbour sediment, on the other hand, being more moist and higher in silt and clay content, fed quite easily. Both test runs otherwise took place without notable incident.

The processed solids recovered in the TDU catch-pot were mostly inorganic agglomerates roughly 1 cm in size. This is equivalent to the spacing of the flights of the screw mechanisms that drive the waste into the TDU and transport it across the surface of the tin bath. A small amount of tin was collected in the catch-pot along with the processed solids. In each test however, the tin amounted to less than 2% of the total mass of material collected. This is a much lower fraction than has been found in previous treatability tests, and possibly due to the fact that the level of tin with respect to the sides of the bath was much lower than in previous tests. It was therefore more difficult for the tin to work its way over the physical threshold separating the bath from the catch-pot.

Table 6 and Table 7 summarize the lab results for Tests #1 and #2, respectively. They present the contaminant concentrations of PCP, Total CPs and any SVOCs that were above method detection limit. Table 8 presents the percent desorption of PCBs, CPs and EPA 8270 PAHs from the NWP soil and TBH sediment waste samples in Tests #1 and #2, respectively.

Table 6, Table 7 and Table 8 show that the desorption of both PCBs and CPs was high. Concentrations were reduced to ng/g levels. (The percent desorption of PCBs could not be estimated for Test #2 because the concentration present in the TBH waste was already down to



Table 6 RESULTS SUMMARY - TEST #1 - NWP SOIL

	Raw Waste ( $\mu\text{g/g}$ )	Processed Solids ( $\mu\text{g/g}$ )	Pre-Run Scrubber ( $\mu\text{g/L}$ )	Post-run Scrubber ( $\mu\text{g/L}$ )
Contaminant	Result	Result	Result	Result
<u>PCBs</u>				
Total PCBs	2.7	0.0025	5.9	0.66
<u>Surrogate Recoveries (%)</u>				
PCB 14	50	93	97	74
PCB 65	1	83	85	73
PCB 166	73	79	87	71
PCB 204	125	92	138	110
<u>CPs</u>				
Pentachlorophenol	2.2	0.0024	ND(0.03)	ND(0.03)
Total CPs	2.41	0.0043	0.04	0.10
<u>Surrogate Recovery (%)</u>				
2,4,6-Tribromophenol	27	85	69	61
<u>SVOCs</u>				
Phenol	ND(0.01)	0.75	ND(0.2)	ND(0.2)
1,4-dichlorobenzene	0.004	ND(0.003)	0.32	0.21
Isophorone	ND(0.04)	0.19	ND(0.03)	ND(0.03)
1,2,4-Trichlorobenzene	0.014	ND(0.002)	ND(0.09)	ND(0.09)
Naphthalene	0.32	0.94	260	2000
Acenaphthylene	0.07	ND(0.005)	130	540
Fluorene	0.05	0.013	28	280
Diethylphthalate	ND(0.008)	ND(0.01)	0.3	0.4
Phenanthrene	0.65	0.034	190	890
Anthracene	0.16	0.012	14	170
Di-n-Butylphthalate	ND(0.09)	0.030	4.8	4.2
Fluoranthene	0.69	0.011	96	630
Pyrene	0.68	0.014	55	340
Butylbenzylphthalate	0.14	ND(0.01)	ND(0.3)	ND(0.3)
Benzo[a]anthracene	0.32	0.014	9.9	69
Chrysene	0.55	0.018	19	120
Bis(2-ethylhexyl)phthalate	0.51	0.13	39	13
Di-n-octylphthalate	0.14	ND(0.1)	3.4	1.9
Benzo[b]fluoranthene	0.60	ND(0.07)	70	260
Benzo[k]fluoranthene	0.55	ND(0.08)	21	35
Benzo[a]pyrene	0.48	0.012	36	100
Indeno(1,2,3-c,d)pyrene	0.48	ND(0.006)	15	58
Dibenzo[a,h]anthracene	ND(0.006)	ND(0.004)	ND(1)	6
Benzo[g,h,i]perylene	0.48	ND(0.01)	12	39
<u>Surrogate Recovery %</u>				
Phenol-d6	21	36	12	12
2,4,6-Tribromophenol	29	48	46	48
Nitrobenzene-d5	30	35	40	38
2-Fluorobiphenyl	47	49	46	44
Terphenyl-d14	50	62	56	58

I = INTERFERENCE; ND = NOT DETECTED (METHOD DETECTION LIMIT IN BRACKETS)

Table 7 RESULTS SUMMARY - TEST #2 - TBH SEDIMENT

	Raw Waste ( $\mu\text{g/g}$ )	Processed Solids ( $\mu\text{g/g}$ )	Pre-Run Scrubber ( $\mu\text{g/L}$ )	Post-Run Scrubber ( $\mu\text{g/L}$ )
Contaminant	Result	Result	Result	Result
<u>PCBs</u>				
Total PCBs	0.0077	0.0096	5.9	0.66
<u>Surrogate Recoveries (%)</u>				
PCB 14	106	96	97	74
PCB 65	105	86	85	73
PCB 166	68	85	87	71
PCB 204	117	99	138	110
<u>CPs</u>				
Pentachlorophenol	0.45	0.0026	0.06	0.1
Total CPs	0.58	0.0096	0.29	0.35
<u>Surrogate Recovery (%)</u>				
2,4,6-Tribromophenol	75	14	64	69
<u>SVOCs</u>				
Phenol	ND(0.01)	ND(0.002)	ND(0.2)	140
1,4-dichlorobenzene	0.007	0.007	0.19	0.18
Naphthalene	9.1	1.9	730	1800
Acenaphthylene	0.15	0.008	270	460
Acenaphthene	5.8	0.04	51	160
Fluorene	5.2	0.016	84	490
Diethylphthalate	ND(0.008)	ND(0.01)	0.5	0.4
Phenanthrene	22	0.13	360	890
Anthracene	3.0	0.016	40	180
Di-n-Butylphthalate	ND(0.09)	0.014	3.7	2.7
Fluoranthene	17	0.073	190	450
Pyrene	13	0.035	120	260
Butylbenzylphthalate	ND(0.04)	ND(0.01)	ND(0.3)	100
Benzo[a]anthracene	4.4	0.010	23	6.5
Chrysene	3.9	0.013	39	97
Bis(2-ethylhexylphthalate)	0.54	0.2	11	20
Di-n-octylphthalate	0.14	ND(0.1)	1.1	5.5
Benzo[b]fluoranthene	4.8	ND(0.07)	87	72
Benzo[k]fluoranthene	4.4	ND(0.08)	12	110
Benzo[a]pyrene	4.5	ND(0.09)	38	36
Indeno(1,2,3-c,d)pyrene	2.6	ND(0.006)	28	28
Dibenzo[a,h]anthracene	0.42	ND(0.004)	2	4
Benzo[g,h,i]perylene	2.4	ND(0.01)	15	22
<u>Surrogate Recovery (%)</u>				
Phenol-d6	18	31	13	20
2,4,6-Tribromophenol	38	58	51	60
Nitrobenzene-d5	30	39	38	51
2-Fluorobiphenyl	48	49	43	56
Terphenyl-d14	59	68	54	61

ND = NOT DETECTED (METHOD DETECTION LIMIT IN BRACKETS)

**Table 8** PERCENT DESORPTION OF CONTAMINANTS IN WASTE SAMPLES

	Test #1	Test #2
Contaminant	NWP Soil	TBH Sediment
Total PCBs	99.91 %	NA (Concentration in waste already at ng/g level)
EPA 8270 CPs	99.86 %	98.58 %
EPA 8270 PAHs	82.4 %	97.8 %

the ng/g level.) The Canadian Council of Ministers of the Environment (CCME) Interim Remediation Criteria for PCBs in soil are 50, 5, and 0.5  $\mu\text{g/g}$  for commercial/industrial, residential, and agricultural/parkland, respectively. The corresponding CCME criteria for CPs are 10, 1, and 0.05  $\mu\text{g/g}$ , respectively. Thus for Test #1, the TDU has reduced the concentrations of PCBs and CPs in the NWP soil waste to well below the strictest of the applicable criteria (in the case of PCBs, two orders of magnitude below the criterion). The concentrations in the TBH waste in Test #2 were already below the applicable criteria before processing. Recovery of CP and PAH surrogates were generally low. The levels of CPs and PAHs in both waste and processed solids may therefore be somewhat higher than shown in Table 6 and Table 7, and the figures for EPA 8270 CPs and EPA 8270 PAHs in Table 8 have a relatively high uncertainty.

DRE estimates cannot be made from the data collected, since the process gas was not sampled during either test. However, some idea of the reactor's performance can be gained from the scrubber water results. For example, if the concentration of a major waste contaminant rises substantially in the scrubber water during waste processing, then the reactor has not been adequately destroying that contaminant. It can be seen from Table 6 and Table 7 that PCB and CP concentrations in the scrubber water changed insignificantly during Test #1 and #2, indicating virtually complete destruction of desorbed PCBs and CPs. Concentrations of several PAHs (most notably naphthalene) in the scrubber water increased during testing. Small PAH

molecules, such as naphthalene, are inevitably formed during the ECO LOGIC Process, as they are products of incomplete destruction of larger organic molecules. These small PAHs are commonly absorbed in the scrubber water. Levels of other SVOCs in the scrubber water were not substantially changed during either test. Overall the scrubber water results indicate that the reactor destroyed desorbed contaminants quite adequately and normally.

Following the completion of Tests #1 and #2, a review of the TDU design was conducted, with a view to improving desorption performance and solving the problem of molten tin being included with the collected solids. Consequently, the TDU design modifications described in section 4.2 were conceived and implemented. Detailed results for the five test runs conducted using the TDM are presented in Appendix C.

Table 9 summarizes the results for Tests #3 to #7. Table 10 shows the percent desorption of Total PCBs and EPA 8270 PAHs for the same tests. (Due to lab equipment problems, no analyses for CPs were possible.) PCB concentrations were reduced to two orders

**Table 9 RESULTS SUMMARY - TESTING OF NEW THERMAL DESORPTION UNIT**

	Test #3 NWP Soil		Test #4 NWP Soil		Test #5 TBH Sediment		Test #6 TBH Sediment		Test #7 TBH Sediment
(Concentrations in µg/g)	Waste	Processed Solids	Waste	Processed Solids	Waste	Processed Solids	Waste	Processed Solids	Processed Solids
Total PCBs	440	0.0039	520	0.016	710	0.028	790	0.0097	0.065
<u>Surrogate Recoveries (%)</u>									
PCB 14	68	81	80	72	81	63	82	76	43
PCB 65	62	70	76	60	74	59	73	72	50
PCB 166	89	76	130	63	69	77	75	90	86
PCB 204	81	77	112	67	79	90	90	102	119
Total EPA 8270 PAHs	274.5	2.80	257.6	0.172	137.9	0.077	117.2	0.010	0.333
<u>Surrogate Recoveries (%)</u>									
Anthracene-d10	80	80	32	83	82	52	76	68	104
Benzo(a)anthra cene-d12	104	92	96	106	92	64	86	72	124

**Table 10** PERCENT DESORPTION OF CONTAMINANTS IN WASTE SAMPLES USING NEW TDM

	Test #3	Test #4	Test #5	Test #6	Test #7
Contaminant	NWP Soil	NWP Soil	TBH Sediment	TBH Sediment	TBH Sediment
Total PCBs	99.9991%	99.9997%	99.996%	99.9988%	99.991%
EPA 8270 PAHs	98.98%	99.93%	99.94%	99.991%	99.74%

of magnitude below the strictest of the CCME criteria ( $0.5 \mu\text{g/g}$ ). This might not seem like an improvement in desorption performance compared with Tests #1 and #2 were it not for the fact that the input wastes in Tests #3 to #7 were spiked to the high levels indicated in Table 9. Table 10 shows that the TDM is capable of 5 nine's, i.e. 99.999% desorption of PCBs. The results in Table 9 and Table 10 also show significantly reduced PAH levels in the processed material. PAHs were reduced by 99% or better in all five tests, which is a significant improvement compared with previous treatability test results.

Test #7 was conducted with a tin bath temperature of  $500^{\circ}\text{C}$ , as opposed to  $600^{\circ}\text{C}$  for all the other tests. Even so, the concentration of Total PCBs in the processed solids was still well below the strictest CCME criteria of  $0.5 \mu\text{g/g}$ . Percent desorption was still above 99.99% for PCBs, though below 99.99% for PAHs. This indicates that the tin temperature can be dropped as low as  $500^{\circ}\text{C}$  without major consequences on desorption performance. (The same waste was processed in Test #7 as in Tests #5 and #6, hence no waste contaminant concentrations are shown for Test #7.) The ball mill operation interval was 10 minutes for Tests #3 and #5, and 20 minutes for Tests #4, #6 and #7. There is no dramatic difference in the test results that might be explained by the difference in ball mill run times, suggesting that a 10-minute waste residence time in the mill should be adequate for complete desorption. This is

excellent compared with the residence intervals required by other technologies (typically 15 to 40 minutes).

## 7 DISCUSSION OF RESULTS

The overall results from this treatability study are excellent. Both PCBs and CPs in the Northern Wood Preservers' soil and Thunder Bay Harbour sediment were well desorbed by the TDU. Results from supplementary tests using spiked samples of the same wastes in the new TDM were even better. The TDM clearly is capable of desorbing PCBs and CPs to well below CCME and TSCA criteria, typically by two orders of magnitude. Combined with the rest of the ECO LOGIC process system, which can deliver 6 nine's of PCB destruction, the TDM can efficiently remediate PCB-contaminated soil or sediment, using a process that does not have the technical problems or high cost of incineration.

The projected cost for soil or sediment remediation using the full-scale process unit is \$400 per tonne of waste. This cost will not be affected significantly by the waste moisture content. The ECO LOGIC Process is enhanced, not impeded by the presence of water. High-moisture wastes, therefore, while increasing the heating load needed for complete waste desorption, reduce the demand for energy and hydrogen in the reactor. The net result on waste processing cost is minimal. Extremely high-moisture wastes (i.e. > > 50% moisture) may actually be dewatered before processing to reduce the mass of TDM waste feed material, with the excess water being processed directly by the reactor. Future sediment remediation contracts will employ full-scale TDMs. The pilot-scale process unit, while useful for future process demonstrations and small remediation contracts involving high-strength wastes, is not commercially viable for low-strength wastes like contaminated sediments.

## 8

## CONCLUSIONS FROM LAB-SCALE TESTING

- (1) Desorption of contaminants from both the NWP soil and the TBH sediment waste using the TDU was excellent. Levels of PCBs and CPs were reduced to well below the strictest of the applicable CCME Interim Soil Remediation criteria (in the case of PCBs, two orders of magnitude below the strictest criterion). Some tin from the TDU was mixed in and collected with the processed solids. Contaminant concentrations in the scrubber water indicated excellent destruction of the contaminants desorbed from the waste.
- (2) Tests of the redesigned Thermal Desorption Mill (TDM) using spiked samples of the same wastes yielded excellent results also. The waste samples were spiked to levels in the range 500-800  $\mu\text{g/g}$  PCBs. The concentration of PCBs in the processed solids was found to be in the  $\text{ng/g}$  range, which corresponds to 99.999% desorption of PCBs. Additionally, desorption of PAHs was demonstrated to be as high as 99.999%, something that even the original TDU design did not achieve. All contact between waste and molten tin was prevented, so no tin was lost from the TDM. Overall, the TDM and ECO LOGIC Process have been shown quite capable of remediating soil from the Northern Wood Preservers' site and sediment from Thunder Bay Harbour. The test results indicate that a 10-minute waste residence interval should be adequate for complete PCB desorption, and that the tin temperature can fall as low as  $500^{\circ}\text{C}$  without penalizing desorption performance. Some development of the full-scale TDM waste feed mechanism will be necessary to ensure its ability to convey dry, sandy wastes.

APPENDIX A

DATA QUALITY

ASSURANCE PLAN



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## **1A DATA QUALITY ASSURANCE PLAN**

### **1.1A Quality Assurance Objectives**

The overall objective of the sampling and analysis effort is to provide data for evaluating the destruction and removal efficiencies of the ECO LOGIC Process that are precise, accurate, comparable, representative, and complete.

### **1.2A Performance Monitoring**

Performance monitoring techniques will help determine whether there are any problems in the whole method, from extraction to instrumental analysis. Performance monitoring devices include method spikes, analytical blanks, surrogate spiking and duplicate analysis.

#### **1.2.1A Laboratory Blank**

A laboratory blank is analysed to ensure that all laboratory glassware and reagents are free of interfering compounds. One analytical blank per sample extraction batch will be analysed.

#### **1.2.2A Surrogate Spiking**

Blanks, matrix spikes, and process samples will be spiked with surrogate compounds prior to extraction to demonstrate recoveries through all phases of the analytical procedure. The compounds spiked and their concentrations are given in Table 1A.

#### **1.2.3A Detection Limits**

The instrumental detection limits (IDL) are calculated by determining the average noise level (area count) of 10-20 peaks and multiplying by 3, and then multiplying by the response factors.

Table 1A SURROGATE SPIKES		
<i>Analyte Group</i>	<i>Surrogates</i>	<i>Conc. *</i>
PCBs	PCB 14	1 µg/mL
	PCB 65	1 µg/mL
	PCB 166	1 µg/mL
	PCB 204	1 µg/mL
Semivolatile Organics	Nitrobenzene-d <sub>5</sub>	25 µg/mL
	2-Fluorobiphenyl	25 µg/mL
	Terphenyl-d <sub>14</sub>	25 µg/mL
	Phenol-d <sub>6</sub>	100 µg/mL
	2,4,6-Tribromophenol	100 µg/mL
Chlorophenols	2,4,6-Tribromophenol	200 µg/mL

\* Concentration in final extract if recovered at 100%.

$$IDL = 3 \times \text{noise} \times RF \quad (1)$$

The method or sample detection limits (MDL or SDL) are then calculated by treating the IDL as you would a sample by applying the appropriate factors (ie dilution (DF), final volume (FV), sample mass (m) or volume (v)).

$$MDL = \frac{IDL \times FV \times DF}{m \text{ OR } v} \quad (2)$$

#### **1.2.4A Instrument Performance Monitoring**

##### **1.2.4.1A Control Charting**

A control sample will be analysed daily. It contains one or more compounds that represent all groups being analysed on a particular instrument. The peak area of the compounds will be plotted on a control chart. By plotting in this manner, downward trends in system efficiency will be noticed and the appropriate maintenance measure can be applied. By developing a history of instrument performance in this way it may be possible to predict when problems might occur and take preventative action.

##### **1.2.4.2A Autotune**

For the MSD's additional performance information can be gained by performing autotunes. Autotunes will show if there is a leak in the system or if the detector itself needs maintenance. Autotunes will be done before samples are run on the instrument, and the results must show the absence of air and an optimal detector response before samples are permitted to be analysed. A poor autotune suggests that maintenance may be required.

##### **1.2.4.3A Regular Maintenance**

Included in the routine maintenance of the GC/MSD systems are regular changing of the inlet septa and cleaning or replacement of inlet sleeves. Column performance is monitored through the daily check sample and such measures as column solvent washing, cutting, and replacement are performed as needed.

If peak area control charting or the autotune indicate a loss in detector sensitivity, which cannot be remedied by the above procedures, the ion source will be cleaned. An ongoing record of instrument performance is maintained.

### **1.3A Laboratory Records**

All information about a sample in every step of the analysis is recorded in a laboratory notebook. There is one laboratory book for each major project.

### **1.4A Archiving**

Excess sample material will be stored for a period of three months after final results are reported. Following this period, the sample material will be discarded unless ECO LOGIC is otherwise notified. If desired, the sample material may be returned to the client at their expense.

All chromatograms, mass spectra, hard copies of reports, chain of custodies, project books, and any other material relating to a project will be kept in a project file or box for future reference. Computer files such as data files, integration files, quantitation files and report files will be stored on cartridge tape or floppy diskette after data interpretation is completed.

### **1.5A Qualitative QA Objectives**

Comparability is a measure of the confidence with which one data set can be compared to another. The following measures will be taken to ensure the comparability of the data.

- Standardized written sampling and analysis procedures;
- Standard handling procedures used for all collected samples;
- Standard analytical procedures and experienced personnel used throughout the program;
- A uniform supply of sampling containers used; and
- Results reported in consistent units.

### **1.6A Monitoring Procedures**

All critical information relating to the overall performance of the process is monitored by the operator. Key process parameters include:

- reactor temperature (850 to 950°C)
- reactor pressure (-10" to 10" H<sub>2</sub>O)
- TDU tin (550 to 600°C)
- TDU hydrogen atmosphere (350 to 500°C)
- scrubber exit temperature (35 to 40°C)
- CIMS-500 analyses (<1000 ppm benzene, <100 ppm chlorobenzene)
- hydrogen concentration is adequate (>50% dry basis)

A significant process feature is the CIMS-500 chemical ionization mass spectrometer system. This process gas analyzer is capable of measuring organic compounds in gas streams on a continuous basis at part per billion levels. As such, it can monitor for products of incomplete reduction and essentially measure destruction efficiency on a continuous basis. For HCB destruction, chlorobenzene is monitored as an indicator of destruction efficiency. If the chlorobenzene concentration begins to rise, the operator is alerted with a low-level alarm.

#### **1.6.1A Process Monitoring**

Temperatures, pressures, flow rates and, other parameters are monitored at critical locations in the process. This is accomplished using thermocouples, pressure transmitters, flowmeters, and gas analyzers. Table 2A gives the equipment specifications for the various types of monitors. The specifications for the CIMS-500 are given in Table 3A.

Process data is also manually logged every 15 minutes by the operator when he makes a process inspection tour. This serves to keep the operator involved with the process and to alert the operator to slow trends that might not otherwise be obvious.

#### **1.6.2A Custody Procedures**

A Chain of Custody form will be filled out by the sampler immediately after sampling. The information on the sampling container is recorded as well as more detailed information, such as project name, project number, client name, analysis completion date, and the parameters

for analysis. The sampler will sign the form at the bottom and is responsible for the samples until signed over to another person or the laboratory.

Once samples are brought to the laboratory, custody is switched to the lab. Each sample is then logged in and assigned a laboratory number prior to storage. The Chain of Custody form is signed and dated by the sample submitter and the laboratory receiver. Information is taken from the Chain of Custody Record form and sample container label and recorded onto a sample initialization form (log-in sheet), where each sample is assigned a laboratory number. The location in which a particular sample can be found is also recorded on this form.

After a sample is given a laboratory number, it will be identified by that number only during all parts of the analysis. This number is the only thing linking the sample to its field identification and, therefore, is clearly and legibly marked on all containers having the sample or sample extract within it. All samples, regardless of analysis parameters, are stored in a refrigerator at 4°C. All samples are extracted within 14 days of sampling.

Table 2A PROCESS MONITORING EQUIPMENT SPECIFICATIONS

Equipment Type:	Temperature Probe	Pressure Gauges	Oxygen Analyzer
Parameters:	Temperature	Pressure	O <sub>2</sub> concentration
Manufacturer:	Thermo-Kinetics/ Yokogawa	Dwyer Magnehelic	Yokogawa
Model Number:	Type K	2000 Series	Z021C/D
Date Manufactured:	1992	1990	1990
Sensing Elements:	Type K Thermocouple	Diaphragm	Zirconium oxide sensor
Interferences/ Limitations:	Up to 1250°C	Condensation	High hydrocarbon concentration interference
Range of Measurements:	-200°C to 1250°C	0.5", 0-10", 0-20" H <sub>2</sub> O	0-25% O <sub>2</sub>
Accuracy:	±0.75% or 2.2°C	±5%	±0.1%
Precision:	0.1°C	±1%	0.01%
Monitoring Stations:	1,2,5,7,8,12	1,3,9	9
Calibration Procedure:	Check at 0°C & 100°C	Check against water manometer	Zero with N <sub>2</sub> span with certified calibration gas
Frequency:	On Installation	Once per month	Every 24 hours
Documentation:	Manufacturer's specs, calibration log book	Calibration log book	Recorded by process controller, calibration log book
Standards Source:	Ice water, boiling water, barometric pressure	N/A	CANOX
Precision:	±1°C	±0.2" H <sub>2</sub> O	±0.05% O <sub>2</sub>
Accuracy:	±1%	±1%	±5%



Table 2A PROCESS MONITORING EQUIPMENT SPECIFICATIONS (Continued)

Equipment Type:	Mass Flow Meter	Scale
Parameters:	Gas Flow	Weight
Manufacturer:	MKS Instruments	O'Haus
Model Number:	1259C	E400D
Date Manufactured:		
Sensing Elements:	Thermoelectric	Mechanical
Interferences/ Limitations:	Inlet Pressure < 150 psi	10-30°C
Range of Measurements:	0-20,000 SCCM (N <sub>2</sub> )	0-400g/40g
Accuracy:	0.8%	
Precision:	0.2%	0.007g
Monitoring Stations:	4,6,10,12	N/A
Calibration Procedure:	Bubble meter	Known Weight
Frequency:	Weekly	As Required
Documentation:	Recorded by process controller, calibration log book	Calibration log book
Standards Source:	CANOX	
Precision:		
Accuracy:		

Table 3A CIMS-500 SPECIFICATIONS

Equipment Type:	Chemical Ionization On-Line Mass Spectrometer
Parameters:	Benzenes, chlorobenzenes, VOCs, PAHs
Manufacturer:	V&F Analystechnik, Absam, Austria
Model Number:	CIMS-500
Date Manufactured:	1990
Sensing Elements:	Mass selective quadrupole/high speed electron multiplier/digital particle counting system
Interferences/Limitations:	N0 GC peak separation - compounds of equal mass may register together
Range of Measurement:	10 ppb - % level
Monitoring Stations:	MS10
Predicted Range of Measurement:	Trace components: 10 ppb - 10 ppm Bulk components: 10 ppm - % levels
Calibration Procedure:	Permeation cell for selected trace components/verification with certified permeation tubes. Bulk gas calibration with gas cylinder mixture.
Frequency:	Every 4 hours or as needed
Documentation:	Calibration periods/results manually typed on monitoring screen indicating time, date, process stage, etc.
Standards Source:	Canadian Liquid Air, NBS traceable permeation tubes
Standards Purity:	NBS traceable standards Bulk calibration gas of certified grade
Precision:	10% relative standard deviation of repeated permeation cell injections

# APPENDIX B

## TEST RESULTS

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Table 1B LAB RESULTS FOR TEST #1 - NWP SOIL

Lab ID:	94-091	94-044	94-046	94-056	94-105	94-103
Field ID:	Soxhlet		Processed	R.O.	Pre-run	Post-run
	Blank	Waste	Waste	Blank	Scrubber	Scrubber
Units:	(µg/g)	(µg/g)	(µg/g)	(µg/L)	(µg/L)	(µg/L)
Percent Solids:		71.6	99.9			
<b>PCB ISOMER</b>						
Mono	ND(0.000007)	0.0017	0.0002	ND(0.0005)	0.37	ND(0.0008)
Di	ND(0.00002)	0.019	ND(0.00009)	ND(0.001)	0.47	ND(0.003)
Tri	ND(0.00001)	0.35	0.00008	0.0007	0.62	0.028
Tetra	0.00005	1.3	0.0013	ND(0.002)	2.8	0.25
Penta	0.00017	0.58	0.00086	0.003	1.3	0.3
Hexa	ND(0.00003)	0.28	ND(0.00006)	ND(0.002)	0.27	0.077
Hepta	ND(0.00003)	0.12	ND(0.00004)	ND(0.003)	0.041	ND(0.007)
Octa	ND(0.00005)	0.043	ND(0.00009)	ND(0.008)	ND(0.009)	ND(0.009)
Nona	ND(0.00005)	ND(0.003)	ND(0.00007)	ND(0.009)	ND(0.01)	ND(0.009)
Deca	ND(0.00005)	ND(0.002)	ND(0.00004)	ND(0.01)	ND(0.01)	ND(0.01)
Total	0.00022	2.7	0.0025	0.0037	5.9	0.66
<b>Surrogate Recovery %</b>						
PCB 14	86	50	93	64	97	74
PCB 65	81	I	83	64	85	73
PCB 166	87	73	79	64	87	71
PCB 204	106	125	92	93	138	110
<b>Target Chlorophenol</b>						
2-Chlorophenol	ND(0.2)	ND(0.0003)	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
2,6-Dichlorophenol	ND(0.2)	ND(0.0003)	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
2,5-Dichlorophenol	ND(0.2)	ND(0.0003)	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
2,3-Dichlorophenol	ND(0.2)	0.003	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
3,5-Dichlorophenol	ND(0.2)	ND(0.0003)	ND(0.2)	ND(0.03)	ND(0.03)	0.1
3,4-Dichlorophenol	ND(0.2)	ND(0.0003)	0.7	ND(0.03)	ND(0.03)	ND(0.03)
2,4,6-Trichlorophenol	ND(0.2)	0.001	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
2,3,6-Trichlorophenol	ND(0.2)	0.0038	0.7	ND(0.03)	ND(0.03)	ND(0.03)
2,4,5-Trichlorophenol	ND(0.2)	0.0053	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
2,3,5-Trichlorophenol	ND(0.2)	0.0058	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
2,3,4-Trichlorophenol	ND(0.2)	0.0046	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
3,4,5-Trichlorophenol	ND(0.2)	0.057	ND(0.2)	ND(0.03)	0.04	ND(0.03)
2,3,5,6-Tetrachlorophenol	ND(0.2)	0.023	0.5	ND(0.03)	ND(0.03)	ND(0.03)
2,3,4,5-Tetrachlorophenol	ND(0.2)	0.11	ND(0.2)	ND(0.03)	ND(0.03)	ND(0.03)
Pentachlorophenol	ND(0.2)	2.2	2.4	ND(0.03)	ND(0.03)	ND(0.03)
<b>Surrogate Recovery (%)</b>						
2,4,6-Tribromophenol	71	27	85	74	69	61
<b>Target Semivolatile</b>						
Phenol	ND(0.002)	ND(0.01)	0.75	ND(0.2)	ND(0.2)	ND(0.2)
Bis(2-chloroethyl)ether	ND(0.001)	ND(0.006)	ND(0.001)	ND(0.05)	ND(0.05)	ND(0.05)
2-Chlorophenol	ND(0.01)	ND(0.004)	ND(0.01)	ND(0.2)	ND(0.2)	ND(0.2)
1,3-Dichlorobenzene	ND(0.003)	ND(0.003)	ND(0.003)	ND(0.06)	ND(0.06)	ND(0.06)
1,4-dichlorobenzene	ND(0.003)	0.004	ND(0.003)	0.32	0.32	0.21
1,2-dichlorobenzene	ND(0.003)	ND(0.003)	ND(0.003)	ND(0.07)	ND(0.07)	ND(0.07)
Bis(2-chloroisopropyl)ether	ND(0.003)	ND(0.002)	ND(0.003)	ND(0.07)	ND(0.07)	ND(0.07)
Hexachloroethane	ND(0.003)	ND(0.1)	ND(0.003)	ND(0.1)	ND(0.1)	ND(0.1)
N-Nitrosodi-n-propyl amine	ND(0.008)	ND(0.2)	ND(0.008)	ND(0.4)	ND(0.4)	ND(0.4)
Nitrobenzene	ND(0.02)	ND(0.05)	ND(0.02)	ND(0.09)	ND(0.09)	ND(0.09)
Isophorone	ND(0.002)	ND(0.04)	0.19	ND(0.03)	ND(0.03)	ND(0.03)
2-Nitrophenol	ND(0.03)	ND(0.02)	ND(0.03)	ND(0.2)	ND(0.2)	ND(0.2)

Table 1B LAB RESULTS FOR TEST #1 - NWP SOIL

Lab ID:	94-091	94-044	94-046	94-056	94-105	94-103
Field ID:	Soxhlet		Processed	R.O.	Pre-run	Post-run
	Blank	Waste	Waste	Blank	Scrubber	Scrubber
Units:	(µg/g)	(µg/g)	(µg/g)	(µg/L)	(µg/L)	(µg/L)
Percent Solids:		71.6	99.9			
Target Semivolatile (Continued)						
2,4-Dimethylphenol	ND(0.2)	ND(0.04)	ND(0.2)	ND(0.2)	ND(0.2)	ND(0.2)
Bis(2-chloroethoxy)methane	ND(0.003)	ND(0.1)	ND(0.003)	ND(0.04)	ND(0.04)	ND(0.04)
2,4-Dichlorophenol	ND(0.003)	ND(0.03)	ND(0.003)	ND(0.1)	ND(0.1)	ND(0.1)
1,2,4-Trichlorobenzene	ND(0.002)	0.014	ND(0.002)	ND(0.09)	ND(0.09)	ND(0.09)
Naphthalene	0.0056	0.32	0.94	ND(0.02)	260	2000
Hexachlorobutadiene	ND(0.005)	ND(0.008)	ND(0.005)	ND(0.2)	ND(0.2)	ND(0.2)
4-Chloro-3-methylphenol	ND(0.007)	ND(0.02)	ND(0.007)	ND(0.2)	ND(0.2)	ND(0.2)
Hexachlorocyclopentadiene	ND(0.009)	ND(0.01)	ND(0.009)	ND(0.3)	ND(0.3)	ND(0.3)
2,4,6-Trichlorophenol	ND(0.03)	ND(0.02)	ND(0.03)	ND(0.2)	ND(0.2)	ND(0.2)
2-Chloronaphthalene	ND(0.001)	ND(0.006)	ND(0.001)	ND(0.05)	ND(0.05)	ND(0.05)
Acenaphthylene	ND(0.005)	0.07	ND(0.005)	ND(0.03)	130	540
Dimethylphthalate	ND(0.002)	ND(0.08)	ND(0.002)	ND(0.04)	ND(0.04)	ND(0.04)
2,6-Dinitrotoluene	ND(0.005)	ND(0.03)	ND(0.005)	ND(0.2)	ND(0.2)	ND(0.2)
Acenaphthene	ND(0.001)	0.04	ND(0.001)	ND(0.4)	22	150
2,4-Dinitrophenol	ND(0.02)	ND(0.03)	ND(0.02)	ND(0.7)	ND(0.7)	ND(0.7)
2,4-Dinitrotoluene	ND(0.004)	ND(0.09)	ND(0.004)	ND(0.1)	ND(0.1)	ND(0.1)
4-Nitrophenol	ND(0.01)	ND(0.07)	ND(0.01)	ND(3)	ND(3)	ND(3)
Fluorene	ND(0.001)	0.05	0.013	ND(0.05)	28	280
4-chlorophenyl phenyl ether	ND(0.003)	ND(0.01)	ND(0.003)	ND(0.1)	ND(0.1)	ND(0.1)
Diethylphthalate	ND(0.01)	ND(0.008)	ND(0.01)	ND(3)	0.3	0.4
4,6-Dinitro-2-methylphenol	ND(0.04)	ND(0.05)	ND(0.04)	ND(0.4)	ND(0.4)	ND(0.4)
N-Nitrosodiphenylamine	ND(0.004)	ND(0.009)	ND(0.004)	ND(0.2)	ND(0.2)	ND(0.2)
4-bromophenyl phenyl ether	ND(0.007)	ND(0.01)	ND(0.007)	ND(0.2)	ND(0.2)	ND(0.2)
Hexachlorobenzene	ND(0.006)	ND(0.009)	ND(0.006)	ND(0.2)	ND(0.2)	ND(0.2)
Pentachlorophenol	ND(0.02)	3.4	ND(0.02)	ND(0.5)	ND(0.5)	ND(0.5)
Phenanthrene	ND(0.001)	0.65	0.034	0.34	190	890
Anthracene	ND(0.001)	0.16	0.012	ND(0.6)	14	170
Di-n-Butylphthalate	ND(0.002)	ND(0.09)	0.030	2.2	4.8	4.2
Fluoranthene	ND(0.001)	0.69	0.011	0.14	96	630
Benzidine	ND(0.01)	ND(1)	ND(0.01)	ND(0.9)	ND(0.9)	ND(0.9)
Pyrene	ND(0.002)	0.68	0.014	ND(0.3)	55	340
Butylbenzylphthalate	ND(0.01)	0.14	ND(0.01)	ND(0.3)	ND(0.3)	ND(0.3)
Benzo[a]anthracene	ND(0.002)	0.32	0.014	ND(0.2)	9.9	69
3,3'-Dichlorobenzidine	ND(0.009)	ND(0.06)	ND(0.009)	ND(0.7)	ND(0.7)	ND(0.7)
Chrysene	ND(0.002)	0.55	0.018	ND(0.2)	19	120
Bis(2-ethylhexyl)phthalate	ND(0.003)	0.51	0.13	4.9	39	13
Di-n-octylphthalate	ND(0.1)	0.14	ND(0.1)	2.3	3.4	1.9
Benzo[b]fluoranthene	ND(0.07)	0.60	ND(0.07)	ND(0.2)	70	260
Benzo[k]fluoranthene	ND(0.08)	0.55	ND(0.08)	ND(0.2)	21	35
Benzo[a]pyrene	ND(0.09)	0.48	0.012	ND(0.2)	36	100
Indeno(1,2,3-c,d)pyrene	ND(0.006)	0.48	ND(0.006)	ND(0.2)	15	58
Dibenzo[a,h]anthracene	ND(0.004)	ND(0.006)	ND(0.004)	ND(1)	ND(1)	6
Benzo[g,h,i]perylene	ND(0.01)	0.48	ND(0.01)	ND(0.4)	12	39
Surrogate Recovery %						
Phenol-d6	19	21	36	19	12	12
2,4,6-Tribromophenol	37	29	48	54	46	48
Nitrobenzene-d5	36	30	35	44	40	38
2-Fluorobiphenyl	43	47	49	50	46	44
Terphenyl-d14	56	50	62	70	56	58

ND = NOT DETECTED (METHOD DETECTION LIMIT IN BRACKETS)

**Table 2B LAB RESULTS FOR TEST #2 - THUNDER BAY HARBOUR  
SEDIMENT**

Lab ID:	94-091	94-050	94-052	94-056	94-105	94-103
Field ID:	Soxhlet		Processed	R.O.	Pre-run	Post-run
	Blank	Waste	Waste	Blank	Scrubber	Scrubber
Units:	(µg/g)	(µg/g)	(µg/g)	(µg/L)	(µg/L)	(µg/L)
Percent Solids:		51.7	99.9			
<b>PCB ISOMER</b>						
Mono	ND(0.000007)	ND(0.005)	0.00018	ND(0.0005)	0.37	ND(0.0008)
Di	ND(0.00002)	ND(0.00005)	0.00016	ND(0.001)	0.47	ND(0.003)
Tri	ND(0.00001)	ND(0.0003)	0.00073	0.0007	0.62	0.028
Tetra	0.00005	ND(0.005)	0.0041	ND(0.002)	2.8	0.25
Penta	0.00017	0.0038	0.0034	0.003	1.3	0.3
Hexa	ND(0.00003)	0.0039	0.001	ND(0.002)	0.27	0.077
Hepta	ND(0.00003)	ND(0.0004)	ND(0.00006)	ND(0.003)	0.041	ND(0.007)
Octa	ND(0.00005)	ND(0.001)	ND(0.0002)	ND(0.008)	ND(0.009)	ND(0.009)
Nona	ND(0.00005)	ND(0.0005)	ND(0.00006)	ND(0.009)	ND(0.01)	ND(0.009)
Deca	ND(0.00005)	ND(0.0007)	ND(0.00005)	ND(0.01)	ND(0.01)	ND(0.01)
Total	0.00022	0.0077	0.0096	0.0037	5.9	0.66
<b>Surrogate Recovery %</b>						
PCB 14	86	106	96	64	97	74
PCB 65	81	105	86	64	85	73
PCB 166	87	68	85	64	87	71
PCB 204	106	117	99	93	138	110
<b>Target Chlorophenol</b>						
2-Chlorophenol	ND(0.0002)	ND(0.0005)	ND(0.0002)	ND(0.03)	ND(0.03)	ND(0.03)
2,6-Dichlorophenol	ND(0.0002)	ND(0.0005)	ND(0.0002)	ND(0.03)	ND(0.03)	ND(0.03)
2,5-Dichlorophenol	ND(0.0002)	ND(0.0005)	ND(0.0002)	ND(0.03)	ND(0.03)	ND(0.03)
2,3-Dichlorophenol	ND(0.0002)	0.0062	0.002	ND(0.03)	ND(0.03)	ND(0.03)
3,5-Dichlorophenol	ND(0.0002)	ND(0.0005)	ND(0.0002)	ND(0.03)	0.1	0.2
3,4-Dichlorophenol	ND(0.0002)	ND(0.0005)	ND(0.0002)	ND(0.03)	ND(0.03)	ND(0.03)
2,4,6-Trichlorophenol	ND(0.0002)	0.012	0.0008	ND(0.03)	ND(0.03)	ND(0.03)
2,3,6-Trichlorophenol	ND(0.0002)	ND(0.0005)	ND(0.0002)	ND(0.03)	ND(0.03)	ND(0.03)
2,4,5-Trichlorophenol	ND(0.0002)	0.011	0.001	ND(0.03)	ND(0.03)	ND(0.03)
2,3,5-Trichlorophenol	ND(0.0002)	0.0054	ND(0.0002)	ND(0.03)	ND(0.03)	ND(0.03)
2,3,4-Trichlorophenol	ND(0.0002)	ND(0.0005)	ND(0.0002)	ND(0.03)	ND(0.03)	ND(0.03)
3,4,5-Trichlorophenol	ND(0.0002)	0.043	0.001	ND(0.03)	0.06	ND(0.03)
2,3,5,6-Tetrachlorophenol	ND(0.0002)	0.033	0.0022	ND(0.03)	0.07	0.05
2,3,4,5-Tetrachlorophenol	ND(0.0002)	0.015	ND(0.0002)	ND(0.03)	ND(0.03)	ND(0.03)
Pentachlorophenol	ND(0.0002)	0.45	0.0026	ND(0.03)	0.06	0.1
<b>Surrogate Recovery (%)</b>						
2,4,6-Tribromophenol	71	75	14	74	64	69
<b>Target Semivolatile</b>						
Phenol	ND(0.002)	ND(0.01)	ND(0.002)	ND(0.2)	ND(0.2)	140
Bis(2-chloroethyl)ether	ND(0.001)	ND(0.006)	ND(0.001)	ND(0.05)	ND(0.05)	ND(0.05)
1,3-Dichlorobenzene	ND(0.003)	ND(0.003)	ND(0.003)	ND(0.06)	ND(0.06)	ND(0.06)
1,4-dichlorobenzene	ND(0.003)	0.007	0.007	0.32	0.19	0.18
1,2-dichlorobenzene	ND(0.003)	ND(0.003)	ND(0.003)	ND(0.07)	ND(0.07)	ND(0.07)
Bis(2-chloroisopropyl)ether	ND(0.003)	ND(0.002)	ND(0.003)	ND(0.07)	ND(0.07)	ND(0.07)
Hexachloroethane	ND(0.003)	ND(0.1)	ND(0.003)	ND(0.1)	ND(0.1)	ND(0.1)
N-Nitrosodi-n-propyl amine	ND(0.008)	ND(0.2)	ND(0.008)	ND(0.4)	ND(0.4)	ND(0.4)
Nitrobenzene	ND(0.02)	ND(0.05)	ND(0.02)	ND(0.09)	ND(0.09)	ND(0.09)
Isophorone	ND(0.002)	ND(0.04)	ND(0.002)	ND(0.03)	ND(0.03)	ND(0.03)
2-Nitrophenol	ND(0.03)	ND(0.02)	ND(0.03)	ND(0.2)	ND(0.2)	ND(0.2)
2,4-Dimethylphenol	ND(0.2)	ND(0.04)	ND(0.2)	ND(0.2)	ND(0.2)	ND(0.2)

**Table 2B LAB RESULTS FOR TEST #2 - THUNDER BAY HARBOUR SEDIMENT**

Lab ID:	94-091	94-050	94-052	94-056	94-105	94-103
Field ID:	Soxhlet		Processed	R.O.	Pre-run	Post-run
	Blank	Waste	Waste	Blank	Scrubber	Scrubber
Units:	(µg/g)	(µg/g)	(µg/g)	(µg/L)	(µg/L)	(µg/L)
Percent Solids:		51.7	99.9			
<b>Target Semivolatile (Continued)</b>						
Bis(2-chloroethoxy)methane	ND(0.003)	ND(0.1)	ND(0.003)	ND(0.04)	ND(0.04)	ND(0.04)
2,4-Dichlorophenol	ND(0.003)	ND(0.03)	ND(0.003)	ND(0.1)	ND(0.1)	ND(0.1)
1,2,4-Trichlorobenzene	ND(0.002)	ND(0.004)	ND(0.002)	ND(0.09)	ND(0.09)	ND(0.09)
Naphthalene	0.006	9.1	1.9	ND(0.02)	730	1800
Hexachlorobutadiene	ND(0.005)	ND(0.008)	ND(0.005)	ND(0.2)	ND(0.2)	ND(0.2)
4-Chloro-3-methylphenol	ND(0.007)	ND(0.02)	ND(0.007)	ND(0.2)	ND(0.2)	ND(0.2)
Hexachlorocyclopentadiene	ND(0.009)	ND(0.01)	ND(0.009)	ND(0.3)	ND(0.3)	ND(0.3)
2-Chloronaphthalene	ND(0.001)	ND(0.006)	ND(0.001)	ND(0.05)	ND(0.05)	ND(0.05)
Acenaphthylene	ND(0.005)	0.15	0.008	ND(0.03)	270	460
Dimethylphthalate	ND(0.002)	ND(0.08)	ND(0.002)	ND(0.04)	ND(0.04)	ND(0.04)
2,6-Dinitrotoluene	ND(0.005)	ND(0.03)	ND(0.005)	ND(0.2)	ND(0.2)	ND(0.2)
Acenaphthene	ND(0.001)	5.8	0.04	ND(0.4)	51	160
2,4-Dinitrophenol	ND(0.02)	ND(0.03)	ND(0.02)	ND(0.7)	ND(0.7)	ND(0.7)
2,4-Dinitrotoluene	ND(0.004)	ND(0.09)	ND(0.004)	ND(0.1)	ND(0.1)	ND(0.1)
4-Nitrophenol	ND(0.01)	ND(0.07)	ND(0.01)	ND(3)	ND(3)	ND(3)
Fluorene	ND(0.001)	5.2	0.016	ND(0.05)	84	490
4-chlorophenyl phenyl ether	ND(0.003)	ND(0.01)	ND(0.003)	ND(0.1)	ND(0.1)	ND(0.1)
Diethylphthalate	ND(0.01)	ND(0.008)	ND(0.01)	0.3	0.5	0.4
4,6-Dinitro-2-methylphenol	ND(0.04)	ND(0.05)	ND(0.04)	ND(0.4)	ND(0.4)	ND(0.4)
N-Nitrosodiphenylamine	ND(0.004)	ND(0.009)	ND(0.004)	ND(0.2)	ND(0.2)	ND(0.2)
4-bromophenyl phenyl ether	ND(0.007)	ND(0.01)	ND(0.007)	ND(0.2)	ND(0.2)	ND(0.2)
Hexachlorobenzene	ND(0.006)	ND(0.009)	ND(0.006)	ND(0.2)	ND(0.2)	ND(0.2)
Phenanthrene	ND(0.001)	22	0.13	0.34	360	890
Anthracene	ND(0.001)	3.0	0.016	ND(0.6)	40	180
Di-n-Butylphthalate	ND(0.002)	ND(0.09)	0.014	2.2	3.7	2.7
Fluoranthene	ND(0.001)	17	0.073	0.14	190	450
Benzidine	ND(0.01)	ND(1)	ND(0.01)	ND(0.9)	ND(0.9)	ND(0.9)
Pyrene	ND(0.002)	13	0.035	ND(0.3)	120	260
Butylbenzylphthalate	ND(0.01)	ND(0.04)	ND(0.01)	ND(0.3)	ND(0.3)	100
Benzo[a]anthracene	ND(0.002)	4.4	0.010	ND(0.2)	23	6.5
3,3'-Dichlorobenzidine	ND(0.009)	ND(0.06)	ND(0.009)	ND(0.7)	ND(0.7)	ND(0.7)
Chrysene	ND(0.002)	3.9	0.013	ND(0.2)	39	97
Bis(2-ethylhexyl)phthalate	ND(0.003)	0.54	0.2	4.9	11	20
Di-n-octylphthalate	ND(0.1)	0.14	ND(0.1)	2.3	1.1	5.5
Benzo[b]fluoranthene	ND(0.07)	4.8	ND(0.07)	ND(0.2)	87	72
Benzo[k]fluoranthene	ND(0.08)	4.4	ND(0.08)	ND(0.2)	12	110
Benzo[a]pyrene	ND(0.09)	4.5	ND(0.09)	ND(0.2)	38	36
Indeno(1,2,3-c,d)pyrene	ND(0.006)	2.6	ND(0.006)	ND(0.2)	28	28
Dibenzo[a,h]anthracene	ND(0.004)	0.42	ND(0.004)	ND(1)	2	4
Benzo[g,h,i]perylene	ND(0.01)	2.4	ND(0.01)	ND(0.4)	15	2
<b>Surrogate Recovery %</b>						
Phenol-d6	19	18	31	19	13	20
2,4,6-Tribromophenol	37	38	58	54	51	60
Nitrobenzene-d5	36	30	39	44	38	51
2-Fluorobiphenyl	43	48	49	50	43	56
Terphenyl-d14	56	59	68	70	54	61

ND = NOT DETECTED (METHOD DETECTION LIMIT IN BRACKETS)



Table 3B      TDU TEST START UP PROCEDURES - NWP SOIL		
SBV Test ID:	WTC Test #1	Date: February 3, 1994
Operator:	DK	
Action	Time	Initial
Process control computer & CIMS operational	1000	DK
Begin N <sub>2</sub> Purge	1000	DK
After 5 volume exchanges with N <sub>2</sub> and O <sub>2</sub> analyser <0.4%, switch on reactor glo-bar	1150	DK
Turn on Recirc pump and Scrubber pump	1230	MJR
Take sample of pre-run Scrubber water	1245	MJR
Reactor temperature > 500°C, start H <sub>2</sub> purge* stop N <sub>2</sub> purge, turn on TDU heaters**	1300* 1300**	MJR
Reactor temperature > 850°C, start recirc heater	1330	MJR
Reactor temperature > 900°C and TDU > 550°C, connect exit gas stream to sampling apparatus, record test start in log book	1425	MJR
Start waste feed	1430	MJR

Table 4B OPERATOR LOG - NWP SOIL

CLIENT: WTC Test #1		DATE: February 3, 1994					
OPERATOR: DK, MR, SF							
TIME:	1330	1400	1430	1445	1500	1515	1530
<b>DESTRUCTOR</b>							
Pressure (in H <sub>2</sub> O)	3.5	2.2	2.5	2.5	3.0	3.0	2.5
Reactor Temp In (°C)	749	831	864	869	863	853	855
Reactor Temp Out (°C)	762	867	898	899	897	895	893
Recirc Heater Temp (°C)	23	103	141	141*	274	324	346
Scrubber Temp (°C)	12	14	15	16	17	19	21
Scrubber: pH	7.16	7.16	7.25	7.25	7.25	6.75	6.74
<b>TDU</b>							
Bath Temp (°C)	240	536	594	605	597	606	598
Breech Temp (°C)	344	496	539	551	519	517	508
Speed Control Setting (rpm)	-	-	2	2	3	4	4
<b>OTHER</b>							
O <sub>2</sub> Analyser (%)	0.17	0.185	0.19	0.19	0.19	0.20	0.20
Dry Gas Meter (Exit) (US Gal)	2170.3	2171.0	2172.0	2172.2	2172.7	2173.2	2173.6
Dry Gas Meter (Recirc) (US Gal)	2110.8	2111.7	2112.4	2112.7	2113.0	2113.3	2113.6

TIME:	1545	1600
<b>DESTRUCTOR</b>		
Pressure (in H <sub>2</sub> O)	3.0	2.5
Reactor Temp In (°C)	848	850
Reactor Temp Out (°C)	894	894
Recirc Heater Temp (°C)	360	363
Scrubber Temp (°C)	23	25
Scrubber: pH	6.74	6.52
<b>TDU</b>		
Bath Temp (°C)	607	581
Breech Temp (°C)	513	484
Speed Control Setting (rpm)	5	-
<b>OTHER</b>		
O <sub>2</sub> Analyser (%)	0.21	0.20
Dry Gas Meter (Exit) (US Gal)	2174.2	2174.6
Dry Gas Meter (Recirc) (US Gal)	2114.0	2114.2

\* = BAD CONNECTION

Table 5B      TDU TEST SHUT DOWN PROCEDURES - NWP SOIL		
SBV Test ID:	WTC Test #1	Date: February 3, 1994
Operator:	DK, MR, SF	
Action	Time	Initial
Waste feed hopper emptied. Stop feed screw	1555	MJR
Disconnect and seal exit gas sample apparatus, sample scrubber water, record test stop in log book	1611	MJR
Shut off reactor glo-bar*, recirc gas heater**, TDU heaters***	1610* 1620** 1605***	MJR
Shut off H <sub>2</sub> purge, start N <sub>2</sub> purge		MJR
Reactor temperature < 100°C, shut off N <sub>2</sub> purge, shut off Recirc pump, shut off scrubber pump	Feb.4/94 0930	DK
Remove processed soils from the TDU catch pot for analysis	Feb.4/94 1040	SF

Table 6B      TDU TEST START UP PROCEDURES - THUNDER BAY HARBOUR SEDIMENT		
SBV Test ID:	WTC Test #2	Date: February 8, 1994
Operator:	DK	
Action	Time	Initial
Process control computer & CIMS operational		
Begin N <sub>2</sub> Purge	1100	DK
After 5 volume exchanges with N <sub>2</sub> and O <sub>2</sub> analyser <0.4%, switch on reactor glo-bar	1145	DK
Turn on Recirc pump and Scrubber pump	1145	DK
Take sample of pre-run Scrubber water		
Reactor temperature > 500°C, start H <sub>2</sub> purge* stop N <sub>2</sub> purge, turn on TDU heaters**	1300** 1326*	DK
Reactor temperature > 850°C, start recirc heater	1330	DK
Reactor temperature > 900°C and TDU > 550°C, connect exit gas stream to sampling apparatus, record test start in log book	1540	MJR
Start waste feed	1542	MJR

Table 7B OPERATOR LOG - THUNDER BAY HARBOUR SEDIMENT

CLIENT:	WTC Test #2						
OPERATOR:	DK, MR, SF					DATE: February 8, 1994	
TIME:	1330	1400	1430	1505	1535	1545	1600
DESTRUCTOR							
Pressure (in H <sub>2</sub> O)	4.0	3.0	3.0	3.0	2.5	3.5	3.0
Reactor Temp In (°C)	727	807	814	812	793	787	778
Reactor Temp Out (°C)	747	839	848	836	814	807	807
Recirc Heater Temp (°C)	12	13	14	53	109	120	143
Scrubber Temp (°C)	14	16	16	17	18	19	20
Scrubber: pH	6.95	6.95	720	7.20	7.20	7.20	7.37
TDU							
Bath Temp (°C)	276	510	525	473	609	603	590
Breech Temp (°C)	332	458	469	415	549	544	517
Speed Control Setting (rpm)	-	-	-	-	2	2	2
OTHER							
O <sub>2</sub> Analyser (%)	0.16	0.18	0.18	0.19	0.19	0.19	0.20
Dry Gas Meter (Exit) (US Gal)	2178.6	2179.6	2180.4	2181.4	2182.3	2182.6	2183.1
Dry Gas Meter (Recirc) (US Gal)	2117.2	2118.2	2118.9	2119.5	2120.0	2120.3	2120.6

TIME:	1615	1630	1645	1700	1715	1730	1745
DESTRUCTOR							
Pressure (in H <sub>2</sub> O)	3.0	2.5	3.0	2.5	2.5	2.5	2.0
Reactor Temp In (°C)	781	784	786	791	793	794	799
Reactor Temp Out (°C)	818	829	834	840	844	849	851
Recirc Heater Temp (°C)	201	238	250	258	261	266	265
Scrubber Temp (°C)	23	26	28	30	32	35	36
Scrubber: pH	7.37	7.62	7.62	7.41	7.41	7.41	7.45
TDU							
Bath Temp (°C)	608	597	598	599	600	600	602
Breech Temp (°C)	507	489	493	494	495	489	490
Speed Control Setting (rpm)	2	2	2	2	2	2	-
OTHER							
O <sub>2</sub> Analyser (%)	0.20	0.20	0.19	0.18	0.18	0.18	0.18
Dry Gas Meter (Exit) (US Gal)	2183.7	2184.3	2184.7	2185.2	2185.6	2186.1	2186.5
Dry Gas Meter (Recirc) (US Gal)	2121.2	2121.9	2122.4	2122.8	2123.3	2123.8	2124.3

Table 8B      TDU TEST SHUT DOWN PROCEDURES - THUNDER BAY HARBOUR SEDIMENT		
SBV Test ID:	WTC Test #2	Date: February 8, 1994
Operator:	DK	
Action	Time	Initial
Waste feed hopper emptied. Stop feed screw	1745	DK
Disconnect and seal exit gas sample apparatus, sample scrubber water, record test stop in log book	-	-
Shut off reactor glo-bar*, recirc gas heater**, TDU heaters***	1800* 1756** 1753***	DK
Shut off H <sub>2</sub> purge, start N <sub>2</sub> purge	1756	DK
Reactor temperature < 100°C, shut off N <sub>2</sub> purge, shut off Recirc pump, shut off scrubber pump	Feb.9/94 1000	DK
Remove processed soils from the TDU catch pot for analysis	Feb.9/94 1100	SF

**APPENDIX C**

**TEST RESULTS FOR THE NEW**

**THERMAL DESORPTION MILL**

LIST OF TABLES

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Table 1C TEST RESULTS FOR THE NEW THERMAL DESORPTION MILL - PCBs

Lab ID:	Test #3 PCBs			Test #4 PCBs			Test #5 PCBs		
	94-256	94-203	94-231	94-256	94-232	94-233	94-256	94-236	94-241
Field ID:	Soxhlet	Waste	Process	Soxhlet	Waste	Process	Soxhlet	Waste	Process
	Blank		Waste	Blank		Waste	Blank		Waste
Percent Solids:		88	100		88	100		54	100
<u>PCB Isomer</u>									
Mono	nd(0.00003)	nd (0.1)	nd (0.00009)	nd(0.00003)	nd (0.1)	nd (0.00005)	nd(0.00003)	nd (0.2)	nd (0.00008)
Di	nd(0.00009)	0.4	nd (0.00007)	nd(0.00009)	0.6	0.00023	nd(0.00009)	1	0.0003
Tri	0.00056	2	0.00035	0.00056	3.1	nd (0.0001)	0.00056	3.4	0.0015
Tetra	0.0015	96	0.0011	0.0015	86	0.0006	0.0015	120	0.0019
Penta	0.0033	210	0.0016	0.0033	270	0.0008	0.0033	390	0.017
Hexa	0.0018	100	0.00088	0.0018	120	nd (0.0002)	0.0018	140	0.0072
Hepta	nd(0.0001)	28	nd (0.0001)	nd(0.0001)	34	nd (0.00009)	nd(0.0001)	49	0.0016
Octa	nd(0.00007)	4.5	nd (0.0001)	nd(0.00007)	5.7	nd (0.00009)	nd(0.00007)	10	nd (0.0002)
Nona	nd(0.00002)	nd (0.06)	nd (0.0004)	nd(0.00002)	nd (0.04)	nd (0.0004)	nd(0.00002)	nd (0.5)	nd (0.0003)
Deca	nd(0.00006)	nd (0.03)	nd (0.0001)	nd(0.00006)	nd (0.01)	nd (0.0001)	nd(0.00006)	nd (0.1)	nd (0.0002)
Total	0.0072	440	0.0039	0.0072	520	0.0016	0.0072	710	0.028
<u>Surrogate Recovery %</u>									
PCB 14	71	68	81	71	80	72	71	81	63
PCB 65	66	62	70	66	76	60	66	74	59
PCB 166	82	89	76	82	130	63	82	69	77
PCB 204	90	81	77	90	112	67	90	79	90

Lab ID:	Test #6 PCBs			Test #7 PCBs	
	94-256	94-242	94-246	94-355	94-352
Field ID:	Soxhlet	Waste	Process	Soxhlet	Process
	Blank		Waste	Blank	Waste
Percent Solids:		54	100		100
<u>PCB Isomer</u>					
Mono	nd(0.00003)	nd (0.2)	nd (0.00004)	nd (0.00004)	nd (0.00005)
Di	nd(0.00009)	1	nd (0.0001)	nd (0.00006)	nd (0.00009)
Tri	0.00056	4.1	nd (0.0002)	0.00042	0.00076
Tetra	0.0015	140	0.0021	0.00071	0.012
Penta	0.0033	400	0.0056	0.0032	0.033
Hexa	0.0018	180	0.0016	0.0008	0.016
Hepta	nd(0.0001)	53	0.00043	0.0002	0.0034
Octa	nd(0.00007)	9.9	nd (0.0001)	nd (0.0005)	nd (0.0003)
Nona	nd(0.00002)	nd (0.4)	nd (0.0002)	nd (0.0003)	nd (0.0003)
Deca	nd(0.00006)	nd (0.1)	nd (0.00007)	nd (0.0001)	nd (0.0002)
Total	0.0072	790	0.0097	0.0053	0.065
<u>Surrogate Recovery %</u>					
PCB 14	71	82	76	69	43
PCB 65	66	73	72	67	50
PCB 166	82	75	90	77	86
PCB 204	90	90	102	104	119

nd - non detect

D - diluted out of range

**Table 2C TEST RESULTS FOR THE NEW THERMAL DESORPTION MILL - PAHs**

Lab ID: Field ID:	Test #3 PAHs			Test #4 PAHs			Test #5 PAHs		
	94-256	94-203	94-231	94-256	94-232	94-233	94-256	94-236	94-241
	Soxhlet Blank	Waste	Process Waste	Soxhlet Blank	Waste	Process Waste	Soxhlet Blank	Waste	Process Waste
<b>PAH</b>									
Naphthalene	nd(0.002)	5.9	2.6	nd(0.002)	5.3	0.029	nd(0.002)	6.4	0.07
Acenaphthylene	nd(0.003)	0.58	0.084	nd(0.003)	0.61	nd(0.003)	nd(0.003)	0.38	nd(0.003)
Acenaphthene	nd(0.005)	0.72	0.029	nd(0.005)	0.91	nd(0.005)	nd(0.005)	2.3	nd(0.005)
Fluorene	nd(0.005)	0.94	nd(0.005)	nd(0.005)	0.94	nd(0.005)	nd(0.005)	2.9	nd(0.005)
Phenanthrene	nd(0.004)	9.8	0.049	nd(0.004)	10	0.016	nd(0.004)	9.9	0.0067
Anthracene	nd(0.004)	4.4	nd(0.004)	nd(0.004)	1.9	0.017	nd(0.004)	2.3	nd(0.004)
Fluoranthene	nd(0.004)	40	0.018	nd(0.004)	21	0.095	nd(0.004)	22	nd(0.004)
Pyrene	nd(0.004)	50	0.018	nd(0.004)	48	0.015	nd(0.004)	25	nd(0.004)
Benzo(a)anthracene	nd(0.007)	11	nd(0.007)	nd(0.007)	9.6	nd(0.007)	nd(0.007)	7.4	nd(0.007)
Chrysene	nd(0.007)	26	nd(0.007)	nd(0.007)	24	nd(0.007)	nd(0.007)	13	nd(0.007)
Benzo(b)fluoranthene	nd(0.008)	33	nd(0.008)	nd(0.008)	40	nd(0.008)	nd(0.008)	17	nd(0.008)
Benzo(k)fluoranthene	nd(0.009)	21	nd(0.009)	nd(0.009)	20	nd(0.009)	nd(0.009)	8.1	nd(0.009)
Benzo(a)pyrene	nd(0.009)	22	nd(0.009)	nd(0.009)	22	nd(0.009)	nd(0.009)	9.3	nd(0.009)
Indeno[1,2,3-c,d]pyrene	nd(0.007)	20	nd(0.007)	nd(0.007)	23	nd(0.007)	nd(0.007)	5.5	nd(0.007)
Dibenzo[a,h]anthracene	nd(0.01)	4.2	nd(0.01)	nd(0.01)	5.3	nd(0.01)	nd(0.01)	1.1	nd(0.01)
Benzo[g,h,i]perylene	nd(0.01)	25	nd(0.01)	nd(0.01)	25	nd(0.01)	nd(0.01)	5.3	nd(0.01)
<b>Surrogate Recovery %</b>									
Anthracene-d10	70	80	80	70	32	83	70	82	52
Benzo(a)anthracene-d12	79	104	92	79	96	106	79	92	64

Lab ID: Field ID:	Test #6 PAHs			Test #7 PAHs	
	94-256	94-242	94-246	94-355	94-352
	Soxhlet Blank	Waste	Process Waste	Soxhlet Blank	Process Waste
<b>PAH</b>					
Naphthalene	nd(0.002)	4.6	nd(0.002)	nd(0.002)	0.49
Acenaphthylene	nd(0.003)	0.29	nd(0.003)	nd(0.003)	0.019
Acenaphthene	nd(0.005)	1.9	nd(0.005)	nd(0.005)	0.0079
Fluorene	nd(0.005)	2.4	nd(0.005)	nd(0.005)	0.011
Phenanthrene	nd(0.004)	8.5	0.0095	nd(0.004)	0.099
Anthracene	nd(0.004)	1.8	nd(0.004)	nd(0.004)	0.015
Fluoranthene	nd(0.004)	19	nd(0.004)	nd(0.004)	0.022
Pyrene	nd(0.004)	22	nd(0.004)	nd(0.004)	0.021
Benzo(a)anthracene	nd(0.007)	6.1	nd(0.007)	nd(0.007)	0.02
Chrysene	nd(0.007)	11	nd(0.007)	nd(0.007)	0.021
Benzo(b)fluoranthene	nd(0.008)	14	nd(0.008)	nd(0.008)	0.027
Benzo(k)fluoranthene	nd(0.009)	7.1	nd(0.009)	nd(0.009)	nd(0.009)
Benzo(a)pyrene	nd(0.009)	7.7	nd(0.009)	nd(0.009)	0.021
Indeno[1,2,3-c,d]pyrene	nd(0.007)	4.9	nd(0.007)	nd(0.007)	nd(0.007)
Dibenzo[a,h]anthracene	nd(0.01)	1	nd(0.01)	nd(0.01)	nd(0.01)
Benzo[g,h,i]perylene	nd(0.01)	4.9	nd(0.01)	nd(0.01)	nd(0.01)
<b>Surrogate Recovery %</b>					
Anthracene-d10	70	76	68	84	104
Benzo(a)anthracene-d12	79	86	72	104	124

nd - non detect