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BIODEGRADATION OF HD AND TETRYTOL HYDROLYSATES IN A PILOT SCALE IMMOBILIZED CELL BIOREACTOR

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To address public concerns over the safe destruction of the U.S. chemical weapon stockpile, the Assembled Chemical Weapons Assessment (ACWA) Program identified neutralization followed by biodegradation as having potential for destroying the chemical agent and burster charge fills of chemical agent projectiles. This report describes the pilot scale study conducted by the authors at the U.S. Army Edgewood Chemical Biological Center (ECBC), APG, MD. This research builds upon the neutralization/biodegradation process developed at ECBC and expands that process to include the burster fills found in mustard (HD) agent projectiles. A 1000-gal immobilized cell bioreactor (ICB) system was used to demonstrate the ability of an activated sludge consortium to degrade the chemical constituents of the US Army's 105-mm chemical agent projectile. The study demonstrated the ability of the Water Hydrolysis of Energetic and Agent Technologies (WHEAT) and the ICB to degrade 99.999% of hydrolyzed HD and its schedule-two breakdown products. During the test period, the ICB system processed 16,896 lb of HD hydrolysate and 660 lb of tetrytol hydrolysate. During the system validation, HD hydrolysate and its breakdown products were degraded to below detectable limits in all system effluents. Through a three-step process, 50% of the effluent was recycled into the ICB. Liquid effluents were characterized as non-toxic by the Toxicity Characteristic Leaching Procedure (TCLP). The ICB, process, and study findings are presented.

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PREFACE

The work described in this report was authorized under Project Nos. 778017 and 778117, Assembled Chemical Weapons Assessment (ACWA) Program. This work was started in September 1998 and completed in May 1999.

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1. INTRODUCTION

The purpose of this report is to describe the demonstration of an Immobilized Cell Bioreactor (ICB) system's ability to degrade a mixture of hydrolyzed mustard agent (HD) and hydrolyzed tetrytol. This study is part of a larger demonstration plan sponsored by the Program Manager for Assembled Chemical Weapon Assessment (ACWA) Program.

Water Hydrolysis of Energetic and Agent Technology (WHEAT) is an alternate process to incineration proposed for complete destruction of warfare munitions containing energetics, propellants and mustard or nerve agents. The WHEAT technologies will be used to demonstrate destruction of materials representative of M60 105 mm projectiles (HD/Tetrytol), M426 projectiles (GB/Comp B) and M55 Rockets (VX/Comp B/M28 propellant). The WHEAT technologies include water jet for cutting and boring operations to remove components from metal projectiles, a hydrolyzation step to detoxify and make biologically available projectile chemical components, and a high temperature steam process for 5X treatment of metal parts and other solid wastes. A more complete description of the entire study is available in the ACWA Demonstration Study Plan[1].

The assessment goals include:

1. Assess alternate chemical destruction technologies to the current baseline "incineration" technology.
2. To confirm that the proposed technologies represent a total solution for assembles chemical weapons.
3. To define the type, quantity and chemical/physical characteristics of process emissions to assist in future permitting efforts.
4. To define critical design characteristics and operating parameters.
5. Utilize data for potential full-scale assembled weapons destruction systems.

The biological breakdown of the hydrolyzed agents, energetics and propellants was demonstrated in three bioreactors. The GB and VX hydrolysate reactors were operated at the Army Chemical Agent Munitions Disposal System (CAMDS) Site in Toole, Utah. The focus of this report, the hydrolyzed HD/Tetrytol ICB system, was operated at U.S. Army Edgewood Research, Development and Engineering Center at Aberdeen Proving Ground, MD.

The development o the ICB pilot scale design is based on a history of past benchtop/laboratory-scale studies with hydrolyzed HD. The use of Sequencing Batch Reactors (SBR) has demonstrated the ability to successfully degrade hydrolyzed HD[2] and was subsequently selected as the process-of-choice for chemical destruction of HD in ton containers at the Edgewood Chemical Biological Center (ECBC). Further research in the area of neutralization/biodegradation by Guelta and DeFrank,[3] of ECBC, led to success in the degradation of HD hydrolysate and VX nerve agent by ICBs. This study further laid the groundwork for expansion of the process to a pilot scale system that includes treatment of the burster fills found in mustard agent projectiles. This report describes the demonstration run of a pilot-scale ICB system and its ability to degrade a feedstock of chemical agent (hydrolyzed HD as the sole carbon source for the microbial culture) mixed with energetic (Tetrytol) in proportions that simulate the contents of the M60 projectile. The HD hydrolysate will provide the sole carbon source for the microbial culture. Tetrytol hydrolysate, the hydrolyzed energetic component of the projectile, will also be included in the feed system.

2. METHODS AND MATERIALS

2.1 HD HYDROLYSATE PRODUCTION

The mustard hydrolysate component of the HD/Tetrytol feed was provided by the Chemical Transfer Facility (CTF) at the Edgewood Area of Aberdeen Proving Ground. Mustard neutralization runs from June 8 to September 11, 1998 produced the selected batches (or "runs") of hydrolyzed HD. The lab-scale process was previously described by Harvey et al.[2] For this pilot-scale demonstration mustard agent (3.8% w/w) was mixed with hot water (approximately 180° C) in a reaction vessel, while vigorously agitated to form HD hydrolysate over a reaction time of about two hours. The acidic hydrolysate was then neutralized with solid NaOH during a cool down period, so that the resulting mixture, at a pH of 11-12, would be less toxic to microorganisms and subject to further biological breakdown. A sample from each hydrolysate batch was analyzed for residual HD, total organic carbon (TOC), organic chemical composition, sulfonium ion concentration, and metals content. Descriptive statistics of Schedule-2 compounds, pH and TOC, which characterize the HD hydrolysate, are presented in Table 3. Details about the procedure, facility and equipment used are described in the report, "HD Hydrolysate Production for the Assembled Chemical Weapons Assessment Program"[4].

2.2 TETRYTOL HYDROLYSATE PRODUCTION

The Tetrytol hydrolysate was produced at Pantex nuclear weapons plant near Amarillo, Texas. Tetrytol is an explosive mixture of 75% tetryl and 25 % trinitrotoluene (TNT). The tetrytol used to simulate the explosive component of the projectile was obtained from demolition blocks. The hydrolysate was produced in a 200-gallon reaction vessel by slowly adding 63 lbs. of tetrytol to 931 lbs. of a 6% solution of NaOH in water. The mixture is heated to approximately 90° C for 8 hours under constant agitation. The hydrolysate is then neutralized with sulfuric acid and filtered during the drumming process.

2.3 SYSTEM OVERVIEW

Honeywell International, the demonstration sub-contractor for the bioreactor technologies, supplied the mobile bioreactor systems. The ICB system was operated by ECBC research employees from the Environmental Technology Team. The principle components of the HD/Tetrytol bioreactor system were housed in two 40 by 8-foot transportation containers that were delivered on tractor trailer trucks. Once on site the containers were placed in a process building and the required utilities were connected.

The bioreactor system is diagramed in Figure 1 below. Major components include a feed tank for mixing HD and Tetrytol hydrolysates, a 1000-gallon steel ICB tank (the ICB), a 100-gallon flocculation reactor, a 300-gallon clarifier, 1000-gallon recycle water storage tank and a water recycle system consisting of activated carbon, microfiltration and reverse osmosis filter cartridges. The system was designed to process 200 gallons of feedstock per day containing 40 gallons of HD hydrolysate and 1.9 gallons of Tetrytol hydrolysate. Sixty-five percent of the 200-gallon-per-day effluent was to be recycled into the system after sludge removal and desalination. A salt brine/sludge concentrate of the effluent was collected for disposal as hazardous waste.

2.4 ICB SYSTEM DESCRIPTION AND OPERATION

The feed tank is a 200-gallon polyethylene tank approximately 6 feet tall. The tank was charged daily with a mixture of 40 gallons of HD hydrolysate and 1.9 gallons of Tetrytol hydrolysate. To help control pH, 2 lbs. of sodium bicarbonate was added to the feed daily. The mixture was brought to volume with water from the recycle water tank. An electric stirrer was mounted to the lid to mix the feed constituents. Feed was pumped into the ICB through an inline pump and flowmeter. The flow was set to empty the feed tank by around 0900 hrs each day. A recirculating loop pumped effluent from the second chamber of the ICB (there are three chambers) into a recycle loop to pick-up feed and allow for pH monitoring and control.

The recycle loop was used to control pH and add required micronutrients into the system. A pH probe inserted in-line signaled a controller to add 10 % sodium hydroxide into the recycled effluent and micronutrient stream. Although not part of the original configuration, a second pH control loop was added between the third and second chambers when pH control became a problem. The pH controllers were set to maintain a pH of 7.5 to approximately 8.0.

The first 500-gallon chamber of three in the ICB was intended to be the site of principle feed degradation. The second and third chambers, 250 gallons each, were intended to remove any residual quantities of TDG that may be present in the effluent.

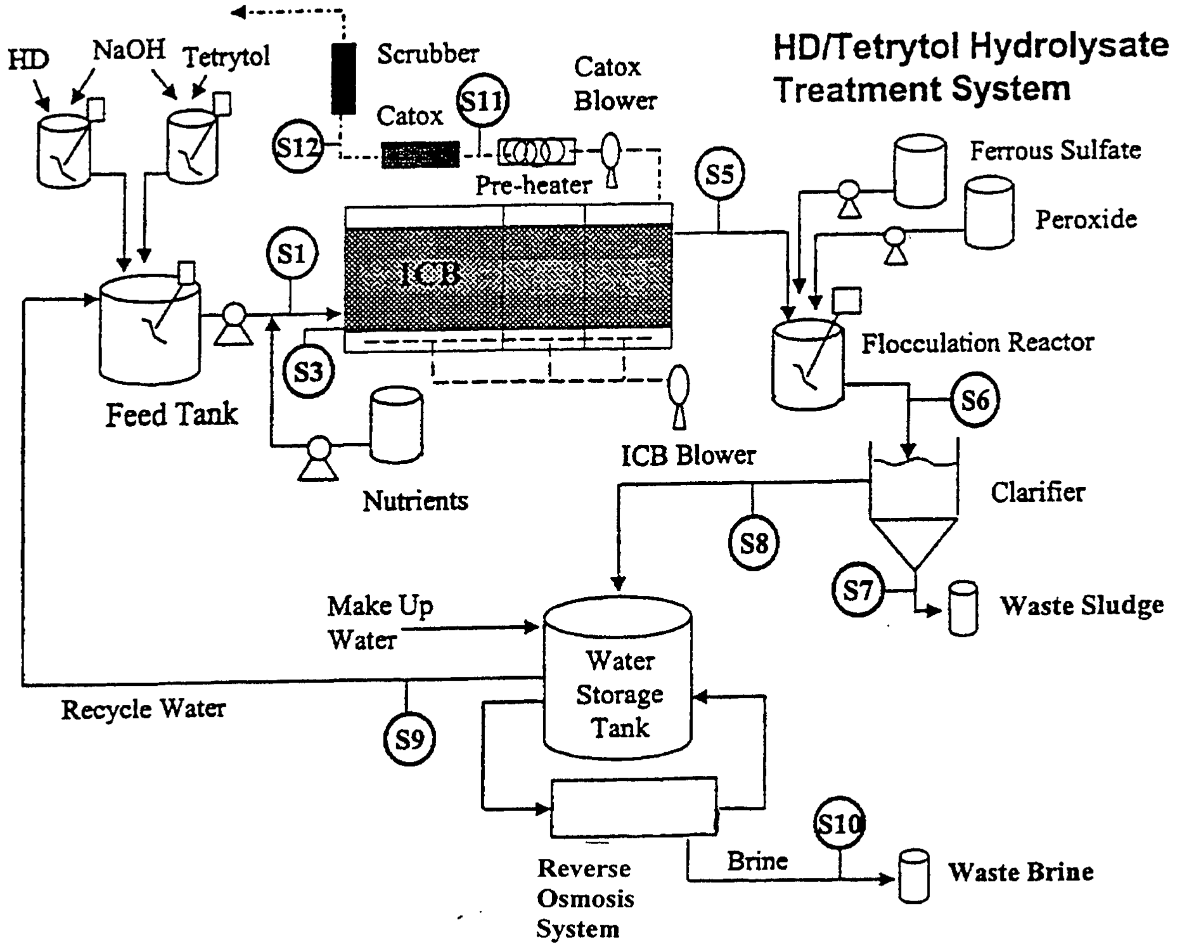

Figure 1. Schematic Diagram of the HD/Tetrytol Hydrolysate ICB System

Air was supplied to the ICB from a blower mounted outside the facility. The blower piped 50 actual cubic feet per minute (acfm) air into course diffusion bubblers in the bottom of the ICB. Air exhausted from the ICB was drawn off with a blower then pumped through a catalytic oxidation (CATOX) unit mounted inside the trailer followed by a High Efficiency Particulate (HEPA)/charcoal filter system before being exhausted outside. The CATOX unit heated the air to 450°C to destroy any volatile organic chemicals. The CATOX produced an acid that was then neutralized by a lime scrubber before venting to the HEPA filter.

Liquid effluent overflowed from one chamber to the next and then into a 55-gallon sump outside the ICB. Effluent was pumped from the sump in trailer one to a 90-gallon polyethylene flocculation reaction tank in trailer two. Ferrous sulfate and 30 percent hydrogen peroxide solutions were dispensed into the continuously stirred tank to promote flocculation of the biomass. The flocculated effluent was then pumped into the top of the inclined plate clarifier. Flocculated sludge settled to the bottom of the clarifier and was pumped into 275-

gallon tote tanks and discarded as hazardous waste. Clarified effluent went into the 1000-gallon recycle water holding tank.

A Reverse Osmosis (RO) system was used to prevent excessive salt and dissolved solids buildup in the recycled water (effluent). For two hours-per-day a circulating loop pumped effluent from the recycle water tank to the Reverse Osmosis (RO) desalination system. Permeate (desalinated water) from the RO unit was returned to the recycle water holding tank. Concentrated salt brine was pumped into the 275 gal waste tank for disposal through the Hazardous Waste Tracking System (HWTS). The original estimate was to produce 35 gallons per day of salt brine. When salt concentrations in the recycle water became higher than expected, the system was operated for longer periods to generate approximately 100 gallons of brine per day. A more complete description of the ICB system with wiring and flow schematics is available in the test demonstration study plan[1].

Trailer 1 contained the feed tank, 1000-gallon ICB, foam knock out drum, sump drum, CATOX unit and nutrient tank. Most of these components are pictured in Figure 2 below.

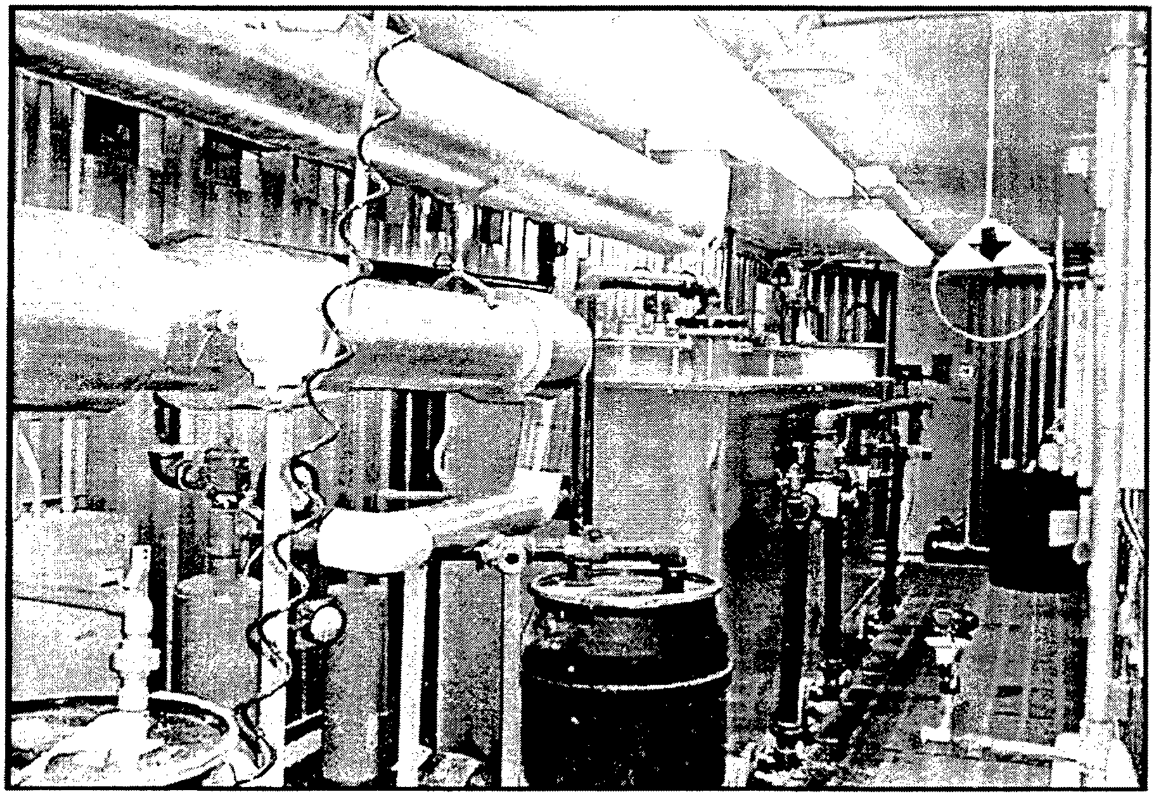

Figure 2. Components of the ICB System Housed in Trailer One

Trailer 2 contained support components for water recycling. These components include the flocculation reactor, inclined plate clarifier, dirty water holding tank, Reverse

Osmosis system and 1000-gallon recycle water holding tank. These systems are pictured in Figure 3.

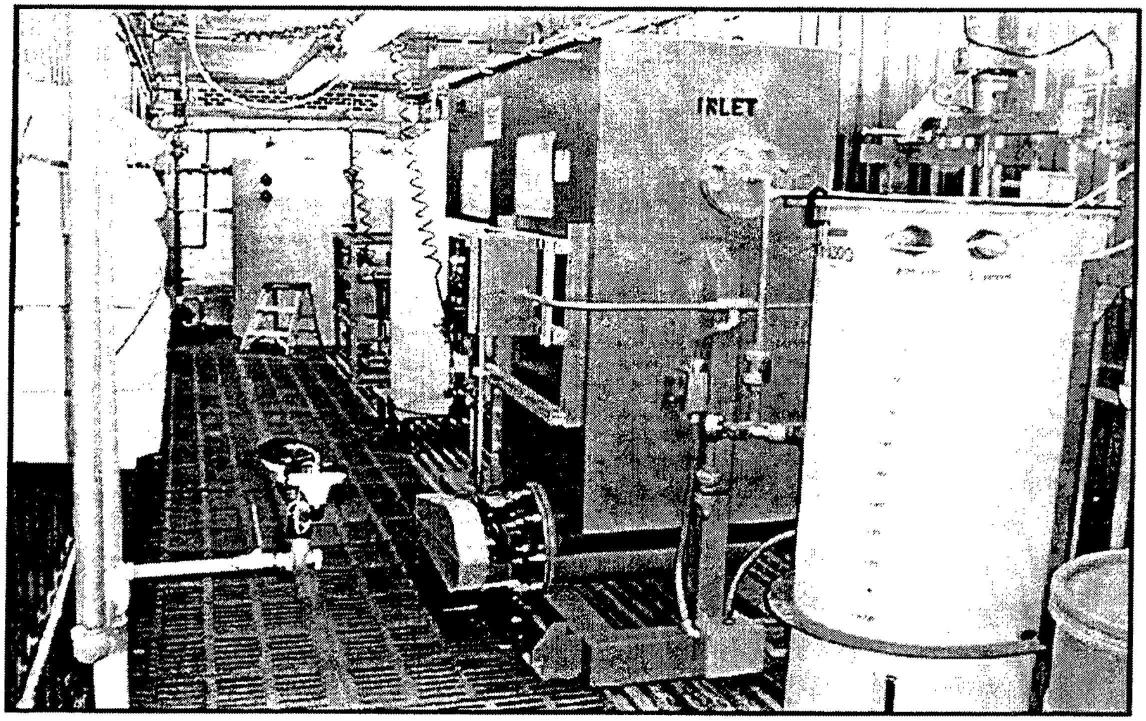

Figure 3. ICB System Components Housed in Trailer Two

The ICB was initially filled with tap water for system checks and verification. Upon startup of the system, enough water was removed to allow addition of 55 gallons of a bacterial inoculum cultured by Allied Signal and 55 gallons of sludge collected from Back River[6] publicly owned treatment works (POTW). During the run, additional sludge from Back River was added to the ICB. The system was started initially in a batch mode to allow bacterial growth and adaptation to the feed stream. The system was allowed a 20-day run-up period before starting the 40-day validation run at the required feed rate of 200-gallons per-day.

2.5 SYSTEM SAMPLING

Two types of samples were taken during ICB operations, process and validation samples. The validation sampling occurred one day per week during the 40-day validation period. These samples were used as the official ACWA endpoints for measuring the technology's overall ability to perform the task of degrading the HD/Tetrytol hydrolysates as well as characterizing each step in the process. In the event that one portion of the technology didn't perform well it could be discerned from the overall performance. These data could be used to identify process deficiencies or strengths that might require further engineering. The data from these samples might also eventually be required for a later scale-up or permitting process, including National Environmental Policy Act (NEPA) and Resource Conservation

Recovery Act (RCRA) compliance. The samples from the validation runs were shipped to independent labs, selected by A. D. Little[7], the study-coordinating contractor, that practiced stringent quality assurance/quality control (QA/QC) verification. Validation sample data was compiled by A.D. Little in a database that included all the technologies in the ACWA demonstration program. Regular validation sample points were established throughout the process. These sample points and the system diagram are represented in Figure 1 by "S" plus number symbols.

Process monitoring samples were taken several times per week when required to monitor biomass growth and efficiency during the run-up period and in order to make adjustments to daily operating parameters. The process monitoring samples allowed for timely turnaround of basic process information. These data were used to adjust operational parameters during the initial 20-day run up period and during the 40-day validation period. The sample points identified for taking validation samples were also used for process monitoring. Analysis of these samples was done onsite using portable Hach kit analysis, GC/MS and some off-site Hach kit analysis.

2.6 PROCESS MONITORING

Process monitoring readings were taken twice daily. These readings included feed levels, feed rate, feed and ICB temperature, CATOX temperature, nutrient solution level, air and exhaust flow rate and recycle water conductivity. Process monitoring samples were taken several times per week. Chemical Oxygen Demand (COD) and pH samples were taken from S1, S3, and S5 (see Figure 1, Table 1), daily. COD, ammonia and phosphate levels were measured using Hach chemical analysis kits onsite. Samples were analyzed for thiodiglycol concentrations using GC/MS.

2.7 VALIDATION SAMPLING

Validation samples were taken 1 time per week on the same day each week during the 40-day validation period. Feed rate was maintained at 40 gallons per-day of HD hydrolysate in a 200-gallon feed batch for 5 days prior to the first validation sample. Samples were collected by ECBC operators and transferred to the ACWA analytical coordinator for packaging and shipment to appropriate labs for analysis. Sample location and analytes are listed in Table 1 below.

Analytes by sample location and type		
S1 Bioreactor Feed	S3 ICB Bioreactor Chamber #1	S5 Bioreactor Outlet
a. Agent HD b. Schedule-2 compounds c. TNT and Tetryl d. Energetics breakdown products e. Agent breakdown products f. VOCs, g. Semi-VOCs, h. Metals' i. Mercury j. Dioxins/ furans' k. Total Organic Carbon (TOC) l. Chemical Oxygen m. Demand (COD) n. Anions: Sulfate, Phosphate o. Nitrite, Nitrate, Chloride	a. Agent HD b. Schedule-2 compounds c. TNT and Tetryl d. Energetics breakdown products e. Agent breakdown products f. VOCS, g. Semi-VOCs, h. Metals i. Mercury j. TOC k. COD l. Anions: Sulfate, Phosphate, m. Nitrite, Nitrate, Chloride	a. Agent HD b. Schedule-2 compounds c. TNT and Tetryl d. Energetics breakdown products e. Agent breakdown products f. VOC'S, g. Semi-VOCs, h. Metals i. Mercury j. TOC k. COD l. Anions: Sulfate, Phosphate m. Nitrite, Nitrate, Chloride
S6 Flocculation Reactor	S7 Clarifier Sludge	S8 Clarifier Overflow
a. Agent HD b. Schedule-2 compounds c. TNT and Tetryl d. Energetics breakdown products e. Agent breakdown products f. VOCs, g. Semi-VOCs h. Metals i. Mercury j. Anions: Sulfate, Phosphate k. Nitrite, Nitrate, Chloride	a. Agent HD b. Schedule-2 compounds c. TNT and Tetryl d. Energetics breakdown products e. Agent breakdown products f. VOC'S, g. Semi-VOCs, h. Metals i. Mercury j. Dioxins/furans k. Anions l. Waste Characterization	a. Agent HD b. Schedule-2 compounds c. TNT and Tetryl d. Energetics breakdown products e. Agent breakdown products f. VOC'S, g. Semi-VOCs, h. Metals i. Mercury j. Dioxins/furans k. Anions l. Aldehydes & ketones, m. Formaldehyde, acetone
S10 RO Retentate Brine	S11 and S12 (ICB offgas and CATOX outlet)	
a. Hazardous Waste characterization b. Ignitability, pH, hydrogen cyanide, hydrogen sulfide, TCLP analysis c. Schedule-2 compounds	a. Agent HD b. Schedule-2 compounds c. Agent hydrolysis products. TNT and Tetryl d. Energetics breakdown products e. VOC'S, f. Semi-VOCs, g. Ammonia h. H_2S i. Dioxins/furans j. HCl/HF k. NO l. SO_x m. Aldehydes and ketones (sample S12 only)	

Table 1. Validation Sample Schedule, Locations, and Analytes

2.8 VALIDATION METHODS FOR CHEMICAL ANALYSIS

a. Method for agent HD: “Systematic Identification of Chemical Warfare Agents, Section B.4. Identification of Non-Phosphorous Warfare Agents", The Ministry of Foreign Affairs of Finland, ISBN 951-46-6386-1, Helsinki, 1982

b. Method for Mustard Degradation Schedule-2 Compounds: “Systematic Identification of Chemical Warfare Agents: B.4. Identification of Precursors of Warfare Degradation Products of Non-Phosphorous Agents, and Some Potential Agents", The Ministry of Foreign Affairs of Finland, ISBN 951-46-6996-7, Helsinki, 1983

c. Method for TNT and Tetryl (Tetrytol and Comp B): ‘EPA Method SW846, Method 8330 for Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC).

d. Method for TNT and Tetryl (Tetrytol and Comp 8): ‘EPA Method SWW, Method 8330 for Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)

e. Method for Mustard Degradation Compounds: ‘Systematic Identification of Chemical Warfare Agents: B.4. Identification of Precursors of Warfare Degradation Products of Non-Phosphorous Agents, and Some Potential Agents’, The Ministry of Foreign Affairs of Finland, ISBN 951-46-6996- 7, Helsinki, 1983

f. Method for VOCs, EPA SW-846, 12196 Revision, 8260B
g. Method for SVOCs, EPA SW-846, 12/96 Revision, 8270C
h. Method for Metals, EPA SW-846, 12/96 Revision, 601 OB
i. Method for Mercury, EPA SW-846, 9/94 Revision, 7470A
j. Method for Dioxins/furans, EPA SW-846, 9194 Revision, 8290
k. Method for Total Organic Carbon, EPA SW-846, 9060
l. Method for COD examination of Water and Wastewater, Method 5220B
m. Method for Anions, EPA SW-846, 9056

3. PROCESS MONITORING DATA AND RESULTS

3.1 FEED RATE

After initial filling and inoculation of the ICB system, the feed rate was varied depending on the response of the inoculum to the feedstock. The first several feedings were done in batch mode; Tetrytol hydrolysate addition was delayed to day 15 due to its late delivery. Reduction in COD levels across the three ICB chambers was monitored for evidence that the ICB organisms were consuming the feedstock. Typically, biomass growth in response to food availability is represented by an increase in COD consumption (reduction in COD level) over a 24-hr period. The goal of the 21-day run-up period was to increase biomass incrementally to result in a 90 percent reduction in COD level (as determined by feed to third chamber effluent sampling) in response to a maximum feeding load of 40-gal per day of HD hydrolysate/1.9 gal per day of Tetrytol hydrolysate. This level of COD reduction, when measured with the Hach kit for process monitoring, is viewed as a good indicator of complete utilization of the hydrolysate as food; the presence of biologically unavailable compounds accounts for remaining measured

COD. Gas chromatography analysis of effluent samples determines actual utilization of thiodiglycol, originating from HD hydrolysate, as food.

The validation portion of the study was designed to follow the 21-day run-up period with continued biodegradation of feedstock at the maximum feeding load of HD hydrolysate. Feed rates (gal per day) are given in Figure 4. Close to schedule, on day 28, the ICB feed mode switched from batch to continuous for a minimum of five days (the hydraulic resonance time for the ICB is five days) prior to the first validation sampling event, in order to have passed the entire 200 gallons of feed through the system before sampling. However, process monitoring samples indicated that the ICB was not keeping up with the planned validation feeding load when COD reduction from the feed to the reactor effluent dropped below the desired 90 percent (Figure 5). Results of validation sample analyses indicated residual thiodiglycol in reactor effluent. The ICB was again placed in batch mode and given no feed until effluent COD levels dropped to near the 90 percent reduction level. During this run in batch mode, an additional 55 gal of sludge collected from Back River, a publicly owned waste treatment works, was added to the ICB. On restart of the continuous feed mode (day 49), the validation feed rate was lowered to 26.6 gal of HD hydrolysate from 40 gal, and 1.1 gal of Tetrytol hydrolysate per day from 1.9 gal in a 200 gal feed volume. This feed schedule was maintained for the remainder of the test.

Adjustments to the feed were performed throughout the test. The feed was supplemented with a micronutrient stream consisting of ammonium bicarbonate and di-potassium hydrogen phosphate. Further discussion of micronutrients appears in section 3.5. Observation of trends in pH values (Figure 4) of feed entering the ICB (as monitored from sample port 1 or S1) indicated a need to buffer effects of both pH variation of HD hydrolysate as well as the contribution of low pH of the recycle water that was added to dilute the feedstock to 200 gal. To that end, upon restart of the continuous mode feeding, sodium bicarbonate was added to the feed to act as buffer. This addition also helped control the pH in the ICB. Further details on pH control are found in section 3.4.

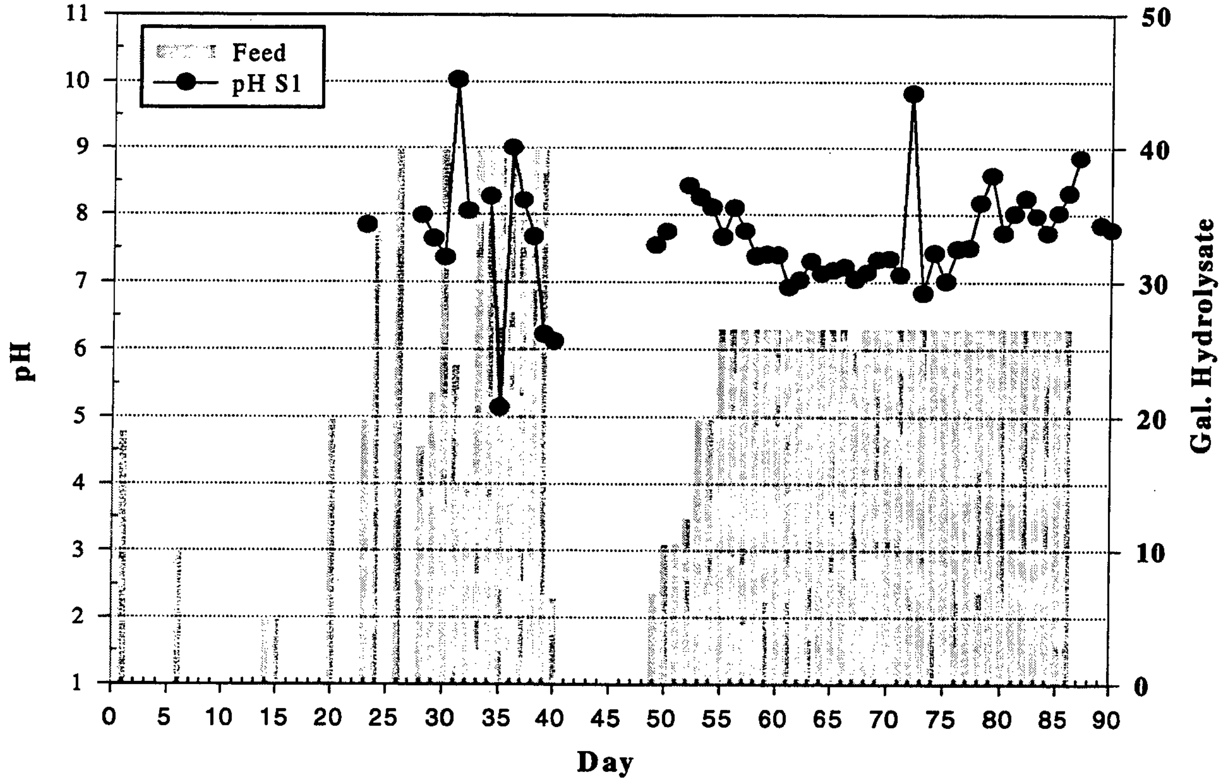

Figure 4. Feed Rate in Gallons Per Day of HD Hydrolysate and pH of Feed from Process Monitoring Samples

3.2 CHEMICAL OXYGEN DEMAND (COD)

COD is a measure of the chemically oxidizable compounds in an aqueous sample. COD was one of the major process parameters used to measure the overall system effectiveness in treating the combined HD/tetrytol hydrolysates. The major sources of COD in the feed and effluent are organics and thiodiglycol. Figure 5 represents COD values over the duration of the test. COD values include those of the feed (S1), ICB chamber 1(S3), ICB outlet (S5) and the COD removal efficiency at the ICB outlet (S5). COD started increasing shortly after initiation of continuous feeding and peaked on day 39. This increase was the leading indicator that the system was not doing well at that point. The load was too high for the amount of biomass that was present in the system. This in part led to a decision to put the ICB in batch mode until a course of action was devised to get the system working more efficiently. By inoculating the system with more sludge, improving pH, control and lowering the load, the system was able to recover. COD removal efficiency at the ICB effluent outlet was above 90 percent for most of the validation run, although the COD did start to rise at the end of the run.

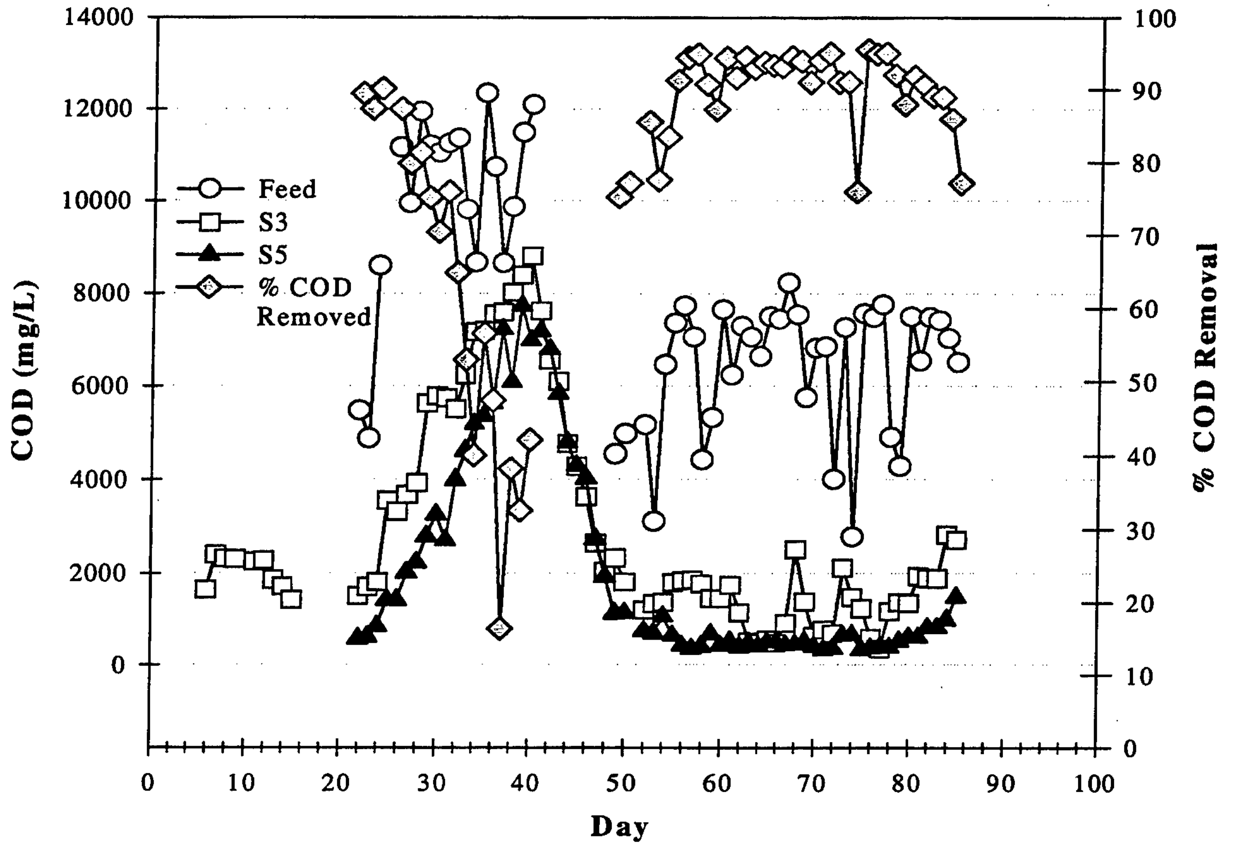

Figure 5. Feed COD and COD Removal Efficiency Values for the HD/Tetrytol ICB

3.3 TOTAL ORGANIC CARBON (TOC)

Total Organic Carbon (TOC) is a measure of the total quantity of organic carbon that is present in an aqueous sample. TOC is a very good process parameter for continuous monitoring and control of bioreactor performance since it can be automated for on-line continuous monitoring of the biological process. Although online monitoring was not available for this project TOC levels were measured weekly (figure 6). In order to more closely monitor organic carbon levels COD values, which is correlated to COD values, was the parameter of choice on a daily or twice daily basis.

COD is measured by the catalytic oxidation of the organic carbon into carbon dioxide and the subsequent measurement of CO_2 by infrared adsorption.

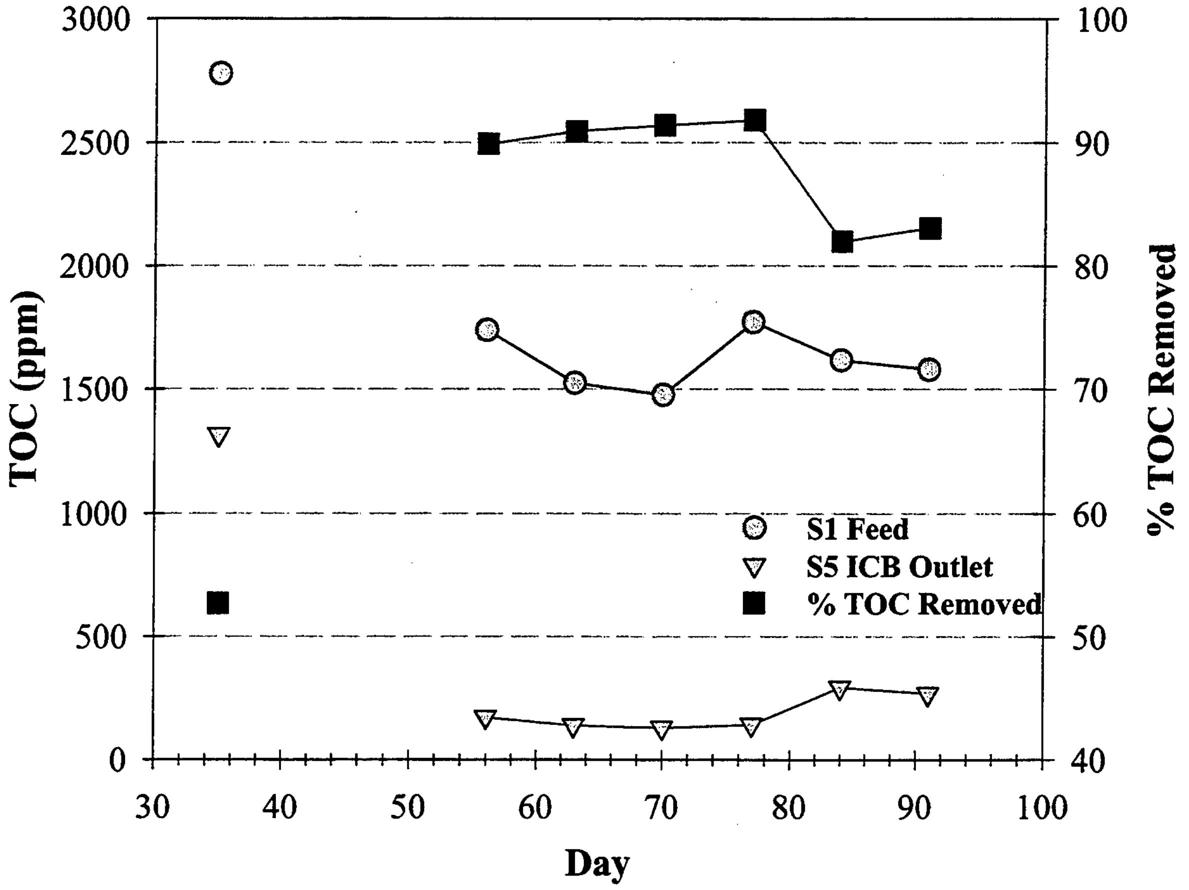

Figure 6. Total Organic Carbon (TOC) Concentration During The ICB Validation

The removal of TOC from within the bioreactor is very similar to that of COD. When the COD in the bioreactor effluent was observed to rise due to unfavorable conditions, TOC also rose. As with COD, the bulk of the TOC was removed in the first chamber of the bioreactor (S3) with further removal or polishing of the TOC occurring in the remaining two chambers. As with the COD, the ICB was capable of removing >90% of the TOC present in the combined energetic and agent hydrolysates. The strong correlation between measured COD and TOC is shown in Figure 7.

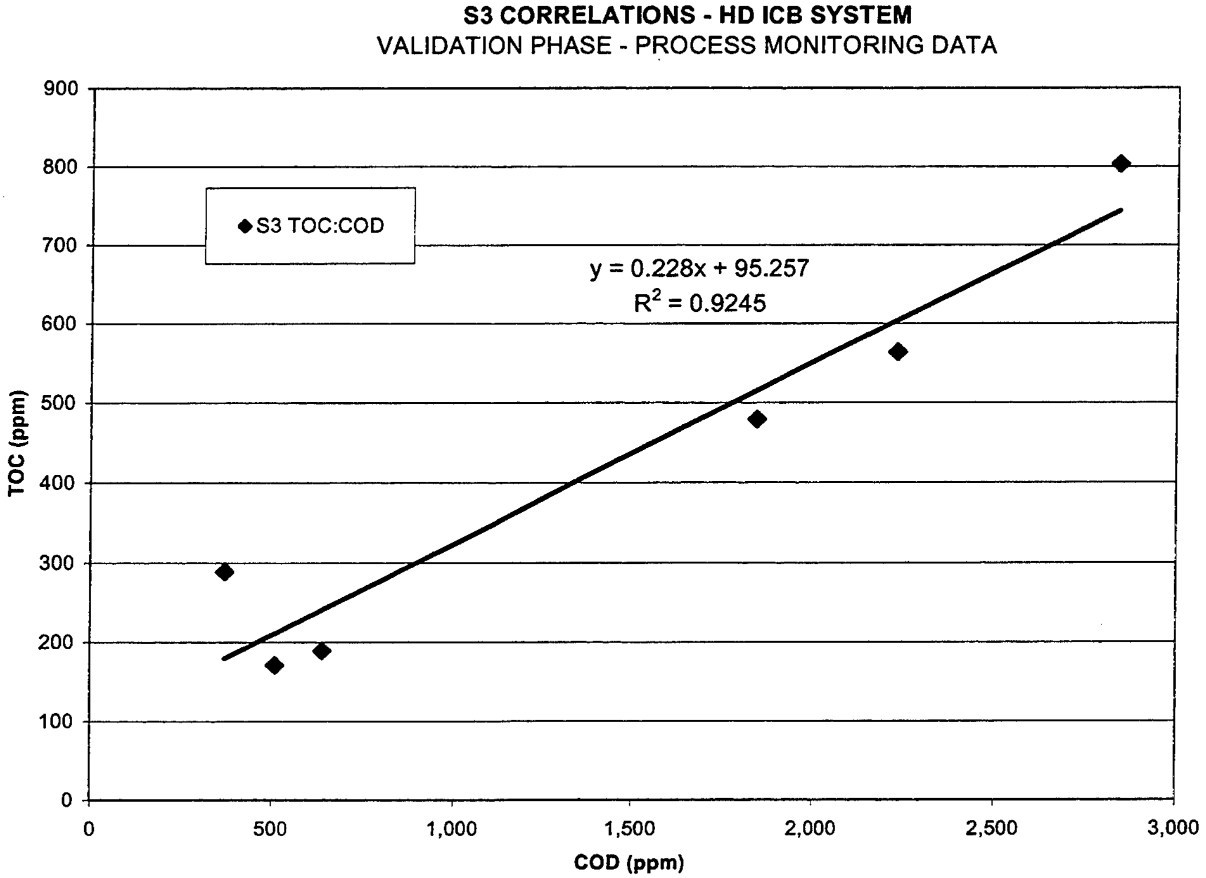

Figure 7. Correlation of COD and TOC Results of ICB Effluent Samples

The COD:TOC correlation for all process monitoring samples was good. The S1 COD/TOC correlation (R^2) is 0.9062, S3 correlation is 0.9245, S5 correlation is 0.9392, and S8 correlation is 0.8802. In the event that one type of analysis is unavailable, a substitution can be made.

3.4 pH

The pH in the ICB was controlled initially by a single pH control loop that added sodium hydroxide to the system whenever the pH reading dropped below 7.5. Process monitoring samples soon showed that this single control loop was unable to effectively control the pH in ICB chambers 2 and 3. The biodegradation of thiodiglycol (TDG) produces an acid that lowers the pH[2]. The pH control was originally only planned for chamber 1 since theoretically, most of the degradation work would be accomplished there, with chambers 2 and 3 simply further polishing the remaining TDG. In practice however, more degradation was accomplished in chambers 2 and 3, thus producing more acidic conditions there. This drop in pH may also have contributed to lower biomass production during run-up and higher COD loading that eventually caused the halt of the validation run. The addition of a second pH loop before restarting the validation test provided more pH control. As a result of this added control the pH in chamber 2 and 3 were more controllable as reflected in the ICB effluent as represented in Figure 8. The pH control continued to be troublesome. Biomaterial frequently collected in the pH probe sensing area caused false readings that were hard to detect. The second pH control loop frequently malfunctioned leading to frequent drops in pH in chambers 2 and 3, in the ICB effluent, and lower system pH toward the end of validation.

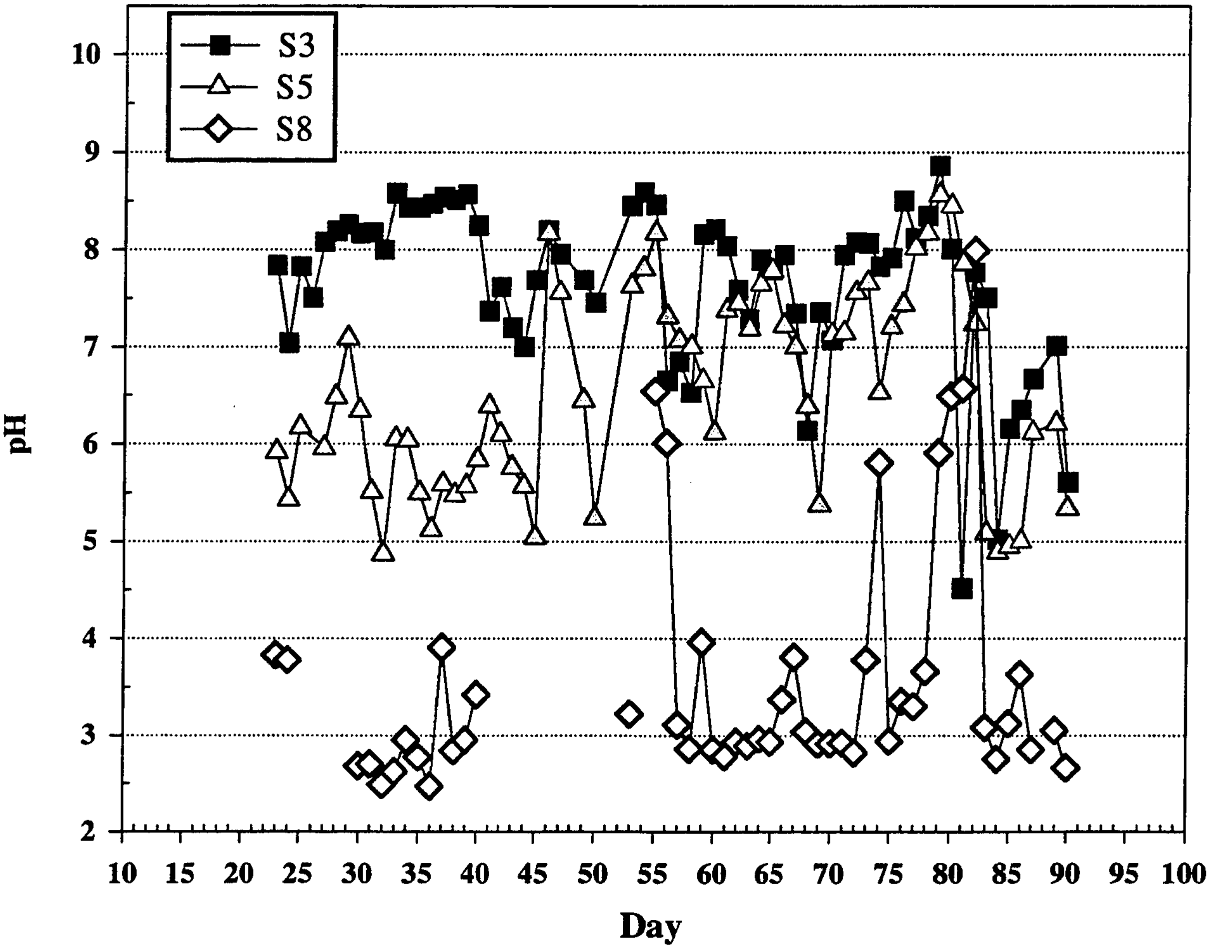

Figure 8. pH of the ICB System at S3, S5, and S8 Sample Locations

Sodium bicarbonate was added to the feed to help buffer the pH of the HD hydrolysate and acidic recycle water. Up to eight pounds of sodium bicarbonate was added to the feed on occasion. While this helped control pH in the ICB, it caused other problems in downstream systems. The Fenton's reactor works best when the pH in that system is around 3.0. The additional sodium bicarbonate in the ICB also helped buffer the pH in the Fenton's reactor causing difficulty with flocculation and eventual spillover of Fenton's reagents into the recycle water tank. The increased bicarbonate also added to the salinity in the recycle water. A compromise of two pounds sodium bicarbonate per day was used for the rest of the validation.

3.5 ICB AMMONIA LEVEL

Ammonia level is an indicator of ICB health. After inoculation ammonia levels can be expected to rise as a result of death of the biomaterial unable to acclimate to the environment and feed stream of the reactor. Nitrogen was added to the reactor as a micronutrient from the mixture in the nutrient tank. Ammonia levels should be controllable to around 10 mg/L in a healthy system. The nitrogen used in the system was changed from ammonium sulfate to ammonium bicarbonate. This helped lower the acid produced in the system, as well as the production of sulfates and SOx. The level of ammonia was decreased and the ammonium bicarbonate replacement also aided in buffering acid production. Figure 9 represents the ammonia concentrations measured during run-up and validation testing.

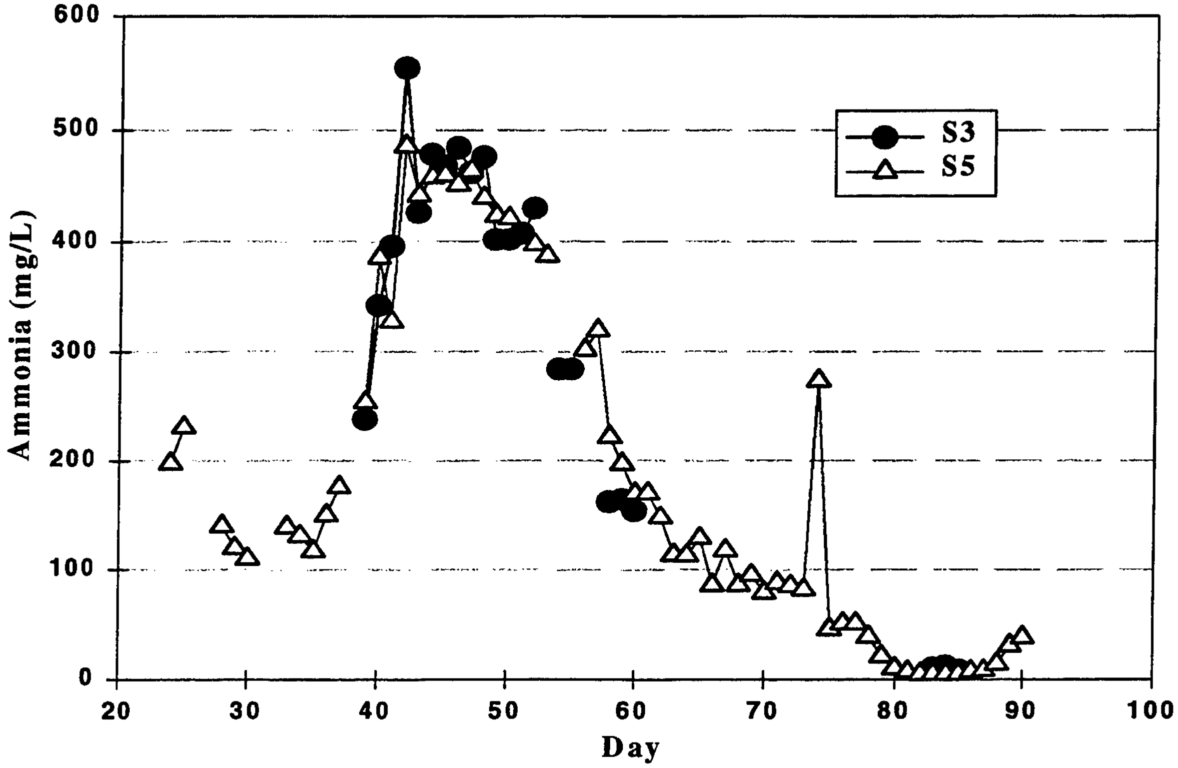

Figure 9. Ammonia Levels in the ICB

3.6 TOTAL SUSPENDED SOLIDS (TSS)

Total suspended solids (TSS) is a measure of the amount of filterable material, both organic and inorganic, present in an aqueous sample. The TSS is important for measuring the health of a bioreactor system and for determining types and amounts of downstream treatment that may be required. It would be somewhat easier to recycle effluent from a system that generates little TSS than from one that generates and discharges large amounts of solids. Since the biomass in an ICB grows on a support it tends to generate little TSS.

Frequently, biomass will slough off of the support material, even in a healthy system. Therefore, a one time per week measurement may not be very representative of the ICB TSS. A system that is stressed may slough large amounts of material suddenly. A sudden increase in TSS within the ICB effluent may indicate a problem within the system. No sudden increases in ICB TSS were noted during the study.

There are also suspended solids in the feed stream that may not be degradable and could contribute directly to the ICB TSS. Figure 10 represents the TSS from the ICB. The average TSS concentration measured at S5 for the validation period was 304 mg/L. This TSS concentration is quite low for a biodegradation system and lends itself well to effluent recycling if an appropriate solids separation can be engineered.

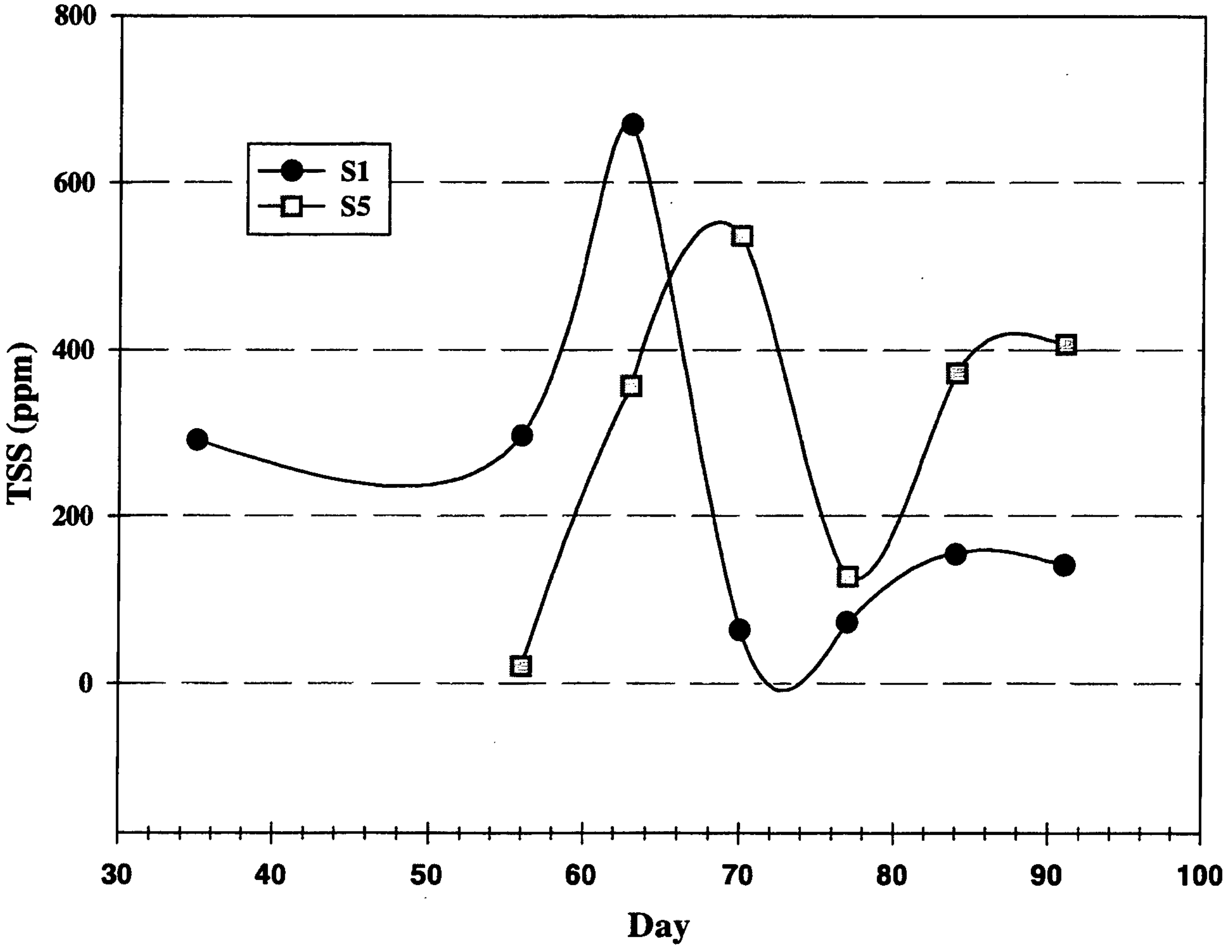

Figure 10. TSS Measurement in the ICB Effluents During Validation

3.7 CONDUCTIVITY

Conductivity is the measure of electrical conductance through an aqueous sample and is related to the concentration of ionizable species dissolved in the sample. It is therefore an indirect measurement of salinity and other conductive dissolved materials in the sample[8]. Measurements for salinity and other dissolved solids were made as part of the validation sampling. It was expected that receipt of validation sampling data may be delayed several weeks so near real-time process monitoring data would be very useful. For this test, the conductivity of various ICB effluents was monitored to track any changes in the total dissolved solids. Changes in conductivity may signal a change in the ICB population health or may help explain noted changes in the culture performance. The appropriate conductivity range for this type of solution has not been previously established. In-house laboratory testing using a 1% NaCl solution in distilled water had a conductivity of 11,600 µS/cm. The most abundant salts in ICB effluents were salts of Na and Cl. The total salt concentrations reported in validation data indicated that salt concentrations never exceeded 1% even though conductivity was as high as 20,000 µS/cm. The higher conductivity readings are assumed to be from combined contributions of salts, metals and other ionizable dissolved solids. During the last week of the run-up and first week of validation COD, TDG and conductivity levels became elevated, resulting in the shutdown and adjustment of operating parameters. The start of the validation period was restarted on day 55 and validation data collected for the first validation week 1 was discarded. The conductivity within the HD/Tetrytol ICB during validation testing is represented in Figure 11. The conductivity of the bioreactor is observed to increase during the first 60 days of the study but reaches equilibrium after about day 65 of the operation. Conductivity increase near the end of the study due to decreased RO operations due to clogged filters and RO system membrane.

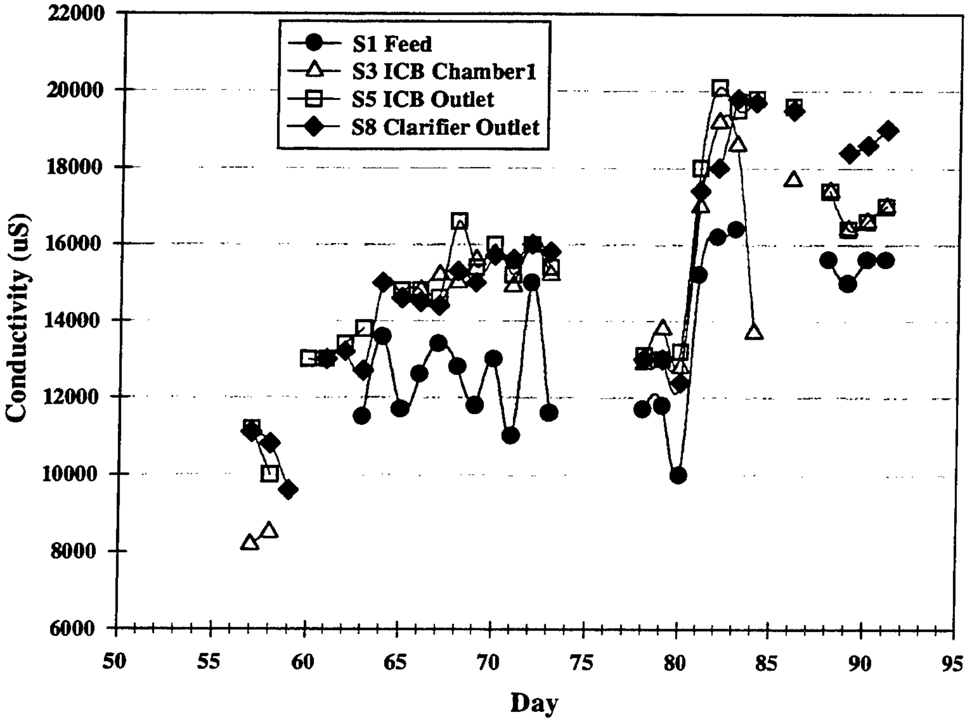

Figure 11. Conductivity of ICB Effluents Measured at 4 Sample Locations

Its undecided if the salt concentrations observed are inhibitory to the ICB culture. Since the sludge used for seeding the ICB came from a freshwater source, the consortium of bacteria can be assumed to be fresh water in nature. Normally freshwater bacteria can tolerate salt concentrations of 1% while others from high saline environments can tolerate salt concentrations to 25%[9]. Since the sludge is so diverse it may contain species that are very salt tolerant as well as salt intolerant. Sludge from a large municipal POTW can also include waste from industrial sources, including bacterial species from high salt and/or high dissolved solids environments.

3.8 TEMPERATURE

Temperature readings in ICB chamber one were taken daily. Cold weather during the winter combined with the low heating capability of the site kept ambient temperatures in the hi-bay between freezing and 50 degrees F. During run-up of the ICB temperature control was a problem. Closure of access ports in the containers housing the system and procurement of electric heaters allowed the system to operate between 70 and 80 degrees F, which are approximate optimal temperatures for this type of system. The temperature profile for the ICB measured in chamber 1 is presented in Figure 12.

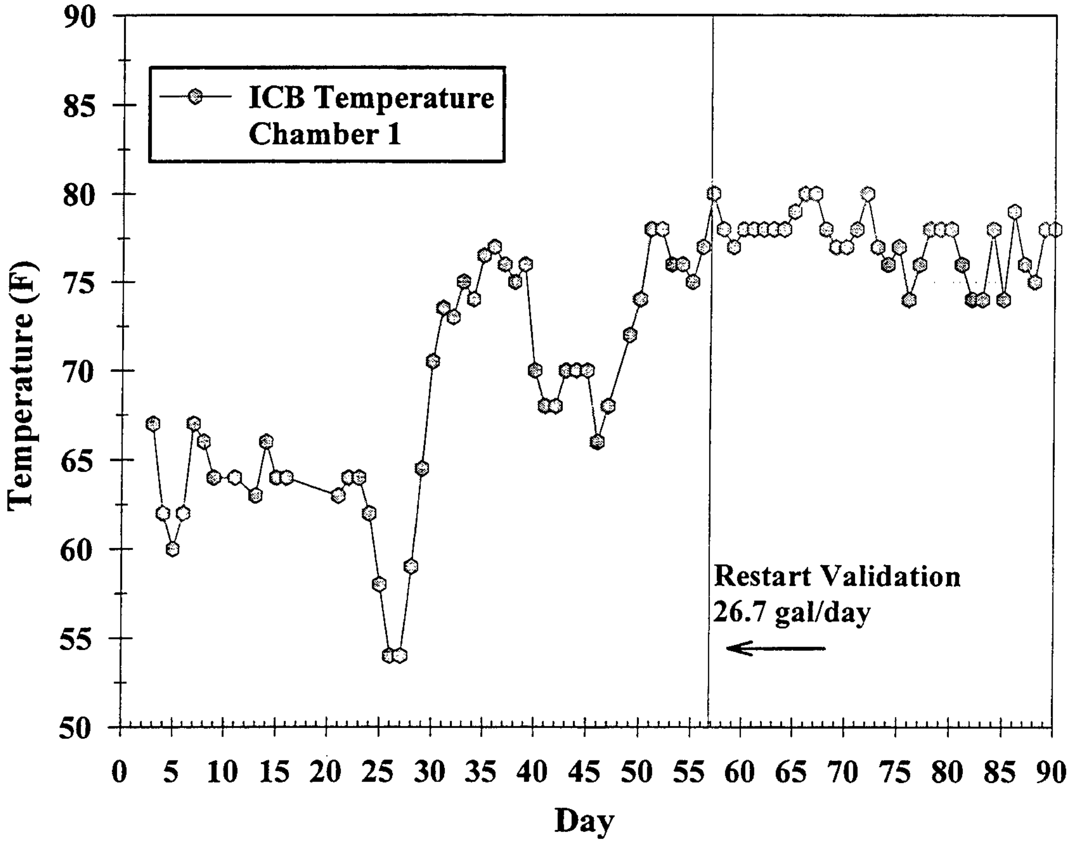

Figure 12. ICB Temperature During The Run-Up and Validation Period

3.9 CATALYTIC OXIDATION

The objective for using a CATOX system is to ensure the elimination of any potential chemical agents and schedule-2 compounds from the exhaust stream. The CATOX pre-heater has the capacity to increase air temperature to 842°F in order to oxidize any impurities. The temperature and volume of the air exhausting from the CATOX was recorded daily. CATOX temperature and flowrate readings are presented in Figure 13. Due to the high free floating solids concentrations in the GB and VX reactors, in Toole, Utah, clogging of the CATOX and lime scrubber was a concern. Daily pressure readings across the HD/Tetrytol ICB CATOX system indicated the pressure drop was less than 0.5 inches of water for the entire run. The HD/Tetrytol ICB experienced no problems with the CATOX system.

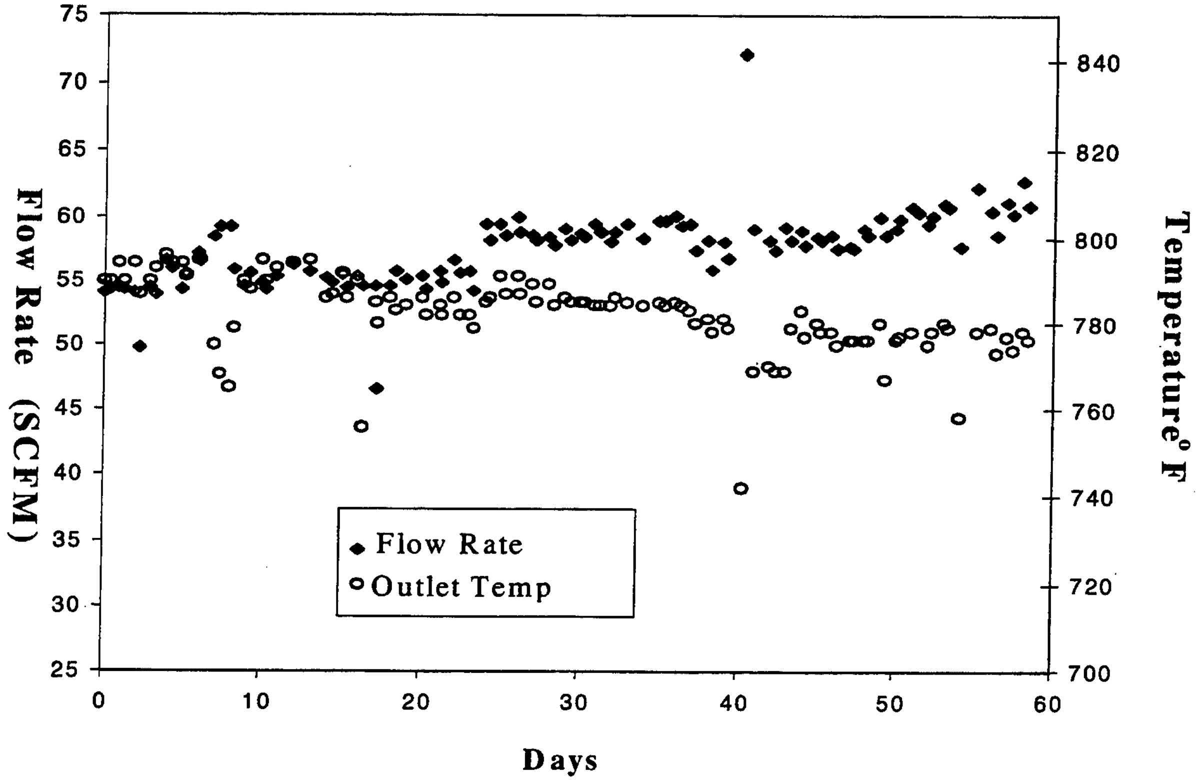

Figure 13. Temperature and Air Flow for the HD/Tetrytol ICB CATOX System

4. VALIDATION DATA AND RESULTS

The validation data was compiled by A.D. Little in a central database. The validation data is used to measure the effectiveness of the ICB system to remove specific compounds like agent, agent breakdown products, and schedule-2 products. From the process analysis data, conditions inside the reactor were optimized for the breakdown process. These operational changes lead to the success or failure in removing those compounds. The data in the sections that follow will show that the ICB was successful at removing all schedule-2 compounds from the system feed and that no agent was reformed during the process. The validation data is also presented to characterize the compounds detected in the feed and effluent streams.

Compounds of other than those of primary concern, such as metals and dioxin production, are not as easily controllable and are more a result of the feed stream components or process waste products. Data on these compounds were collected to characterize the type of output that can be expected from this type of system or a scale up of this process.

4.1 SUMMARY OF HD HYDROLYSATE ANALYSIS

Of primary interest to the ACWA program and Treaty Verification is the destruction of the original agent material. After removal of agent, energetics and explosives from assembled weapons, the component materials are hydrolyzed with water and NaOH. This first step removes much of the toxicity and makes the chemical components biologically available. The hydrolyzation process has been previously explained[2]. As part of validation sampling each batch of the hydrolyzed mustard was analyzed for agent and breakdown products. Analysis of hydrolyzed mustard is summarized in Table 2.

Table 2. Summary Statistics Calculated for Data of Mustard Hydrolysate Batches

	Mean	**Std-D**	**Min.**	**Max.**	
pH	8.25	4.24	1.58	12.84	47
* Thiodiglycol	24060	1324.6	19900	27300	47
Thiodiglycol sulfoxide	99.19	66.21	10.0	285.0	47
1,4-dithane	113.5	46.31	56.0	337.0	47
1,2-bis (2-hydroxyethylthio) ethane	586.9	296.7	287.0	586.9	47
(2-hydroxylethylthio) ethane thiodiglycol	286.7	178.9	85.0	766.0	47
1,2-bis (2-hydroxyethylthio)ethyl ether	703.9	235.2	340.0	1680.0	47
1,4-dithane-1-(2-chloroethane)	76.79	33.84	10.0	156.0	47
Total Organic Carbon	10823	172.9	6060	13500	47

*- The only schedule-2 compound detected.

4.2 TETRYTOL HYDROLYSATE

Data from the tetrytol hydrolysate samples taken during hydrolysate drumming are summarized below in Table 3 below.

Table 3. Summary of the Tetrytol Hydrolysate Characterization Data

Analyte	Result (mg/L)	Lab Qualifier		Analyte	Result (mg/L)	Lab Qualifier
RDX	0.10	<		4-nitrotoluene	0.10	<
Tetryl	0.10	<		2,4-dinitrotoluene	0.10	<
PCP	1.64			2,6-dinitrotoluene	0.10	<
Picric Acid	53.00			3,4-dinitrotoluene	0.49	
Nitrobenzene	0.11	<		2,4,6-Dinitrotoluene	0.07	j
1,3-Dinitrobenzene	0.11	<		2,4,6 Dinitrotoluene	0.06	j
1,3,5-Trinitrobenzene	0.11	<		2,4,6-trinitrotoluene	0.06	j
2-Nitrotoluene	0.10	<		HMX	0.13	<
3-Nitrotoluene	0.10	<		Pentachlorophenol	1.64	
Propanol	0.892	(ug/L)				

Laboratory qualifier "<" equals less than or less than sample detection limit. Laboratory qualifier "j" indicates an estimated value due to poor spike recovery, interference or calibration issues.
See Appendix F for abbreviation identification.

4.3 AGENT AND AGENT BREAKDOWN PRODUCTS

Although no HD was introduced to the ICB feed, all samples taken during the validation period were analyzed for HD to confirm that there was no HD reformation. All sample results indicate HD levels below instrument detection limits of 200 ug/L.

4.4 SCHEDULE-2 COMPOUNDS

Validation samples for schedule-2 compounds were taken on weekly intervals on a designated "validation" sampling day. Of the schedule-2 compounds and HD breakdown products, only thiodiglycol, (schedule-2) thioxane and 1,4-dithane (breakdown products) was ever above analytical detection limits. No other schedule-2 compounds were ever detected in the ICB feed or effluents. The schedule-2 and HD breakdown compounds detected in the ICB effluents are listed in Table 4 below.

Table 4. Schedule-2 and Breakdown Products by Week and Sample Location

Sample Date	Analyte		S3	S5	S6	S10
		(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
March 24	1,4-dithane	15.6	0.7	0.4	ND	ND
	TEP (surrogate)	ND	ND	ND	ND	ND
	*Thiodiglycol	3840	825	ND	ND	ND
	thioxane	24.4	4.9	3.2	1	ND
March 30	1,4-dithane	17.98	5.22	0.52	ND	ND
	TEP (surrogate)	ND	ND	ND	ND	ND
	*Thiodiglycol	3510	55.4	ND	ND	0.299
	thioxane	13.0	4.31	3.28	ND	ND
April 7	1,4-dithane	14.05	2.88	ND	ND	ND
	TEP (surrogate)	ND	ND	ND	ND	ND
	*Thiodiglycol	3310	ND	ND	ND	ND
	thioxane	28.5	6.52	1.15	ND	ND
April 14	1,4-dithane	11.1	1.85	ND	ND	ND
	TEP (surrogate)	ND	ND	ND	ND	ND
	*Thiodiglycol	3257	ND	ND	ND	ND
	thioxane	1	1	ND	ND	ND
April 21	1,4-dithane	8.9	9.43	9.45	ND	ND
	TEP (surrogate)	ND	ND	ND	ND	ND
	*Thiodiglycol	3241	186.3	ND	ND	ND
	thioxane	173.7	6.21	6.27	ND	ND
April 28	1,4-dithane	10.06	9.83	5.05	ND	ND
	TEP (surrogate)	ND	ND	ND	ND	ND
	*Thiodiglycol	3836	12.4	ND	ND	ND
	thioxane	<1	6.96	6.11		

1,4-dithane and thioxane are HD breakdown products
*-thiodiglycol is the only schedule –2 compound

4.5 ENERGETICS AND BEAKDOWN PRODUCTS

Energetic compounds (tetryl and trinitrotoluene) and energetic breakdown products (such as nitro-toluenes or nitrobenzenes) were not detected in the bioreactor, bioreactor effluents or in the feed itself. This indicates that the chemical hydrolysis step was effective at completely degrading these compounds. The products of the energetics and breakdown products were expected to pass through the ICB system. There was some question whether these products would impact the system or reform in any way. Analysis indicates the energetics did not reform and don't appear to have had any negative impact on the biological process. Table 5 indicates the sample detection limit for each analyte. The detection limit is the lower limit at which an analyte can be accurately quantified and discerned from any interference within the sample. Sample detection limits vary for each analyte within a sample and with the number of interfering compounds present. Sample detection limits for process or waste streams are often higher than those for cleaner laboratory solutions.

Table 5. Energetics and Breakdown Products Minimal Detection Limits

Analyte	Concentration (mg/L)					
	S1	S3	S5	S6	S7	S8
1,3,5-Trinitrobenzene	0.11	0.11	0.11	0.11	0.11	0.11
1,3-Dinitrobenzene	0.11	0.11	0.11	0.11	0.11	0.11
2,4,6-Trinitrotoluene	0.1	0.1	0.1	0.1	0.1	0.1
2,4-Dinitrotoluene	0.1	0.1	0.1	0.1	0.1	0.1
2,6-Dinitrotoluene	0.1	0.1	0.1	0.1	0.1	0.1
2,4,6-Dinitrotoluene	0.1	0.1	0.1	0.1	0.1	0.1
2-Nitrotoluene	0.1	0.1	0.1	0.1	0.1	0.1
3-Nitrotoluene	0.1	0.1	0.1	0.1	0.1	0.1
2,4,6-Dinitrotoluene	0.1	0.1	0.1	0.1	0.1	0.1
4-Nitrotoluene	0.1	0.1	0.1	0.1	0.1	0.1
HMX	0.13	0.13	0.13	0.13	0.13	0.13
Nitrobenzene	0.11	0.11	0.11	0.11	0.11	0.11
Picric Acid	25	25	25	25	25	25
RDX	0.1	0.1	0.1	0.1	0.1	0.1
Tetryl	0.1	0.1	0.1	0.1	0.1	0.1

4.6 TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) TEST

The Toxicity Characteristic Leaching Procedure (TCLP) is a USEPA mandated test that evaluates liquids, sludges and solids for toxic characteristics. These characteristics include the presence of certain organic or inorganic contaminants that if detected above a certain regulatory limit, make the sample inherently toxic and therefore hazardous. Waste streams found to have constituents above a states regulatory limit would have to be disposed of in a approved toxic waste landfill or incinerator. The brine (S10) and sludge (S7) generated by the HD/Tetrytol-ICB pilot were analyzed using the TCLP test. None of the TCLP compounds of interest that were detected in the sludge or brine samples were above the regulatory limit that would make the sludge or brine toxic. Therefore, the waste material from the ICB system would be classified as a non-hazardous material based solely on toxic characteristics and would be suitable for disposal in conventional landfills or by disposal through a Toxic Substance Disposal Facility (TSDF). The state of Maryland has a special added provision for determining appropriate disposal methods for process wastes. In Maryland any waste originating from a military chemical agent receives a MD-02 hazardous waste designation regardless of the TCLP results. Waste generated from the HD/Tetrytol pilot-scale operation was disposed of as hazardous waste (MD-02) through the APG TSDF. The eventual site of a full-scale facility will come under the regulatory limits of that states specific requirements for operating and waste disposal permitting.

It should be noted that in a full-scale application of the ICB/CATOX system, these sludges and brines would be dried to greatly reduce their volume and to recover more water for reuse. However, the drying of sludge and brines in a water recovery system greatly reduces the volumes of brine and sludge waste streams, it also concentrates the constituents thereby increasing the likelihood of exceeding the states TCLP regulatory limits. The TCLP results are summarized in Table 6. Full data sets are listed in Appendix A.

Table 6. Summary of TCLP Results

	S7			S10		
Chemical	Mean (µg/L)	Std-D (µg/L)	#(pos/bdl)	Mean (µg/L)	Std-D (µg/L)	#(pos/bdl
2-Butanone	1209.6	1239.5	5/0	374.6	255.4	5/0
Acetaldehyde	600	0	1/1	80	0	1/1
Acetone	200	0	1/0	80	0	1/0
Benzene	70	0	1/3			0/4
Chloromethane	60	0	1/0			0/1
Ethanol	60	0	1/0			0/1
P-xylene	40	0	1/0			0/0
Mercury	0.16	0	1/2	0.11	0	1 / 2
Barium	189.0	134.6	4/0	131.1	124.7	5/0
Cadmium	5	0	1/3			0/4
Chromium	30.4	22.6	5/0	413.5	590.2	4/1
Lead	34.87	13.7	3/1			1 / 4
Selenium	43.9	0	1 / 3	50.6	0	1 / 4
Silver	13.3	12.4	2 / 2	17.05	15.9	2 / 2
Arsenic	101.6	89.0	3 / 1	57.5	13.5	3 / 1
pH	2.85	0.77		3.1		

#(pos/bdl):number of positive detections/number below detectable limits

4.7 SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs)

Semi-Volatile organics represent a class of organic pollutants that have reasonably high vapor pressures or are somewhat polar in nature and are not easily air stripped from solution. Included in this group of compounds are many aromatic compounds like phenols and polynuclear aromatic hydrocarbons (PAHs). Phenolics may be formed during the chemical hydrolysis of the energetic compounds. Some schedule-2 compounds such as 1,4-dithiane and 1,4-thioxane behave as semi-volatile organics. Analysis of the fate of SVOCs in the HD/Tetrytol-ICB show that these compounds were at relatively low concentrations and were frequently removed to below the level of analytical detection in the sludge and brine effluents. The sludge and brine effluents were difficult matrices for the analytical labs to analyze. The lower detection limits for some of the SVOCs were high compared to some environmental samples, as measured by the % recovery of internal standards added during the analytical procedure. Detection limits were similar to many industrial wastewater samples. The SVOC analyses of the bioreactor sludge samples found only very low concentrations. A summary of SVOC concentrations for S1, S5, S7, and S8 is presented in Table 7 below. Complete SVOC data sets are included in Appendix B.

Table 7. Summary of SVOC Data

	S1 (Feed)			S5 (ICB effluent)			S7 (sludge)			S8 (clarifier)		
Analyte	**Mean µg/L)**	**Std-d µg/L)**	**n**	**Mean (µg/L)**	**Std-d (µg/L)**	**n**	**Mean µg/L)**	**td-d µg/L**	**n**	**Mean µg/L)**	**Std-d (µg/L**	**n**
2,2'-Ethanol-Di-Ethanol	4233.33	2490.64	3							8.00		1
2,2'-Ditho-Bis-Ethanol	3700.00	953.94	3				103.07	83.71	3			
2,2'-Thio-Bis-Ethanol	29000.00		1									
1,4-dithiane	2150.00	1202.08	2	621.00	920.96	4				32.00	14.73	3
Dichlorocyclohexane isomer	56.00		1									
1,4-oxathiane	4000.00		1	697.67	1128.12	3	62.00		1	118.00	115.96	2
2,2'-dithiobisethanol	10000.00		1									
Hexadecanoic acid				66.00		1	31.85	15.75	4			
Benzeneacetic acid				200.00		1	30.00		1			
1,2,5-trithiepane							21.00	2.64	3	50.00		1
1,3,5-trithiane							46.50	36.06	2			
Benzaldehyde, 4-methyl							23.00		1	6.00		1
Benzene, 1,3-dimethyl							52.00	25.53	3			
Butonic acid							20.00		1			
Octadecanoic acid							8.90		1			
Oleic acid							47.00	26.85	3			
Ethyl benzene							21.00		1			
P-xylene							69.00		1			
4-methyl phenol							69.00		1	30.00		1
Butonic acid, 2 methyl							66.00		1			
Hydrazine 1,2-dimethyl							59.00		1			
Maltol										4.00		1
1,3-dithiolane										80.00		1
4,4-dioxide, 1,4-oxathiane										90.00		1
4-oxide, 1,4-oxathiane										100.00		1

4.8 VOLATILE ORGANIC COMPOUNDS (VOCs)

Volatile organic compounds represent a class of organic pollutants that have reasonably low vapor pressures and are easily air stripped from solution. Included in this group of compounds are many low molecular weight aromatic compounds like benzene, toluene, ethyl benzene and xylenes (BTEX compounds), low molecular weight ketones, like acetone and 2-butanones, and many chlorinated organics. VOCs are present in the agent and energetic hydrolysates. The major VOCs present in the agent hydrolysate were 1,2-dichloroethane and chloromethane, whereas the major VOCs present in the energetic hydrolysates were the BTEX compounds. One interesting observation was that the Fenton's reaction appeared to produce small quantities of ketones (acetone and 2-butanone). However, these compounds did not build up in the recycled water because they were removed by the biological activity. With the exception of acetone and 2-butanone, the effluent from the bioreactor had < 1 ppm of any VOC compound. A summary of VOC analysis for S1, S5, S7, and S8 are listed in Table 8. Complete tabular data are presented in Appendix C.

Table 8. Summary of VOC Data

	S1			**S5**			**S7**			**S8**		
Analyte	**Mean (µg/L)**	**Std-d (µg/L)**	**n**	**Mean (µg/L)**	**Std-d (µg/L)**	**N**	**Mean (µg/L)**	**Std-d (µg/L)**	**n**	**Mean (µg/L)**	**Std-d (µg/L)**	**n**
1,2-dichloroethane	955.00	167.63	4	10.00		1	10.00	1.41	2	6.00		1
2-butanone	156.67	55.07	3	19.00	14.14	2	1955.00	2043.54	2	464.00	258.22	5
Chloromethane	2300.00	1374.77	3	12.50	4.95	2	320.00		1	21.00	7.07	2
1,4-dithiane	89.33	9.02	3	36.33	46.61	3						
1,4-oxathiane	56.00		1	21.33	16.29	3						
Chloroform	28.00		1									
Acetone	235.00	7.07	2	39.67	37.53	3	940.00	933.38	2	277.50	166.00	4
Acetic acid methyl ester				7.00		1						
Silane				9.00		1						
Xylene							155.50	105.36	2	46.00	23.52	3
2,3-butanedione							77.00	46.67	2			
Acetaldehyde							44.5	12.02	2			
Carbonyl sulfide							7.20		1			
Acetonitrile							14.00		1			
Ethylbenzene							14.00		1	11.00	1.14	2
Carbon disulfide										36.00		1

4.9 ALDEHYDES

Aldehydes are among the by-products formed by microbial metabolism, especially under anoxic or air limiting conditions. They can also be formed as by-products of the Fenton's reaction. Low levels of acetaldehyde and formaldehyde were present in the bioreactor clarifier effluent, as shown in the Table 9.

Table 9. Aldehydes by Week and Sample Location

Sample Date & Location	Acetaldehyde (ug/L)	Formaldehyde (ug/L)	Propanal (ug/L)
3/3 HD S8	15,000	17,000	500
3/24 HD S8			
3/31 HD S8			
4/7 HD S8			
4/14 HD S8			
4/21 HD S8	1,100	2,800	240
4/28 HD S8	770	11,000	74

Analyte detected above the quantitation limit

Like the ketones, acetone and 2-butanone, these compounds did not build up in the recycle water used in the process due to the fact that they are consumed by microbes in the aerobic phase of the biological process.

4.10 DIOXINS AND FURANS

Dioxins and Furans are a class of highly toxic and carcinogenic organic molecules, often formed from other organic molecules at high temperatures. The temperatures used for hydrolysis as well as in the bioreactor were at less than 100°C. The analyses show than dioxins and furans were detectable in a range generally very close to detection limits. The detection is near the limit of quantitation; any hits at these low levels are suspect and are at the level of environmental noise. Several potentially positive hits for dioxins and furans in the ICB feed are well below the EPA Universal Treatment Standard of 63,000 pg/L (40CFR, 268.48). The levels in the bioreactor effluent were generally not detectable, except for a few rare occasions. The levels of dioxins and furans detected are at the limit of detection and are consistent with uncontaminated environments, see "Dioxins and Furans in Bed Sediment and Fish Tissue of the Willamette Basin, Oregon", 1992-1995, US Geological Survey, Water resources Investigations Report 97-4082-D, 1998, USGS. The dioxin and furan results are listed in Table 10.

Table 10. Dioxins and Furans by Weeks and Sample Location

Analyte March 3,1999	**S1 (pg/L)**	**S7 (pg/L)**	**S8 (pg/L)**	**March 24, 1999**	**S1 (pg/L)**	**S7 (pg/L)**	**S8 (pg/L)**
1,2,3,4,6,7,8-HPCDD	38.3	63.1		1,2,3,4,6,7,8-HPCDD	23.7	6.2	4.4
1,2,3,4,6,7,8-HPCDF	13.8	32.7		1,2,3,4,6,7,8-HPCDF	65.9	3.9	2.0
2,3,4,6,7,8-HXCDF	23.7	7.5		2,3,4,6,7,8-HXCDF	41.6	5.5	3.2
OCDD	73.1	592.5		OCDD	171.0	66.7	41.1
OCDF	195.8	72.9		OCDF	643.4	4.1	3.2
TOTAL HEPTA-DIOXINS	38.3	63.1		TOTAL HEPTA-DIOXINS	23.7	6.2	4.4
TOTAL HEPTA-FURANS	13.8	42.9		TOTAL HEPTA-FURANS	65.9	3.9	2.0
TOTAL PENTA-FURANS	20.8	8.3		TOTAL PENTA-FURANS	15.9	3.0	2.2
Analyte April 7, 1999	**S1 (pg/L)**	**S7 (pg/L)**	**S8 (pg/L)**	**April 14, 1999**	**S1 (pg/L)**	**S7 (pg/L)**	**S8 (pg/L)**
1,2,3,4,6,7,8-HPCDD	34.2		10.3	1,2,3,4,6,7,8-HPCDD	11.3	6.1	
1,2,3,4,6,7,8-HPCDF	11.1		5.3	1,2,3,4,6,7,8-HPCDF	5.2	3.4	
2,3,4,6,7,8-HXCDF	**21.1**		5.0	2,3,4,6,7,8-HXCDF	9.7	5.9	
OCDD	37.7		10.5	OCDD	14.9	7.8	
OCDF	**90.9**		12.0	OCDF	11.2	5.7	
TOTAL HEPTA-DIOXINS	34.2		10.3	TOTAL HEPTA-DIOXINS	11.3	6.1	
TOTAL HEPTA-FURANS	11.1		5.3	TOTAL HEPTA-FURANS	5.2	3.4	
TOTAL PENTA-FURANS	21.0		5.3	TOTAL PENTA-FURANS	12.2	5.1	
Analyte April 21, 1999	**S1 (pg/L)**	**S7 (pg/L)**	**S8 (pg/L)**	**April 28, 199**	**S1 (pg/L)**	**S7 (pg/L)**	**S8 (pg/L)**
1,2,3,4,6,7,8-HPCDD	20.6	19.8	7.8	1,2,3,4,6,7,8-HPCDD	12.4		12.0
1,2,3,4,6,7,8-HPCDF	13.3	11.7	6.1	1,2,3,4,6,7,8-HPCDF	7.3		7.5
2,3,4,6,7,8-HXCDF	12.2	8.8	9.2	2,3,4,6,7,8-HXCDF	8.3		9.9
OCDD	15.6	13.3	15.9	OCDD	11.1		14.2
OCDF	62.9	11.3	12.6	OCDF	14.0		19.4
TOTAL HEPTA-DIOXINS	20.6	19.8	7.8	TOTAL HEPTA-DIOXINS	12.4		12.0
TOTAL HEPTA-FURANS	13.3	11.7	6.1	TOTAL HEPTA-FURANS	7.3		7.5
TOTAL PENTA-FURANS	15.8	7.8	8.3	TOTAL PENTA-FURANS	9.3		10.0

Analyte detected above the quantitation limit

Values not highlighted are the sample detection limit.

*- chemicals acronyms for dioxins and furans are listed in Appendix F

4.11 METALS

The only metals detected in the bioreactor and effluent samples consistently above a concentration of 1 ppm were calcium, iron, magnesium, potassium and sodium. All these metals were either added as nutrients (calcium, magnesium and potassium) or were added as part of the flocculation chemistry (iron) or through the pH control system (sodium). All the toxic metals, such as arsenic, chromium, copper and mercury were present at low concentrations if detected at all. It appears that the Fenton's reaction caused a slight increase in metal concentrations, probably by releasing metals entrapped by microbial biomass. The results from the metals analysis are summarized in Table 11. Tabular data are presented in Appendix D.

Table 11. Summary of Metals Data for ICB Effluent

Metals	S1		S5		S6		S7		S8	
	(mg/L)	(std-d)	(mg/L)	(std-d)	(mg/L)	(std-d)	(mg/L)	(std-d)	(mg/L)	(std-d)
Aluminum	3.39	7.33	4.75	7.25	4.03	7.08	11.40	8.38	3.50	7.29
Antimony	0.05	0.01	0.07	0.03	0.07	0.02	0.48	0.65	0.05	0.03
Arsenic	0.14	0.17	0.14	0.18	0.14	0.17	0.38	0.28	0.14	0.18
Barium	0.04	0.01	0.03	0.02	0.02	0.01	0.09	0.07	0.01	0.01
Calcium	15.13	2.46	16.77	2.80	20.01	4.05	62.03	101.44	19.35	2.82
Chromium	0.14	0.06	0.03	0.03	0.08	0.05	0.28	0.29	0.06	0.03
Cobalt	0.02	0.00	0.02	0.00	0.02	0.01	0.07	0.08	0.02	0.01
Copper	0.07	0.03	0.04	0.03	0.05	0.03	0.27	0.24	0.03	0.02
Iron	19.28	10.40	13.10	9.58	149.76	53.95	568.83	287.71	71.86	23.22
Lead	0.07	0.00	0.07	0.00	0.07	0.00	0.07	0.00	0.07	0.00
Magnesium	6.10	1.01	6.52	1.86	7.18	1.95	21.08	31.14	6.92	1.65
Manganese	0.42	0.22	0.32	0.22	1.58	0.52	2.72	2.99	1.38	0.37
Mercury	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Molybdenum	0.03	0.00	0.03	0.00	0.03	0.00	0.03	0.00	0.03	0.00
Nickel	0.21	0.06	0.09	0.10	0.20	0.07	0.46	0.37	0.18	0.06
Phosphorous	9.93	2.80	22.20	8.44	46.06	63.97	106.30	99.95	1.75	0.91
Potassium	91.24	56.43	148.43	19.88	143.77	9.89	655.67	1251.44	147.27	29.20
Sodium	3027.14	1015.61	4052.86	466.36	4082.86	562.90	11565.00	18688.15	4054.29	366.65
Thallium	0.09	0.00	0.10	0.02	0.10	0.01	0.50	0.71	0.09	0.01
Tin	0.06	0.02	0.06	0.02	0.08	0.02	0.43	0.25	0.06	0.02
Zinc	0.58	0.22	0.48	0.19	0.98	0.50	1.45	1.25	1.77	1.39
Selenium	0.04	0.00	0.04	0.00	0.04	0.00	--	--	0.04	0.00
Silver	0.01	0.00	0.01	0.00	0.01	0.00	--	--	0.01	0.00
Vanadium	0.02	0.01	0.02	0.01	0.02	0.01	0.03	0.00	0.02	0.01

4.12 CYANIDE AND SULFIDE

Cyanide and sulfide can become toxic to microorganisms over a threshold value. Samples from the outfall of the ICB system were analyzed for Cyanide and Sulfide as part of waste stream characterization. Cyanide was not detected in any of the sludge or brine samples analyzed. Sulfide was detected in some sludge (s7) and brine samples (S10) as indicated in Table 12.

Table 12. Results of Cyanide and Sulfide Analysis by Week for Sample Locations S7 and S10

Sample ID	Unit Operation	H2S mg/kg	CN mg/kg	pH
3/3 HD S7 3/3 HD S10	Clar Sludge Brine			
3/24 HD S7 3/24 HD S10	Clar Sludge Brine	<24	<0.5	3.1
3/31 HD S7 3/31 HD S10	Clar Sludge Brine			
4/7 HD S7 4/7 HD S10	Clar Sludge Brine		<0.5 <0.5	
4/14 HD S7 4/14 HD S10	Clar Sludge Brine	28.1 40.1	<0.5 <0.5	3.6 3.5
4/21 HD S7 4/21 HD S10	Clar Sludge Brine		<0.5 <0.5	
4/28 HD S7 4/28 HD S10	Clar Sludge Brine	lost	<0.5 <0.5	

4.13 ANIONS

Anions such as chloride, sulfate, nitrate and nitrite enter the ICB system as part of the feed (chloride, nitrate and nitrite) or as part of either the nutrients (sulfate), or are formed during breakdown of the hydrolysate components (e.g. sulfate from TDG). The analyses of anions in the HD/Tetrytol- ICB are listed in Table 13 below.

The major anions were chloride and sulfate, as expected. Chloride is formed during the hydrolysis of HD and sulfate is added as part of the nutrients (di-ammonium sulfate) and is formed from the biodegradation of TDG. To help reduce the amount of sulfate added to the system, the di-ammonium sulfate was replaced with ammonium bicarbonate during testing. During the 40-day validation test, chloride accumulated to a concentration of 3,800 mg/L and sulfate to a concentration of 5,200 mg/L. The build up of anions did not effect organics removal by the bioreactor. Other anions such as phosphate, nitrite and nitrate were either non-detectable or were present at low concentrations. The amount of phosphate added as a nutrient was adequate for promoting biological activity but was consumed during the process so as not to contribute to the build up of salt in the system.

Table 13. Anions by Week and Sample Location

Sample ID	Unit Operation	Cl mg/L	SO4 mg/L	PO4 mg/L	NO2 mg/L	NO3 Mg/L
3/3 HD S1	Feed	4650	2640	144	250	37.00
3/3 HD S3	ICB	4000	3670	147	250	49.00
3/3 HD S5	ICB eff	3810	3890	115	250	28.00
3/3 HD S6	Fnt's Rx	3300	3960	25	250	25.00
3/3 HD S7	Clar Sludge					
3/3 HD S8	Clar Eff	3200	3640	25	250	25.00
3/24 HD S1	Feed	2680	517	19.6	25	12.00
3/24 HD S3	ICB	2340	2980	53.6	25	2.50
3/24 HD S5	ICB eff	2840	4530	57	25	2.50
3/24 HD S6	Fnt's Rx	2870	4950	6.5	25	3.20
3/24 HD S7	Clar Sludge	3630	7880	6.9	4.2	4.10
3/24 HD S8	Clar Eff	2980	5190	13.3	25	2.50
3/31 HD S1	Feed	3490	1710	28.2	25	14.00
3/31 HD S3	ICB	3420	5070	26.2	25	2.50
3/31 HD S5	ICB eff	3360	5060	35.6	25	2.50
3/31 HD S6	Fnt's Rx	3000	5020	2.5	25	8.30
3/31 HD S7	Clar Sludge					
3/31 HD S8	Clar Eff	3150	5200	2.5	25	8.40
4/7 HD S1	Feed	3280	1980	17.3	25	15.50
4/7 HD S3	ICB	3490	4880	6.1	25	2.50
4/7 HD S5	ICB eff	3440	5290	19	25	2.50
4/7 HD S6	Fnt's Rx	3450	5610	2.5	25	9.10
4/7 HD S7	Clar Sludge	3350	6750	2	3.7	2.20
4/7 HD S8	Clar Eff	3250	5180	2.5	25	16.00
Week of 4/14		**No**	**Data**	**Available**		
4/21 HD S1	Feed	3420	1800	9	25	12.70
4/21 HD S3	ICB	3950	4020	40.1	25	10.10
4/21 HD S5	ICB eff	3820	4960	57.6	25	57.60
4/21 HD S6	Fnt's Rx	3710	5360	2.5	25	2.50
4/21 HD S7	Clar Sludge	3610	6650	3.9	2	2.00
4/21 HD S8	Clar Eff	3450	5390	2.5	25	2.50
4/28 HD S1	Feed					
4/28 HD S3	ICB	3570	3900	33.7	25	8.00
4/28 HD S5	ICB eff	3570	3900	33.7	25	8.00
4/28 HD S6	Fnt's Rx	3890	4880	35.3	25	3.22
4/28 HD S7	Clar Sludge	3630	5100	2.5	25	13.00
4/28 HD S8	Clar Eff	3790	5170	2.5	25	6.20

Analyte detected above the quantitation limit

4.14 CATALYTIC OXIDATION (CATOX)

Since HD agent and schedule-2 compounds have low vapor pressures, and no HD was introduced into the ICB, very little of these compounds were anticipated in the ICB exhaust. The CATOX emissions were analyzed for agent and schedule-2 compounds. Almost all compounds were below detectable limits. Values for these compounds are listed in Table14.

Table 14. Results of HD and Schedule-2 Analyses of CATOX Inlet and Outlet Samples

Compound	**Result**	**Detection Limit (ug/M^3)**
2-chloroethyl (2-chloroethoxy) ethyl sulfide	ND	0.21
2-methyl-1propene	ND	0.21
Bis (2-chloroethyl) disulfide	ND	0.21
Bis (2-chloropropyl) sulfide	ND	0.21
2-chloroethyl-4-chlorobutyl sulfide	ND	0.21
2-chloroethyl 3-chloropropyl sulfide	ND	0.21
1,4 dithiane	1 pos, 3 ND	0.21
Divinyl sulfide	ND	0.21
Bis (2-chloroethyl) trisulfide	ND	0.21
1, 2-Bis(2-chloroethylthio) ethane	ND	0.21
1, 4 Oxathiane	1 pos, 3 ND	0.02

Analysis for schedule-2 compounds listed above revealed 2 positive hits occurring on 28 April 99, the last day of validation testing and sampling. Positive hits were both for the inlet side of the CATOX system at sample port S11. No positive hits were detected on the outlet side of the CATOX. The complete set of data for exhaust gas monitoring is available as Appendix E.

The 2 compounds detected were:
1, 4 Dithiane at 9 ug/M^3
1, 4 Oxathiane at 117 ug/M^3

4.15 CALCULATION OF SOLIDS YIELD

The yield of solids in the HD-ICB is the difference between the amount of solids leaving the system compared to the amount of solids entering the system. The solids yield reveals the amount of biomass generated by the ICD system. Since this boimass may require further processing and/or disposal, calculation of the yield is important for process scalability and logistics.

The concentration of TSS in the ICB effluents was measured weekly as part of the validation testing. Values are presented in Table 15 below.

Table 15. Results of Total Suspended Solids Analysis

HD-ICB-TSS DATA (ppm)	**Input (ppm)**	**Outputs (ppm)**	
Sample Date	**S1 Feed**	**S7 Sludge**	**S8 Clarifier**
3-March	292	5467	11
24-March	297		312
31-March	670	1610	5
7-April	64		19
14-April	73		18
21-April	155	930	34
28-April	142	11,200	276
AVG:	242	4,802	96
STD:	211	4,710	136

The daily flow of feed was 200 gallons (757.58 liters) per day. The flow of sludge was 25 gallons (94.7 liters) per day and the flow from the clarifier was 175 gallons (662.9 liters) per day. Therefore, the mass of solids in the feed per day was:

$$242 \text{ mg/L} \times 757.6 \text{ liters} = 183{,}339 \text{ mg } (183.34 \text{ g})$$

The mass of solids in the effluents from the ICB per day was:

$$4{,}802 \text{ mg/L} \times 94.7 \text{ liters} = 454{,}749 \text{ mg } (454.75 \text{ g})$$

$$96 \text{ mg/L} \times 662.9 \text{ liters} = 63{,}638 \text{ mg } (63.64 \text{ g})$$

The average daily solids yield across the HD-ICB was therefore:

$$[454.75 + 63.64] - 183.34 = 335.2 \text{ g/Day}$$

The production of solids was associated with a concurrent consumption and removal of organic material (TOC). The average TOC removal across the ICB system is listed in Table 16.

Table 16. Results of Total Organic Carbon Analysis

HD-IDC TOC Data (mg/L)	**Input (mg/L)**	**Output (mg/L)**	
Sample Date	**S1 Feed**	**S7 Sludge**	**S8 Clarifier**
3-March	2,778	1,007	936
24-March	1,740	259	151
31-March	1,525	64	63
7-April	1,477	75	76
14-April	1,772	na	86
21-April	1,618	98	98
28-April	1,580	195	174
AVG:	1,619	138	108
STD:	117	85	44

Therefore, the removal of TOC across the system is:

Input 1,619 mg/L x 757.58 liters = 1,226,522 mg (1,226.52 g)

Output 138 mg/L x 94.7 liters = 13,068 mg (13.07 g)

108 mg/L x 662.9 liters = 71,593 mg (71.594 g)

TOC Consumption

1,226.52 – [13.07 + 71.59] = 1,141.86 g/Day

The total sludge yield/organics consumed ratio is therefore:

(avg. daily solids/TOC consumption)

335.2/1,141.86= 0.293

The total sludge yield incorporates both the biomass component of sludge as well as the inorganic component such as metals (especially iron) and particulates, such as carbon particles. The biomass yield can be obtained from determining the amount of Volatile Suspended Solids (VSS) generated across the ICB system relative to the amount of TOC consumed. This data is presented in Table 17.

Table 17. Results of Total Volatile Suspended Solids Analysis

HD-IDC VSS Data (mg/L)	**Input (mg/L)**	**Output (mg/L)**	
Sample Date	**S1 Feed**	**S7 Sludge**	**S8 Clarifier**
3-March	250	2933	7
24-March	213		97
31-March	580	2856	5
7-April	45		13
14-April	50		10
21-April	126	482	13
28-April	102	6950	55
AVG:	195	3,305	29
STD:	186	2,683	35

Therefore, the mass of biomass solids (VSS) in the feed to the bioreactor was:

195 mg/L x 757.58 liters = 147,728 mg (147.73 g)

The mass of biomass solids in the effluent from the ICB was:

3,305 mg/L x 94.7 liters = 312,983 mg (312.98 g)

29 mg/L x 662.9 liters = 19,224 mg (19.22 g)

Biomass generated in the ICB was:

[312.98 + 19.22] – 147.73 = 184.47 g/day

The biomass yield/TOC consumed for the ICB was therefore:

184.47 / 1,141.86= 0.16

The ratio of biomass yield/TOC consumed for the ICB (0.16) compares favorably with ratios of biomass yield/TOC consumed for other biological processes, which typically have values between 0.3 and 0.4.

For the validation period 23 March through 27 April the combined RO brine and sludge waste generated, averaged 107.3 gallons/day (944.24 lbs). The amount of ICB effluent recycled to the feed system averaged 46.3 percent for the period. The total liquid waste generated by the HD/Tetrytol ICB for the demonstration study including decontamination solution was 79,900 lbs.

5. DISCUSSION: Problems Encountered

No significant problems were encountered during systemization. However, during the work-up and validation period of the test, problems arose that required corrective actions. The pump that transfers feed from the feed tank to the ICB was very sensitive to head pressure. At first, regulation of the flow to uniformly deliver 200 gallons of feed in 24 hours was nearly impossible. The delivery of 200 gallons of feed often took longer than 24 hours, followed by delivery in less than 24 hours. Re-plumbing, additional valve installation followed by trial and error finally resulted in a valve setting that delivered the 200 gallons of feed in 24 hours. A redesign of the pumping system is required in order to eliminate the effects of head pressure.

ICB inoculation and the beginning of work-up started smoothly. A change in the weather to very cold conditions for Maryland strained the test site heating capabilities. Temperature in the system containers and the temperature of the ICB fell below an optimum range for cell growth. After closure of access holes to allow CATOX sample port installation and procurement of heaters for inside the shipping container, system temperatures became more controllable and more suited for optimal growth conditions.

With temperature restored work-up of the biomass proceeded however low pH in chambers two and three was soon discovered. The capacity of the pH control loop in chamber one and the absence of pH control in chambers two and three were quickly overwhelmed. In theory most of the biodegradation was to be accomplished in chamber one. Perhaps due to insufficient run up and a short schedule, the biomass in chamber one could not handle the level of carbon introduced into the system. Therefore, more acid was produced in the second and third chambers than anticipated. With the addition of both sodium bicarbonate to the feed and a second pH control loop that removed effluent from chamber three, added NaOH and returned to chamber two, the pH became controllable. There were frequent breakdowns in the pH control loops caused by air pockets and biomass clogging lines and pH probes. The pH control systems had to be monitored closely to detect malfunctions.

A return to batch mode was forced when it became obvious that the ICB was being overfed. This overfeeding was brought about by the temperature and pH problems in addition to the tight schedule desired by ACWA. When the ICB was allowed to recover, a decision was made to run at 2/3 the feed strength to shorten the ICB recovery time and to fit several validation runs into the schedule before the May 1 deadline.

While making adjustments for pH control, too much bicarb was added to the ICB. Actually the bicarbonate level was appropriate for the ICB, but caused trouble downstream with the Fenton's reaction. Buffering capacity in the ICB effluent to keep the pH of the Fenton's reactor too high to allow flocculation of the biomass. Therefore, some biomass flowed into the water recycle tank and the RO system. Once the amount of bicarbonate added to the feed was set at two pounds, the ICB pH was controllable and the Fenton's reactor was not affected.

Once on-line, the ICB and the Fenton's reactor operated as engineered. However, the RO system did not. The rise in salt concentrations forced additional RO operation and shortened the life of the RO membrane, requiring frequent membrane replacement. Instead of recycling the desired 65% effluent each day only 50% recycle was maintained. The inability of the RO to keep up must have been due to higher than expected salt concentrations in the effluent from increased pH control. Unless a better approach to salt control is available, use of the RO system will not be the best approach to allow effluent recycling.

Although not apparent during the validation run, after receipt of validation data a problem with the clarifier was suspected. The TSS levels reported had large swings in concentration. Since the sludge pump did not run fulltime but instead was on an automated timer, the sludge available at the time of validation sampling may not have been representative or consistent. There may have been an intermittent blockage or the order of sampling and analysis for TSS may have affected the consistency of the samples. This average TSS value with a very large standard deviation has been used for calculations of mass balance, TOC consumption ratios and yields. Future sampling for TSS should be taken from a system that is operated continuously, like the Fenton's reactor or the ICB out fall to ensure a more representative sample.

A potential problem in the recycle process was the formation of formaldehyde, probably as a result of the Fenton's reaction. Although not specifically identified as a problem, the formaldehyde concentration in the recycle water was high in the validation sample taken 3 March 99, shortly before suspension of the validation run. Formaldehyde again increased to a similar level by the 28 April validation sample (Table 9). An increase in formaldehyde concentrations could reduce biomass activity and its ability to continue degradation of schedule-2 compounds should they reach toxic concentrations. Even though schedule-2 compound degradation was not effected, COD levels did rise near the end of the study, possibly indicating accumulation of non-degradable compounds in the system. One could speculate that a longer validation period might have shown a greater accumulation of non-degradable compounds that could have eventually caused deterioration of ICB performance. A follow-on study of longer duration may be warranted to address issues related to sustainable performance of the ICB and all supporting systems.

6. CONCLUSION

The basic ability of microorganisms cultured from activated sludge to degrade HD hydrolysate under laboratory and bench scale studies was again tested in the 1000-gallon pilot scale ICB system. While laboratory and bench scale studies of this system worked well, it can't be automatically assumed that if scaled up properly, any pilot scale biological system will work as efficiently as its laboratory predecessor. While this ability has been previously demonstrated in SBRs using HD hydrolysate as feedstock, the addition of energetic materials and the proposed utility of an ICB system necessitated a demonstration study of this type.

In addition to the challenge of scaling-up a biological system, what would be the effect of the energetic hydrolysate? The primary goal of the WHEAT system is to provide a total alternative solution to incineration for destroying and detoxifying the combined waste from assembled chemical weapons systems. The hydrolysis step of the WHEAT process eliminated the primary components of the energetic materials, TNT and RDX. It was expected that the energetic wouldn't undergo any further measurable degradation and would simply pass through the system. For the most part that appears to be the case. Most of the energetics and breakdowns were below detection limits in the feed and all effluent samples. The only detectable products were 3,4 Dinitrotoluese and PCP that were very near detection limits and yet appeared to simply pass through the system.

6.1 ACWA PROGRAM GOALS

The ACWA program designated eight specific goals and objectives for the biotreatment and associated systems. From the ACWA Program Study Plan, these objectives are listed below:

1. Validate the ability of the unit operation to eliminate schedule-2 compounds present in the HD/Tetrytol hydrolysate feed.
2. Confirm the absence of HD agent in the unit operations effluents.
3. Validate the ability of the agent hydrolysis process and the ICB, Flocculation Reactor, and Clarifier unit operations to achieve a destruction removal efficiency (DRE) of 99.9999% for HD.
4. Validate the ability of the Energetic Hydrolysis process and the ICB, Flocculation Reactor, and Clarifier unit operations to achieve a DRE of 99.999% for Tetrytol.
5. Develop mass loading and kinetic data that can be used for scale-up of the ICB Flocculation Reactor and Clarifier unit operations.
6. Validate the ability of the Catalytic Treater to eliminate chemical agents and schedule-2 compounds from the ICB process gas stream.
7. Determine the potential impact of operating conditions on the fouling and plugging of the catalytic reactor.
8. Characterize gas, liquid and solid process streams from the ICB, Flocculation Reactor, Clarifier, and Catalytic treater unit operations for selected chemical constituents and physical parameters, and the presence/absence of hazardous, toxic agent and schedule-2 compounds.

6.2 EVALUATION OF ACWA GOALS

We will address each of the ACWA proposed goals in order.

Samples were taken during each of the six weeks of the validation from all sample ports. Results for schedule-2 and breakdown products detected only three analytes: thiodiglycol, thioxane and dithane. Removal of schedule-2 and breakdown products was greater than 99% from the reactor effluent and 100% after the Fenton's reactor. A summary of the schedule-2 results is presented in Table 5 and 18. While concentrations of each analyte varied weekly, there was no apparent trend.

Before startup of the ICB, all drums of HD hydrolysate used in this study were cleared for non-surety operations to the drinking water standard of less than 200 µg/L. Since no mustard agent was introduced into the ICB system, no detection of any agent in ICB effluents was ever anticipated. To ensure there were no HD reformation during the process all samples were analyzed for HD during the validation. All analyses for HD came back below detection limits (200 µg/L). A summary of analysis of the HD hydrolysate used in the study is presented in section 4.1. Analysis of ICB system effluents for schedule-2 and HD hydrolysate breakdown products in are summarized in Table 18 below. Data show the ACWA goals for elimination of HD and schedule-2 products was met by hydrolysis of HD followed by biotreatment with the ICB system.

Table 18. Summary of the ICB Effluent Data of Schedule-2 and Breakdown Products

Sample ID	Unit Operation	TDG Avg. (ppm)	Thioxane Avg. (ppm)	Dithane Avg. (ppm)	Total Avg. (ppm)	Mass lbs./day	Avg. Cumulative % Removal
S1	Feed	3499	40.1	12.95	3552.05	5.92	----
S3	ICB	179.85	4.82	4.98	189.65	0.32	94.59%
S5	ICB effluent	<1	3.34	2.22	5.9	0.01	99.83%
S6	Fentons Reactor	<1	<1	<1	<1	0.00	100.0%
S7	Clarifier Sludge	<1	<1	<1	<1	0.00	
S8	Clarifier Effluent	<1	<1	<1	<1	0.00	
S10	Brine	<1	<1	<1	<1	0.00	

Data in section 4 and Table 18 above show there was no HD in ICB system liquid effluents. Data summarized in section 4 confirms the absence of any HD or schedule-2 compounds in exhaust air from the ICB before and after the CATOX.

Samples taken throughout the validation period confirm the complete removal of HD and tetrytol by hydrolysis of each followed by biotreatment in the ICB system. The hydrolysis process destroyed HD and Tetrytol to below detectable levels. No HD or Tetrytol was reformed or detected in any of the ICB system effluents.

Mass loading data developed for system scale-up was presented in section 4. Concerns for the accuracy and utility of data presented in section 5 still remain. As of this writing, a follow-on Engineering Design Study (EDS) is planned for summer 2000. This EDS will further develop data necessary for future scale-up.

The validation data indicate that no HD or schedule-2 compounds escaped the CATOX system. While ACWA goals were to validate the CATOX ability to destroy HD and schedule-2 compounds, that ability was not demonstrated with the ICB system since no HD and schedule-2 two products were detected in the exhaust gas stream. CATOX testing was run in an operation parallel to this effort to test the CATOX capability in destroying HD and schedule-2 compounds. There was no evidence of fouling or plugging of the ICB CATOX. Data presented in section 4 indicate no decreases in CATOX flow, temperature or increase in back pressure that would indicate any problems with the ICB CATOX system.

Extensive sampling and analysis of the ICB end process and intermediate effluents was undertaken to fully characterize all potential toxins generated by the ICB and all related system effluents. These data are presented in section 4. Many of the analytes found are typical of the biodegradation process and characteristic contaminants left over from the feed stream. Many of the analytes detected have no trend and are not indicative of the ICB system health but represent inorganic constituents of the feed. Numerous analytes were detected in the exhaust gases at concentrations very near the analytical detection limits. The system exhaust gases are not considered very hazardous. In planning discussions for the follow-on EDS, proposals were made to remove the HEPA/charcoal filter from the downstream side of the CATOX. This would have been acceptable if not for the fact that the powered charcoal filter also supplied ventilation for the trailer interior and added protection for the ICB operators.

The sludge and salt brine wastes generated by the ICB were analyzed by APG waste management and through ACWA validation sampling using TCLP criteria. Toxic chemicals that were detected were below regulatory limits. In the state of Maryland, these effluents would not be characterized as toxic waste if not for the Maryland MD-02 code for wastes originating from a chemical agent. These findings are presented in Tables 2 and 7. In connection with construction of a full scale HD agent destruction facility at Aberdeen Proving Ground, MD, the U.S. Army has petitioned the state of Maryland for a de-listing of MD-02 wastes generated in the destruction facility. The EDS study will further define and develop the toxicity of the effluent waste. Since the current plan of the EDS is to use a polymer to facilitate flocculation instead of a Fenton's reagent, waste generated may have an even lower toxicity.

One other major testing goal was the recycling of 65 percent of the ICB effluent in a water conservation effort. If constructed, the full scale ICB system could be operated under fairly stringent water conservation protocols. Nearly half the equipment, on an operations basis, was dedicated to sludge de-watering and water reuse. These systems included a Fentons reactor, inclined plate clarifier, triple filtration/reverse osmosis system and a water recycle/storage tank. As described in section 5, the water recycle system ran into considerable difficulties and had influence over how the ICB pH was eventually controlled.

The EDS planned for summer 2000 will include different technologies for accomplishing water re-use. The Fenton's reagent will be replaced with a polymer to induce flocculation. The system will also demonstrate the ability of a water crystallizer followed by a filter press to remove as much water for recycling as possible. This process, if successful, will remove a much greater portion of the salt and perhaps other inorganics that may have interfered on a very low level with the performance of the ICB.

The ICB system demonstrated its ability to detoxify HD/tetrytol hydrolysate and their breakdown products from the feed stream. Problems that were discussed in section 5 required additional consideration and continued system development. Continued testing with the pilot-scale ICB system is highly recommended. As of March 2000 a follow-on Engineering Design Test of up to four months in duration is planned for summer 2000. The proposed EDS is aimed at resolving the issues raised during demonstration testing and will continue to gather the necessary information required for development of a successful full scale system.

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APPENDIX A

TCLP Results and Regulatory Levels by Week and Sample Location

HD-ICB TCLP Validation Sampling, March 3

Analyte	Concentration (mg/L)		
	S7	S10	Regulatory Level
2-Butanone	0.046	0.54	**200**
2-Fluorobiphenyl	63	71	
2-Fluorophenol	42	32	
2-Methylphenol	0.05	0.05	**200**
Benzene	0.025	0.025	**0.5**
Bromofluorobenzene	93	93	
Carbon Tetrachloride	0.025	0.025	**0.5**
Chlorobenzene	0.025	0.025	**100**
Chloroform	0.025	0.005	**6**
Hexachlorobenzene	0.05	0.05	**0.13**
Hexachlorobutadiene	0.05	0.05	**0.5**
Hexachloroethane	0.05	0.05	**3**
Nitrobenzene	0.05	0.05	**2**
Nitrobenzene-d5	92	89	
Pentachlorophenol	0.12	0.12	**100**
pH	2.8	3.1	
Phenol-d5	70	60	
p-Terphenyl-d14	71	67	
Pyridine	0.05	0.05	**5**
Tetrachloroethene	0.025	0.025	**0.7**
Toluene-d8	98	92	
Total Methylphenol	0.05	0.05	**200**
Trichloroethene	0.025	0.025	**0.5**
Vinyl Chloride	0.05	0.05	**0.2**

Recovery of internal standard
Analyte above quantitation limit

HD-ICB, TCLP, March 24, 1999

Analyte	Units	S7	S8	Regulatory Level
1,1-Dichloroethene	MG/L	0.12	0.025	**0.7**
1,2-Dichloroethane	MG/L	0.12	0.025	**0.5**
1,2-Dichloroethane-d4	% REC	98	100	
1,4-Dichlorobenzene	mg/L	0.05	0.05	**7.5**
2,4,5-Trichlorophenol	mg/L	0.12	0.12	**400**
2,4,6-Tribromophenol	% REC	130	130	
2,4,6-Trichlorophenol	mg/L	0.05	0.05	**2**
2,4-Dinitrotoluene	mg/L	0.05	0.05	**0.13**
2-Butanone	MG/L	2.1	0.5	**200**
2-Fluorobiphenyl	% REC	76	75	
2-Fluorophenol	% REC	79	78	
2-Methylphenol	mg/L	0.05	0.05	**200**
3- and/or 4-Methylphenol	mg/L	0.05	0.05	**200**
ACETALDEHYDE	UG/L	600	80	
ACETONE	UG/L	200	30	
Benzene	MG/L	0.12	0.025	**0.5**
BENZENE, 1,2-DIMETHYL-	UG/L	70	0.025	
Bromofluorobenzene	% REC	99	99	
Carbon Tetrachloride	MG/L	0.12	0.025	**0.5**
Chlorobenzene	MG/L	0.12	0.025	**100**
Chloroform	MG/L	0.12	0.025	**6**
Chloromethane	UG/L	60	0.025	
ETHANOL	UG/L	60	0.025	
Hexachlorobenzene	mg/L	0.05	0.05	**0.13**
Hexachlorobutadiene	mg/L	0.05	0.05	**0.5**
Hexachloroethane	mg/L	0.05	0.05	**3**
Nitrobenzene	mg/L	0.05	0.05	**2**
Nitrobenzene-d5	% REC	79	79	
Pentachlorophenol	mg/L	0.12	0.12	**100**
Phenol-d5	% REC	72	71	
p-Terphenyl-d14	% REC	77	80	
P-XYLENE	UG/L	40	0.05	
Pyridine	mg/L	0.05	0.05	**5**
Tetrachloroethene	MG/L	0.12	0.025	**0.7**
Toluene-d8	% REC	99	98	
Trichloroethene	MG/L	0.12	0.025	**0.5**
Vinyl Chloride	MG/L	0.25	0.05	**0.2**
Mercury, TCLP Leachate	UG/L	0.16	0.1	**200**
Arsenic, TCLP Leachate	UG/L	33	33	**5,000**
Barium, TCLP Leachate	UG/L	122	348	**100,000**
Cadmium, TCLP Leachate	UG/L	5	4	**1,000**
Chromium, TCLP Leachate	UG/L	46.3	26.6	**5,000**
Lead, TCLP Leachate	UG/L	35.3	18	**5,000**
Selenium, TCLP Leachate	UG/L	36	50.6	**1,000**
Silver, TCLP Leachate	UG/L	22.1	28.3	**5,000**

Recovery of internal standard
Analyte above quantitation limit

HD-ICB, TCLP Analysis of Sludge and Brine, March 30, 1999

Analyte	Units	S7 (Bioreactor Sludge)	S10 (Brine)	Regulatory Level
1,1-Dichloroethene	mg/L	0.05	0.025	0.7
1,2-Dichloroethane-d4	% REC	97	98	
1,4-Dichlorobenzene	mg/L	0.05	0.05	7.5
2,4,5-Trichlorophenol	mg/L	0.12	0.12	400
2,4,6-Tribromophenol	% REC	64	86	
2,4,6-Trichlorophenol	mg/L	0.05	0.05	2
2,4-Dinitrotoluene	mg/L	0.05	0.05	0.13
2-Butanone	MG/L	1.1	0.62	200
2-Fluorobiphenyl	% REC	61	73	
2-Fluorophenol	% REC	58	65	
2-Methylphenol	mg/L	0.05	0.05	200
3- and/or 4-Methylphenol	mg/L	0.05	0.05	200
ACETALDEHYDE	UG/L	50	50	
Arsenic, TCLP Leachate	UG/L	204	68.3	5,000
Barium, TCLP Leachate	UG/L	121	126	100,000
Benzene	MG/L	0.05	0.025	0.5
Bromofluorobenzene	% REC	102	106	
Cadmium, TCLP Leachate	UG/L	4	4	1,000
Carbon Tetrachloride	MG/L	0.05	0.025	0.5
Chlorobenzene	MG/L	0.05	0.025	100
Chloroform	MG/L	0.05	0.025	6
Chromium, TCLP Leachate	UG/L	52.4	339	5,000
Hexachlorobenzene	mg/L	0.05	0.05	0.13
Hexachlorobutadiene	mg/L	0.05	0.05	0.5
Hexachloroethane	mg/L	0.05	0.05	3
Lead, TCLP Leachate	UG/L	20.9	18	5,000
Mercury, TCLP Leachate	UG/L	0.1	0.1	200
Nitrobenzene	mg/L	0.05	0.05	2
Nitrobenzene-d5	% REC	66	82	
Pentachlorophenol	mg/L	0.12	0.12	100
Phenol-d5	% REC	61	70	
p-Terphenyl-d14	% REC	73	85	
Pyridine	mg/L	0.05	0.05	5
Selenium, TCLP Leachate	UG/L	43.9	36	1,000
Silver, TCLP Leachate	UG/L	9	9	5,000
Tetrachloroethene	MG/L	0.05	0.025	0.5
Toluene-d8	% REC	100	103	
Trichloroethene	MG/L	0.05	0.025	0.5
Vinyl Chloride	MG/L	0.1	0.05	0.2

Analyte detected above quantitation limit
Recovery of internal standard

HD-ICB, TCLP Data, April 7, 1999

Analytes	Units	S10	Regulatory limits
1,1-Dichloroethene	MG/L	0.025	**0.7**
1,2-Dichloroethane	MG/L	0.025	**0.5**
1,2-Dichloroethane-d4	% REC	110	
1,4-Dichlorobenzene	Mg/L	0.05	**7.5**
2,4,5-Trichlorophenol	Mg/L	0.12	**400**
2,4,6-Tribromophenol	% REC	92	
2,4,6-Trichlorophenol	Mg/L	0.05	**2**
2,4-Dinitrotoluene	Mg/L	0.05	**0.13**
2-Butanone	MG/L	0.19	**200**
2-Fluorobiphenyl	% REC	68	
2-Fluorophenol	% REC	67	
2-Methylphenol	Mg/L	0.05	**200**
3- and/or 4-Methylphenol	Mg/L	0.05	**200**
Arsenic	UG/L	33	**5,000**
Barium	UG/L	67.5	**100,000**
Benzene	MG/L	0.025	**0.5**
Bromofluorobenzene	% REC	103	
Cadmium	UG/L	4	**1,000**
Carbon Tetrachloride	MG/L	0.025	**0.5**
Chlorobenzene	MG/L	0.025	**100**
Chloroform	MG/L	0.025	**6**
Chromium	UG/L	1,270	**5,000**
Cyanide, Reactive	MG/KG	0.5	
Hexachlorobenzene	Mg/L	0.05	**0.13**
Hexachlorobutadiene	Mg/L	0.05	**0.5**
Hexachloroethane	Mg/L	0.05	**3**
Lead	UG/L	18	**5,000**
Mercury	UG/L	0.11	**200**
Nitrobenzene	Mg/L	0.05	**2**
Nitrobenzene-d5	% REC	70	
Pentachlorophenol	Mg/L	0.12	**100**
pH	Standard u	3.4	
Phenol-d5	% REC	72	
p-Terphenyl-d14	% REC	101	
Pyridine	Mg/L	0.05	**5**
Selenium	UG/L	36	**1,000**
Silver	UG/L	5.8	**5,000**
Sulfide, Reactive	MG/KG	24	
Tetrachloroethene	MG/L	0.025	**0.7**
Toluene-d8	% REC	103	
Trichloroethene	MG/L	0.025	**0.5**
Vinyl Chloride	MG/L	0.05	**0.2**

Analyte detected above the quantitation limit
Recovery of internal standard

HD-ICB, TCLP analysis of Sludge and Brine, April 21, 1999

Analyte	Units	S7	S10	Regulatory Limit
1,1-Dichloroethene	mg/L	0.025	0.025	**0.7**
1,2-Dichloroethane	mg/L	0.025	0.025	**0.5**
1,2-Dichloroethane-d4	% REC	107	105	
1,4-Dichlorobenzene	mg/L	0.05	0.05	**7.5**
2,4,5-Trichlorophenol	mg/L	0.12	0.12	**400**
2,4,6-Tribromophenol	% REC	101	76	
2,4,6-Trichlorophenol	mg/L	0.05	0.05	**2**
2,4-Dinitrotoluene	mg/L	0.05	0.05	**0.13**
2-Butanone	mg/L	0.011	0.05	**200**
2-Fluorobiphenyl	% REC	78	67	
2-Fluorophenol	% REC	72	61	
2-Methylphenol	mg/L	0.05	0.05	**200**
3- and/or 4-Methylphenol	mg/L	0.05	0.05	**200**
Arsenic	UG/L	42.2	42.3	**5,000**
Barium	UG/L	122	63	**100,000**
Benzene	mg/L	0.025	0.025	**0.5**
Bromofluorobenzene	% REC	99	101	
Cadmium	UG/L	5	5	**1,000**
Carbon Tetrachloride	mg/L	0.025	0.025	**0.5**
Chlorobenzene	MG/L	0.025	0.025	**100**
Chloroform	MG/L	0.025	0.025	**6**
Chromium	UG/L	6.4	4.2	**5,000**
Hexachlorobenzene	mg/L	0.05	0.05	**0.13**
Hexachlorobutadiene	mg/L	0.05	0.05	**0.5**
Hexachloroethane	mg/L	0.05	0.05	**3**
Lead	UG/L	32.6	32.6	**5,000**
Mercury	UG/L	0.1	0.1	**200**
Nitrobenzene	mg/L	0.05	0.05	**2**
Nitrobenzene-d5	% REC	75	64	
Pentachlorophenol	mg/L	0.12	0.12	**100**
Phenol-d5	% REC	77	65	
p-Terphenyl-d14	% REC	90	73	
Pyridine	mg/L	0.05	0.05	**5**
Selenium	UG/L	59.3	59.3	**1,000**
Silver	UG/L	4.5	3.1	**5,000**
Tetrachloroethene	MG/L	0.025	0.025	**0.7**
Toluene-d8	% REC	98	99	
Trichloroethene	MG/L	0.025	0.025	**0.5**
Vinyl Chloride	MG/L	0.05	0.05	**0.2**

Analyte detected above quantitation limit
Recovery of internal standard

HD-ICB, TCLP Analysis of Sludge and Brine, April 28, 1999

Analyte	Units	S7	S10	Regulatory Limit
1,1-Dichloroethene	MG/L	0.025	0.025	**0.7**
1,2-Dichloroethane	MG/L	0.025	0.025	**0.5**
1,2-Dichloroethane-d4	% REC	107	108	
1,4-Dichlorobenzene	mg/L	0.05	0.05	**7.5**
2,4,5-Trichlorophenol	mg/L	0.12	0.12	**400**
2,4,6-Tribromophenol	% REC	43	69	
2,4,6-Trichlorophenol	mg/L	0.05	0.05	**2**
2,4-Dinitrotoluene	mg/L	0.05	0.05	**0.13**
2-Butanone	MG/L	0.037	0.023	**200**
2-Fluorobiphenyl	% REC	41	78	
2-Fluorophenol	% REC	21	75	
2-Methylphenol	mg/L	0.05	0.05	**200**
3- and/or 4-Methylphenol	mg/L	0.05	0.05	**200**
Arsenic	UG/L	58.7	62	**5,000**
Barium	UG/L	391	50.9	**100,000**
Benzene	MG/L	0.025	0.025	**0.5**
Bromofluorobenzene	% REC	95	94	
Cadmium	UG/L	5	5	**1,000**
Carbon Tetrachloride	MG/L	0.025	0.025	**0.5**
Chlorobenzene	MG/L	0.025	0.025	**100**
Chloroform	MG/L	0.025	0.025	**6**
Chromium	UG/L	16.7	18.4	**5,000**
Hexachlorobenzene	mg/L	0.05	0.05	**0.13**
Hexachlorobutadiene	mg/L	0.05	0.05	**0.5**
Hexachloroethane	mg/L	0.05	0.05	**3**
Lead	UG/L	48.4	32.6	**5,000**
Mercury	UG/L	0.1	0.1	**200**
Nitrobenzene	mg/L	0.05	0.05	**2**
Nitrobenzene-d5	% REC	42	78	
Pentachlorophenol	mg/L	0.12	0.12	**100**
Phenol-d5	% REC	13	78	
p-Terphenyl-d14	% REC	48	71	
Pyridine	mg/L	0.05	0.05	**5**
Selenium	UG/L	59.3	59.3	**1,000**
Silver	UG/L	3.1	3.1	**5,000**
Tetrachloroethene	MG/L	0.025	0.025	**0.7**
Toluene-d8	% REC	95	96	
Trichloroethene	MG/L	0.025	0.025	**0.5**
Vinyl Chloride	MG/L	0.05	0.05	**0.2**

Analyte detected above the quantitation limit
Recovery of internal standard

APPENDIX B

SVOC Data

HD-ICB, Analysis for Semi VOCs, March 3, 1999

Analyte	Concentration (ug/L)					
	S1	S3	S5	S6	S7	S8
1,2-Dichlorobenzene-d4 (S8)	73	110	42	44		56
2,4,6-TRibromophenol 118796 (S	99	201	85	74		16
2-Chlorophenol-d4 (S7)	89	124	82	80		86
2-Fluorobiphenyl (S2)	81	106	108	115		117
2-Fluorophenol (S5)	88	155	77	71		85
ETHANOL, 2,2'-[1,2-ETHANEDIY	2600	400	400	400	400	400
ETHANOL, 2,2'-DITHIOBIS-	3800	400	400	400	400	400
ETHANOL, 2,2'-THIOBIS-	29000	400	400	400	400	400
Nitrobenzene-d5 (S1)	81	182	92	72		57
Phenol-d5 (S4)	93	124	84	83		84
Terphenyl-d14 (S3)	85	154	66	76		83

Analyte detected above the quantitation limit
Recovery of internal standard

HD-ICB, Analysis of Semi-VOCs, March 24, 1999

Analyte	Concentration (ug/L)				
	S1	S3	S5	S7	Field Blank
1,2-Dichlorobenzene-d4 (S8)	0	50	63		49
1,4-DITHIANE	1300	100	200	50	10
2,4,6-TRibromophenol 118796 (S	0	130	120		80
2-Chlorophenol-d4 (S7)	0	99	92		73
2-Fluorobiphenyl (S2)	0	83	96		69
2-Fluorophenol (S5)	0	28	84		69
BENZENESULFONAMIDE, N-BUTYL-	400	100	100	20	9
bis(2-Ethylhexyl)phthalate	2300	100	100	20	10
ETHANOL, 2,2'-[1,2-ETHANEDIY	7100	450	100	20	10
ETHANOL, 2,2'-DITHIOBIS-	4600	280	100	20	10
Nitrobenzene-d5 (S1)	0	98	89		60
Phenol-d5 (S4)	0	85	92		71
Terphenyl-d14 (S3)	0	86	48		80

Analyte detected above quantitation limit
Analyte detected in Field Blank
Recovery of internal standard

HD-ICB, Analysis for Semi-volatiles, March 31, 1999

Analyte	Concentration (ug/L)					
	S1	S3	S5	S6	S7	S8
1,2,5-TRITHIEPANE	5000	500	100	100	19	10
1,2-Dichlorobenzene-d4 (S8)	56	0	53	27		40
1,3,5-TRITHIANE	5000	500	100	100	72	
1,4-DITHIANE	5000	700	200	45	20	15
1,4-OXATHIANE, 4,4-DIOXIDE	5000	500	71	100	62	10
1-PROPENE, 1,2,3-TRICHLORO-	5000	500	100	100	21	10
2,4,6-TRibromophenol 118796 (S	65	0	82	57		65
2-Chlorophenol-d4 (S7)	63	0	79	55		65
2-Fluorobiphenyl (S2)	69	0	72	50		56
2-Fluorophenol (S5)	60	0	72	48		65
9-HEXADECENOIC ACID	5000	500	100	100	33	10
BENZALDEHYDE, 4-METHYL-	5000	500	100	100	23	10
BENZENE, 1,3-DIMETHYL-	5000	500	100	100	46	10
bis(2-Ethylhexyl)phthalate	1500	500	100	100	8	10
BUTYROLACTONE	5000	500	100	100	6.6	10
DICHLOROCYCLOHEXANE ISOMER	40000	500	100	100	20	10
ETHANOL, 2,2'-[1,2-ETHANEDIY	3000	500	100	49	20	8
ETHANOL, 2,2'-DITHIOBIS-	2700	500	100	100	9.2	10
HEXADECANOIC ACID	5000	500	66	100	40	10
MALTOL	5000	500	100	100	20	4
METHYL BENZALDEHYDE ISOMER	5000	500	100	100	20	6
Nitrobenzene-d5 (S1)	60	0	93	60		64
OCTADECANOIC ACID	5000	500	100	100	8.9	10
OLEIC ACID	5000	500	100	100	62	10
Phenol-d5 (S4)	54	0	76	53		61
Terphenyl-d14 (S3)	101	0	87	60		88

Analyte detected above the quantitation limit
Analyte detected in the blank
Recovery of Internal standard

HD-ICB, Analysis for SVOCs, April 14, 1999

	Concentration (ug/L)					
Analyte	**S1**	**S3**	**S5**	**S6**	**S7**	**S8**
1,2,5-TRITHIEPANE				10	24	100
1,2-Dichlorobenzene-d4 (S8)	44	40	54	7		48
1,3,5-TRITHIANE				10	21	100
1,4-DITHIANE		1700	84	6	20	41
1,4-OXATHIANE, 4,4-DIOXIDE		100	22	10	20	36
2,4,6-TRibromophenol 118796 (S	32	78	62	8		64
2-Chlorophenol-d4 (S7)	49	80	64	10		70
2-Fluorobiphenyl (S2)	68	64	80	11		76
2-Fluorophenol (S5)	53	77	68	10		83
BENZENE, 1,2-DIMETHYL-	5000	100	100	10	30	100
DICHLOROCYCLOHEXANE ISOMER	22000	100	100	10	20	100
ETHANOL, 2,2'-DITHIOBIS-	5000	150	100	10	130	100
ETHYL ACETATE	5000	100	100	10	63	100
ETHYLBENZENE	5000	100	100	10	21	100
HEXADECANOIC ACID	5000	50	100	7	45	100
MALTOL	5000	20	100	100	20	100
OLEIC ACID	5000	100	100	100	63	100
Nitrobenzene-d5 (S1)	58	73	85	14		85
Phenol-d5 (S4)	45	77	60	9		71
P-XYLENE	5000	100	100	10	69	100
Terphenyl-d14 (S3)	97	72	30	10		

Analyte detected above the quantitation limit

Analyte detected in the blank

Recovery of internal standard

HD-ICB, Analysis for SVOCs, April 21, 1999

Analyte	S1	S7
1,2,5-TRITHIEPANE	5000	20
1,2-Dichlorobenzene-d4 (S8)	37	
2,4,6-TRibromophenol 118796 (S	57	
2-Chlorophenol-d4 (S7)	52	
2-Fluorobiphenyl (S2)	46	
2-Fluorophenol (S5)	48	
4-METHYLPHENOL		69
BENZENE, 1,2-DIMETHYL-		80
BENZENEACETIC ACID		30
BIS(2-ETHYLHEXYL)PHTHALATE		13
BUTANOIC ACID, 2-METHYL-		20
BUTANOIC ACID, 3-METHYL-		66
DICHLOROCYCLOHEXANE ISOMER	1800	20
DI-N-BUTYLPHTHALATE		11
ETHANOL, 2,2'-DITHIOBIS-		170
HEXADECANOIC ACID		9.4
HYDRAZINE, 1,2-DIMETHYL-		59
Nitrobenzene-d5 (S1)	43	
OLEIC ACID		16
Phenol-d5 (S4)	41	
	48	
THIODIGLYCOL		46

Analyte detected above the quantitation limit

Analyte detected in the blank

Recovery of internal standard

HD-ICB, Analysis for SVOCs, April 28, 1999

	Concentration (ug/L)				
Analyte	**S1**	**S3**	**S5**	**S6**	**S8**
1,2,5-TRITHIEPANE	4000	400	200	100	50
1,3-DITHIOLANE	4000	400	200	100	80
1,4-DITHIANE	3000	2000	2000	100	40
1,4-OXATHIANE	4000	3000	2000	100	200
2,2'-DITHIOBISETHANOL	10000	400	200	100	100
2,4,6-Tribromophenol	69	122	91	99	106
2-Fluorobiphenyl	71	89	66	75	76
2-Fluorophenol	52	96	74	83	81
4,4-DIOXIDE,1,4-OXATHIANE	10000	1000	500	20	90
4-Methylphenol	4000	400	200	100	30
4-OXIDE,1,4-OXATHIANE	4000	400	200	100	100
BENZENEACETIC ACID	4000	400	200	100	100
bis(2-Ethylhexyl)phthalate	4000	70	67	130	33
HEXANOIC ACID	4000	400	200	60	100
MALTOL	4000	400	200	20	100
Nitrobenzene-d5	72	93	67	63	75
Phenol-d5	58	94	68	74	79
Terphenyl-d14	91	98	73	47	50
THIETANE	4000	8000	100	100	100
THIODIGLYCOL	500000	8000	1000	100	100

Analyte detected above the quantitation limit

Analyte detected in the blank

Recovery of internal standard

Blank

APPENDIX C

VOC Data

HD-ICB, Analysis of VOCs in liquid, March 3,1999

Analyte	Concentration (ug/L)				
	S1	S3	S5	S8	Field Blank
1,2-Dichloroethane	1100	250	10	100	10
1,2-Dichloroethane-d4	94	86	88	94	94
2-Butanone	220	4800	29	740	10
Bromofluorobenzene	96	97	99	96	96
Chloromethane	3800	250	10	100	10
Toluene-d8	93	96	95	102	104

Analyte detected above quantitation limit
Recovery of internal standard

HD-ICB Analysis for VOCs, March 24, 1999

Analyte	Concentration (ug/L)						
	S1	S3	S5	S6	S7	S8	Field Blank
1,2-Dichloroethane	800	7	10	50	250	50	10
1,2-Dichloroethane-d4 (SMC3)	91	102	96	98		99	96
1,4-DITHIANE	98	9	13	50	250	50	10
1,4-OXATHIANE	56	22	14	50	250	50	10
2-Butanone	100	15	10	500	3400	500	10
Acetone	100	12	10	240	1600	250	10
Bromofluorobenzene (SMC2)	99	101	100	99		99	99
Chloroform	28	10	10	50	250	50	10
Chloromethane	2000	10	9	50	320	50	10
Toluene-d8 (SMC1)	100	100	102	100		100	100
Xylene (total)	100	10	10	50	230	35	10

Recovery of Internal Standard
Analyte detected above quantitation limit

HD-ICB, Analysis for VOCs,

Analyte	Concentration Ug/L					
	S1	S3	S5	S6	S7	S8
1,2-Dichloroethane	820	3	10	8	11	6
1,2-Dichloroethane-d4 (SMC3)	91	90	89	90		87
1,4-DITHIANE	90	46	6	20	50	20
1,4-OXATHIANE	100	11	10	20	50	20
2,3-Butanedione	100	10	10	20	44	20
2-Butanone	130	12	10	190	380	160
Acetaldehyde	100	10	10	20	36	20
Acetone	230	79	18	140	270	170
Bromofluorobenzene (SMC2)	97	100	99	97		100
Bromomethane	100	10	10	20	24	20
Carbonyl sulfide	100	10	10	20	7.2	20
Chloromethane	1100	10	10	25	27	26
Ethylbenzene	100	10	10	7	30	12
Toluene-d8 (SMC1)	102	101	101	99		100
Xylene (total)	100	10	10	48	200	73

Analyte detected above the quantitation limit
Analyte detected in the blank
Recovery of internal standard

HD-ICB, Analysis for VOCs,

Analyte	Concentration (ug/L)						
	S1	S3	S5	S6	S7	S8	Field Blank
1,2-Dichloroethane	890	8	10	6	9	20	10
1,2-Dichloroethane-d4 (SMC3)	92	90	90	95		91	112
1,4-DITHIANE	78	10	10	10	25	20	10
1,4-OXATHIANE	65	21	10	10	25	20	10
2,3-BUTANEDIONE	100	10	10	10	110	20	10
2-Butanone	100	10	10	66	510	240	10
ACETALDEHYDE	100	10	10	10	53	15	10
Acetone	100	16	18	160	280	170	10
ACETONITRILE	100	10	10	10	14	20	10
Bromofluorobenzene (SMC2)	104	104	104	93		106	95
Chloromethane	770	10	16	25	25	16	10
DITH	100	28	10	10	25	20	10
Ethylbenzene	100	10	10	10	14	10	10
Toluene-d8 (SMC1)	102	102	103	101		101	104
Xylene (total)	100	10	10	10	81	90	10

Detected above the quantitation limit
Recovery of internal standard

HD-ICB, Analysis for VOCs, April 28, 1999

	Concentration (ug/L)				
Analyte	S1	S3	S5	S6	S8
1,2-Dichloroethane	1100	17	5	25	25
1,2-Dichloroethane-d4	106	100	104	105	105
1,4-DITHIANE	80	90	90	25	25
1,4-OXATHIANE	50	40	40	25	25
2-Butanone	120	61	9	220	680
ACETIC ACID, METHYL ESTER	100	10	7	50	50
Acetone	240	30	83	630	520
Benzene	50	4	5	25	25
Bromofluorobenzene	98	92	97	94	98
Carbon Disulfide	50	5	5	8	36
Chloromethane	1500	5	4	61	27
Methylene Chloride	57	3	2	29	30
SILANE	50	5	9	25	25
Toluene-d8	96	96	98	98	98
Vinyl Chloride	100	1	10	50	50
Xylene (total)	50	5	5	25	30

Analyte detected above the quantitation limit

Analyte detected in the blank

Recovery of internal standard

HD-ICB, Analysis of VOCs in liquid, March 3,1999

	Concentration (ug/L)				
Analyte	S1	S3	S5	S8	Field Blank
1,2-Dichloroethane	1100	250	10	100	10
1,2-Dichloroethane-d4	94	86	88	94	94
2-Butanone	220	4800	29	740	10
Bromofluorobenzene	96	97	99	96	96
Chloromethane	3800	250	10	100	10
Toluene-d8	93	96	95	102	104

Analyte detected above quantitation limit

Recovery of internal standard

HD-ICB Analysis for VOCs, March 24, 1999

Analyte	Concentration (ug/L)						
	S1	S3	S5	S6	S7	S8	Field Blank
1,2-Dichloroethane	800	7	10	50	250	50	10
1,2-Dichloroethane-d4 (SMC3)	91	102	96	98		99	96
1,4-DITHIANE	98	9	13	50	250	50	10
1,4-OXATHIANE	56	22	14	50	250	50	10
2-Butanone	100	15	10	500	3400	500	10
Acetone	100	12	10	240	1600	250	10
Bromofluorobenzene (SMC2)	99	101	100	99		99	99
Chloroform	28	10	10	50	250	50	10
Chloromethane	2000	10	9	50	320	50	10
Toluene-d8 (SMC1)	100	100	102	100		100	100
Xylene (total)	100	10	10	50	230	35	10

Recovery of internal standard

Analyte detected above quantitation limit

HD-ICB, Analysis for VOCs,

Analyte	Concentration Ug/L					
	S1	S3	S5	S6	S7	S8
1,2-Dichloroethane	820	3	10	8	11	6
1,2-Dichloroethane-d4 (SMC3)	91	90	89	90		87
1,4-DITHIANE	90	46	6	20	50	20
1,4-OXATHIANE	100	11	10	20	50	20
2,3-Butanedione	100	10	10	20	44	20
2-Butanone	130	12	10	190	380	160
Acetaldehyde	100	10	10	20	36	20
Acetone	230	79	18	140	270	170
Bromofluorobenzene (SMC2)	97	100	99	97		100
Bromomethane	100	10	10	20	24	20
Carbonyl sulfide	100	10	10	20	7.2	20
Chloromethane	1100	10	10	25	27	26
Ethylbenzene	100	10	10	7	30	12
Toluene-d8 (SMC1)	102	101	101	99		100
Xylene (total)	100	10	10	48	200	73

Analyte detected above the quantitation limit

Analyte detected in the blank

Recovery of internal standard

HD-ICB, Analysis for VOCs,

Analyte	Concentration (ug/L)						
	S1	S3	S5	S6	S7	S8	Field Blank
1,2-Dichloroethane	890	8	10	6	9	20	10
1,2-Dichloroethane-d4 (SMC3)	92	90	90	95		91	112
1,4-DITHIANE	78	10	10	10	25	20	10
1,4-OXATHIANE	65	21	10	10	25	20	10
2,3-BUTANEDIONE	100	10	10	10	110	20	10
2-Butanone	100	10	10	66	510	240	10
ACETALDEHYDE	100	10	10	10	53	15	10
Acetone	100	16	18	160	280	170	10
ACETONITRILE	100	10	10	10	14	20	10
Bromofluorobenzene (SMC2)	104	104	104	93		106	95
Chloromethane	770	10	16	25	25	16	10
DITH	100	28	10	10	25	20	10
Ethylbenzene	100	10	10	10	14	10	10
Toluene-d8 (SMC1)	102	102	103	101		101	104
Xylene (total)	100	10	10	10	81	90	10

Detected above the quantitation limit

Recovery of internal standard

HD-ICB, Analysis for VOCs, April 28, 1999

	Concentration (ug/L)				
Analyte	S1	S3	S5	S6	S8
1,2-Dichloroethane	1100	17	5	25	25
1,2-Dichloroethane-d4	106	100	104	105	105
1,4-DITHIANE	80	90	90	25	25
1,4-OXATHIANE	50	40	40	25	25
2-Butanone	120	61	9	220	680
ACETIC ACID, METHYL ESTER	100	10	7	50	50
Acetone	240	30	83	630	520
Benzene	50	4	5	25	25
Bromofluorobenzene	98	92	97	94	98
Carbon Disulfide	50	5	5	8	36
Chloromethane	1500	5	4	61	27
Methylene Chloride	57	3	2	29	30
SILANE	50	5	9	25	25
Toluene-d8	96	96	98	98	98
Vinyl Chloride	100	1	10	50	50
Xylene (total)	50	5	5	25	30

Analyte detected above the quantitation limit

Analyte detected in the blank

Recovery of internal standard

Blank

APPENDIX D

Metals Data

HD- ICB Validation Analysis for Metals - March 3

	Metals Concentration (mg/L)				
Metal	**S1**	**S3**	**S5**	**S6**	**S8**
Al	0.247	0.333	0.031	0.224	0.185
Sb	0.033	0.061	0.03	0.043	0.03
As	0.042	0.025	0.025	0.039	0.025
Ba	0.041	0.036	0.027	0.021	0.005
Ca	15.8	14.723	15.913	19.4	19.45
Cr	0.206	0.013	0.01	0.075	0.1
Cu	0.018	0.021	0.01	0.01	0.01
Fe	30.9	9.612	7.586	166.713	109.4
Hg	0.0002	0.0002	0.0002	0.0002	0.0021
Mg	5.83	5.928	6.491	7.112	7.21
Mn	0.516	0.225	0.229	1.808	1.611
Ni	0.255	0.059	0.034	0.208	0.2
K	209	252.847	160.989	132.632	110.9
Se	0.04	0.04	0.04	0.04	0.04
Ag	0.005	0.005	0.005	0.005	0.01
Na	4910	5330	4670	4640	4210
Sn	0.02	0.02	0.02	0.063	0.02
V	0.01	0.01	0.01	0.012	0.01
Zn	0.701	0.37	0.608	0.924	1.034

Analyte above detection limit

HD-ICB, Analysis for Metals, March 24, 1999

	Concentration (ug/L)						
Analyte	S1	S3	S5	S6	S7	S8	Field Blank
Aluminum, Total	502	502	727	850	1810	502	100
Antimony, Total	47.5	78.7	100	68.2	145	102	9.5
Arsenic, Total	61.5	57.5	57.5	71.5	57.5	57.5	11.5
Barium, Total	65.7	8.8	7.8	10.3	21.3	7	2.3
Calcium, Total	13000	17100	15100	14900	26900	15400	154
Chromium, Total	220	15	15	34.8	44.3	19.8	3
Cobalt, Total	17.7	17.5	17.5	18.5	19.8	21	3.5
Copper, Total	90.3	36.5	28	45.3	62	31.8	6
Iron, Total	3980	1420	2910	117000	69400	57400	97
Magnesium, Total	5160	5790	6150	5800	8430	6140	38.9
Manganese, Total	39.8	138	118	1060	879	767	1
Mercury, Total	1	0.1	0.1	0.24	0.1	0.1	0.1
Nickel, Total	241	30	35.2	124	111	105	6
Potassium, Total	28300	116000	186000	175000	321000	205000	140
Sodium, Total	1860000	2150000	3550000	3250000	4970000	3640000	282
Thallium, Total	90.5	87.5	87.5	116	131	87.5	17.5
Tin, Total	67.5	67.5	67.5	67.5	70.3	67.5	13.5
Zinc, Total	319	186	443	658	372	615	10.3

Analyte detected above quantitation limit

HD-ICB, Analysis for Metals, March 31, 1999

	Concentration (ug/L)						
Analyte	S1	S3	S5	S6	S7	S8	Field Blank
Aluminum, Total	749	616	8090	1070	17700	793	100
Antimony, Total	56.5	47.5	109	83.8	277	47.5	9.5
Arsenic, Total	57.5	57.5	57.5	57.5	57.5	67	11.5
Barium, Total	37	16.3	58.5	15.5	94	16.8	5.1
Calcium, Total	15400	16600	16700	18900	19000	18400	134
Chromium, Total	144	16.5	30.5	59.5	139	54.5	3.8
Cobalt, Total	17.5	17.5	17.5	20.5	25.2	29.3	3.5
Copper, Total	67.7	31	50.2	26.3	228	25.7	10.6
Iron, Total	20200	5010	19200	85300	845000	76200	97
Magnesium, Total	5520	5190	5350	5960	6190	5800	65.7
Manganese, Total	367	192	242	1320	1370	1410	1
Mercury, Total	1	0.1	0.1	0.2	4.3	0.1	0.1
Nickel, Total	173	34.3	53	148	298	178	6
Phosphorus, Total	11300	11300	25500	4430	190000	1110	86
Potassium, Total	91600	141000	135000	138000	137000	140000	140
Sodium, Total	2340000	3990000	3610000	3490000	3320000	3590000	407
Thallium, Total	88.8	87.5	87.5	95	87.5	103	17.5
Tin, Total	67.5	67.5	67.5	67.5	680	67.5	13.5
Zinc, Total	475	122	482	686	736	397	10.9

Analyte detected above the quantitation limit

HD-ICB, Analysis for Metals, April 7, 1999

	Concentration (ug/L)					
Analyte	**S1**	**S3**	**S5**	**S6**	**S7**	**S8**
Aluminum, Total	960	910	972	2180	4170	1540
Antimony, Total	47.5	91.5	80.5	98.3	143	47.5
Barium, Total	28.7	33.2	17.5	31.8	43	23.2
Calcium, Total	13900	19900	17300	25200	21800	21900
Chromium, Total	54.3	24.5	15	73.8	70.7	53.5
Cobalt, Total	17.5	17.5	17.5	31	20.5	17.5
Copper, Total	48	36	29.3	59.5	79	30
Iron, Total	15200	11500	7260	227000	218000	69400
Magnesium, Total	5080	4970	4980	6890	6300	6300
Manganese, Total	430	396	250	2220	1540	1430
Mercury, Total	1	0.1	1.9	0.18	0.28	1.7
Molybdenum, Total	30	30	30	30	30	34.5
Nickel, Total	120	53.7	30	275	200	164
Phosphorus, Total	9550	9930	11900	18400	37100	1030
Potassium, Total	81700	147000	125000	141000	128000	140000
Sodium, Total	2990000	4180000	3560000	3940000	3510000	4050000
Thallium, Total	87.5	123	130	87.5	87.5	93.2
Tin, Total	67.5	67.5	67.5	72.7	169	67.5
Zinc, Total	546	206	243	1100	532	4300

Analyte detected above the quantitation limit

HD-ICB, Analysis for Metals, April 14, 1999

	Concentration (ug/L)					
Analyte	**S1**	**S3**	**S5**	**S6**	**S7**	**S8**
Aluminum, Total	776	1070	990	1570	5140	718
Antimony, Total	47.5	47.5	47.5	47.5	48.3	47.5
Barium, Total	35.8	21.3	14.7	23.8	58.5	10.7
Calcium, Total	15100	12400	12200	15400	17600	18000
Chromium, Total	182	38.2	21	48.5	150	31.3
Copper, Total	74.8	27.7	30.5	38.2	109	25
Iron, Total	15300	6320	5120	88300	521000	33200
Lead, Total	72.5	110	72.5	72.5	72.5	72.5
Magnesium, Total	7810	7760	4740	5370	5390	4800
Manganese, Total	411	167	148	846	990	992
Mercury, Total	1	0.1	0.1	0.1	0.18	0.1
Nickel, Total	163	85	63	117	236	119
Phosphorus, Total	12800	23400	14600	16300	101000	1240
Potassium, Total	82200	159000	147000	157000	144000	160000
Sodium, Total	3460000	4490000	4320000	4610000	4070000	4640000
Tin, Total	67.5	67.5	67.5	67.5	397	67.5
Zinc, Total	467	247	239	391	409	1400

Analyte detected above the quantitation limit

HD-ICB, Analysis for Metals, April 21, 1999

	Concentration (ug/L)					
Analyte	**S1**	**S3**	**S5**	**S6**	**S7**	**S8**
Aluminum, Total	502	1830	2470	2330	2290	743
Barium, Total	28.7	49.3	42.2	44	34.3	18.5
Calcium, Total	12700	18600	20200	24300	18800	18300
Chromium, Total	94.5	68.5	38.5	90	91.8	53.5
Copper, Total	90.3	74.3	63.5	58.2	53	25
Iron, Total	15000	18000	22800	190000	254000	77200
Magnesium, Total	6280	8930	9900	11100	10300	9180
Manganese, Total	381	577	693	2080	1650	1720
Nickel, Total	185	88	87.8	242	216	196
Phosphorus, Total	5380	28000	30800	31700	49700	2270
Potassium, Total	58200	135000	143000	155000	169000	137000
Sodium, Total	2300000	4130000	4390000	4580000	4620000	3980000
Tin, Total	67.5	67.5	67.5	124	220	67.5
Zinc, Total	553	518	724	1930	1390	1730

Analyte detected above the quantitation limit

HD-ICB, Analysis for Metals, April 28, 1999

	Concentration (mg/L)					
Analyte	**S1**	**S3**	**S5**	**S6**	**S7**	**S8**
Aluminum	20	20	20	20	21	20
Arsenic	0.4	0.4	0.4	0.4	0.5	0.4
Calcium	20	20	20	22	26	24
Chromium	0.1	0.1	0.1	0.2	0.8	0.1
Cobalt	0.02	0.02	0.02	0.02	0.05	0.02
Copper	0.09	0.12	0.09	0.11	0.51	0.06
Iron	34.4	29.9	26.8	174	881	80.2
Magnesium	7	6	8	8	14	9
Manganese	0.79	0.69	0.57	1.72	1.96	1.7
Nickel	0.3	0.3	0.3	0.3	0.7	0.3
Phosphorus	10.6	28.3	28.2	30.5	260	3.1
Potassium	87.7	150	142	139	146	138
Sodium	3,330	3,920	4,270	4,070	4,170	4,270
Vanadium	0.02	0.02	0.02	0.02	0.03	0.02
Zinc	1	0.7	0.6	1.2	1.9	2.9

Analyte detected above detection limit
Analyte also detected in Field Blank

APPENDIX E

Exhaust Monitoring Data for Schedule-2 Compounds

Sample Location	Sample Date	Compound	Result ($\mu g/M^3$)	Detection Limit($\mu g/M^3$)
Inlet S11	4/13/99	2-chloroethyl (2-chloroethoxy)	ND	0.21
Inlet S11	4/28/99	2-chloroethyl (2-chloroethoxy)	ND	0.19
Outlet S12	4/13/99	2-chloroethyl (2-chloroethoxy)	ND	0.21
Outlet S12	4/20/99	2-chloroethyl (2-chloroethoxy)	ND	0.20
Outlet S12	4/28/99	2-chloroethyl (2-chloroethoxy)	ND	0.19
Inlet S11	4/13/99	2-methyl-1-propene	ND	0.21
Inlet S11	4/28/99	2-methyl-1-propene	ND	0.19
Outlet S12	4/13/99	2-methyl-1-propene	ND	0.21
Outlet S12	4/20/99	2-methyl-1-propene	ND	0.20
Outlet S12	4/28/99	2-methyl-1-propene	ND	0.19
Inlet S11	4/13/99	Bis (2-chloroethyl) disulfide	ND	0.21
Inlet S11	4/28/99	Bis (2-chloroethyl) disulfide	ND	0.19
Outlet S12	4/13/99	Bis (2-chloroethyl) disulfide	ND	0.21
Outlet S12	4/20/99	Bis (2-chloroethyl) disulfide	ND	0.20
Outlet S12	4/28/99	Bis (2-chloroethyl) disulfide	ND	0.19
Inlet S11	4/13/99	BPCRS	ND	0.21
Inlet S11	4/28/99	BPCRS	ND	0.19
Outlet S12	4/13/99	BPCRS	ND	0.21
Outlet S12	4/20/99	BPCRS	ND	0.20
Outlet S12	4/28/99	BPCRS	ND	0.19
Inlet S11	4/13/99	CCBS	ND	0.21
Inlet S11	4/28/99	CCBS	ND	0.19
Outlet S12	4/13/99	CCBS	ND	0.21
Outlet S12	4/20/99	CCBS	ND	0.20
Outlet S12	4/28/99	CCBS	ND	0.19
Inlet S11	4/13/99	CECPRS	ND	0.21
Inlet S11	4/28/99	CECPRS	ND	0.19
Outlet S12	4/13/99	CECPRS	ND	0.21
Outlet S12	4/20/99	CECPRS	ND	0.20
Outlet S12	4/28/99	CECPRS	ND	0.19
Inlet S11	4/13/99	1,4-DITHANE	ND	0.21
Inlet S11	4/28/99	1,4-DITHANE	9	
Outlet S12	4/13/99	1,4-DITHANE	ND	0.21
Outlet S12	4/20/99	1,4-DITHANE	ND	0.20
Outlet S12	4/28/99	1,4-DITHANE	ND	0.19

Sample Location	Sample Date	Compound	Result ($\mu g/M^3$)	Detection Limit($\mu g/M^3$)
Inlet S11	4/13/99	DVS	ND	0.21
Inlet S11	4/28/99	DVS	ND	0.19
Outlet S12	4/13/99	DVS	ND	0.21
Outlet S12	4/20/99	DVS	ND	0.20
Outlet S12	4/28/99	DVS	ND	0.19
Inlet S11	4/13/99	MTS	ND	0.21
Inlet S11	4/28/99	MTS	ND	0.19
Inlet S11	4/13/99	Q	ND	0.21
Inlet S11	4/28/99	Q	ND	0.19
Outlet S12	4/13/99	Q	ND	0.21
Outlet S12	4/20/99	Q	ND	0.20
Outlet S12	4/28/99	Q	ND	0.19
Inlet S11	4/13/99	THIOXANE	ND	44.11
Inlet S11	4/28/99	THIOXANE	117	
Outlet S12	4/13/99	THIOXANE	ND	0.21
Outlet S12	4/20/99	THIOXANE	ND	0.20
Outlet S12	4/28/99	THIOXANE	ND	0.19

APPENDIX F
Abbreviations and Acronyms for Dioxins, Furans, and Agent Related Breakdown Products

Class	Acronym	CAS Number	Synonym(s)
Agent Related	BPCRS	22535-54-2	ClCH2CH2CH2SCH2CH2CH2Cl
Agent Related	BPCRS	22535-54-2	Bis(3-chloropropyl) sulfide
Agent Related	CCBS	114811-35-7	2-Chloroethyl 4-chlorobutyl sulfide
Agent Related	CCBS	114811-35-7	ClCH2CH2CH2CH2SCH2CH2Cl
Agent Related	CECPRS	71784-01-5	2-Chloroethyl 3-chloropropyl sulfide
Agent Related	CECPRS	71784-01-5	ClCH2CH2CH2SCH2CH2Cl
Agent Related	MTS	19149-77-0	ClCH2CH2SSSCH2CH2Cl
Agent Related	MTS	19149-77-0	Bis(2-chloroethyl) trisulfide
Agent Related	Q	3563-36-8	1,2-Bis(2-chloroethylthio)ethane
Agent Related	Q	3563-36-8	ClCH2CH2SCH2CH2SCH2CH2Cl
Agent Related	Q	3563-36-8	Sesquimustard
Agent Related	DVS	627-51-0	Divinyl sulfide
Agent Related	DVS	627-51-0	Ethene, 1,1'-thiobis-
Agent Related	RDX		Research Department Explosive Cyclotrimethylenetrinitrane (RDX)
Agent Related	HMX		High Melting Explosive Octogen, also cyclotrimethylene-tetranitrane
Agent Related	PCP		Penta-Chloro-Phenol
Dioxin/Furan	1,2,3,4,6,7,8-HpCDD		1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
Dioxin/Furan	1,2,3,4,6,7,8-HpCDF		1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
Dioxin/Furan	1,2,3,4,7,8,9-HpCDF		1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
Dioxin/Furan	1,2,3,4,7,8-HxCDD		1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
Dioxin/Furan	1,2,3,4,7,8-HxCDF		1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
Dioxin/Furan	1,2,3,6,7,8-HxCDD		1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
Dioxin/Furan	1,2,3,6,7,8-HxCDF		1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
Dioxin/Furan	1,2,3,7,8,9-HxCDD		1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
Dioxin/Furan	1,2,3,7,8,9-HxCDF		1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
Dioxin/Furan	1,2,3,7,8-PeCDD		1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
Dioxin/Furan	1,2,3,7,8-PeCDF		1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
Dioxin/Furan	13C-1,2,3,4,6,7,8-HPCDD (IS)	109719-83-7	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin-C13
Dioxin/Furan	13C-1,2,3,4,6,7,8-HPCDF (IS)	109719-84-8	1,2,3,4,6,7,8-Heptachlorodibenzofuran-C13
Dioxin/Furan	13C-1,2,3,4,7,8-HXCDF (IS)	114423-98-2	1,2,3,4,7,8-Hexachlorodibenzofuran-C13
Dioxin/Furan	13C-1,2,3,6,7,8-HXCDD (IS)	109719-81-5	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin-C13
Dioxin/Furan	13C-1,2,3,7,8-PECDD (IS)	109719-79-1	1,2,3,7,8-Pentachlorodibenzo-p-dioxin-C13
Dioxin/Furan	13C-1,2,3,7,8-PECDF (IS)	109719-77-9	1,2,3,7,8-Pentachlorodibenzofuran-C13
Dioxin/Furan	13C-2,3,7,8-TCDD (IS)		2,3,7,8-Tetrachlorodibenzofuran-C13
Dioxin/Furan	13C-OCDD (IS)	114423-97-1	Octachlorodibenzo-p-dioxin-C13
Dioxin/Furan	2,3,4,6,7,8-HxCDF		2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
Dioxin/Furan	2,3,4,7,8-PeCDF		2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
Dioxin/Furan	2,3,7,8-TCDD		2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
Dioxin/Furan	2,3,7,8-TCDF		2,3,7,8-Tetrachlorodibenzofuran (TCDF)
Dioxin/Furan	OCDD	3268-87-9	Octachlorodibenzo-p-dioxin (OCDD)
Dioxin/Furan	OCDF	39001-02-0	Octachlorodibenzofuran (OCDF)
Dioxin/Furan	Total HpCDD		TOTAL HEPTA-DIOXINS
Dioxin/Furan	Total HpCDD		Heptachlorodibenzo-p-dioxins (HpCDD), Total
Dioxin/Furan	Total HpCDF		TOTAL HEPTA-FURANS
Dioxin/Furan	Total HpCDF		Heptachlorodibenzofurans (HpCDF), Total
Dioxin/Furan	Total HxCDD	34465-46-8	TOTAL HEXA-DIOXINS

Class	Acronym	CAS Number	Synonym(s)
Dioxin/Furan	Total HxCDD	34465-46-8	Hexachlorodibenzo-p-dioxins (HxCDD), Total
Dioxin/Furan	Total HxCDF		TOTAL HEXA-FURANS
Dioxin/Furan	Total HxCDF		Hexachlorodibenzofurans (HxCDF), Total
Dioxin/Furan	Total PeCDD		TOTAL PENTA-DIOXINS
Dioxin/Furan	Total PeCDD		Pentachlorodibenzo-p-dioxin (PeCDD), Total
Dioxin/Furan	Total TCDF	55722-27-5	Tetrachlorodibenzofurans (TCDF), Total
Dioxin/Furan	Total PeCDF		Pentachlorodibenzofurans (PeCDF), Total
Dioxin/Furan	Total TCDD		TOTAL TETRA-DIOXINS
Dioxin/Furan	Total TCDD		Tetrachlorodibenzo-p-dioxins (TCDD), Total
Dioxin/Furan	Total TCDF	55722-27-5	TOTAL TETRA-FURANS