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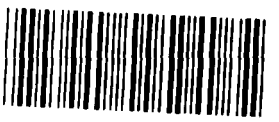

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U.S. Army Program Manager for Chemical Demilitarization

Test Report for Laboratory- and Bench-Scale Biodegradation of Neutralized HD Mustard Agent

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5 February 1996

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SECTION 1
EXECUTIVE SUMMARY

1. EXECUTIVE SUMMARY

1.1 Introduction

The U.S. Army's Program Manager for Chemical Demilitarization (PMCD) and the National Research Council (NRC) have recently completed a congressionally mandated project to examine alternatives to incineration for the destruction of the United States stockpile of chemical weapons. Congress has funded additional evaluation and testing of alternative technologies for the disposal of two chemical warfare agents, mustard agent (HD) and nerve agent (VX). PMCD is responsible for this effort. This research and development (R&D) effort is referred to as the Alternative Technology Program (ATP). The objective of the ATP is to develop the data required to support a decision as to whether a technology should be demonstrated in pilot scale. A Defense Acquisition Board (DAB) will make this final decision in October 1996.

The technologies being tested for the ATP are stand-alone neutralization and neutralization followed by biodegradation. In the Chemical Stockpile Disposal Program (CSDP), neutralization is defined as the act of altering chemical, physical, and toxicological properties to render the chemical agent ineffective for use as intended [Draft Army Regulation (AR) 385-61]. These technologies have been determined to have the best chance to achieve low-temperature [less than or equal to 212°F (100°C)] and low-pressure (less than or equal to 1 atmosphere) destruction of the chemical agents. The Army is pursuing these technologies for use at bulk-only storage sites where ton containers are filled with either HD or VX. Since bulk-only sites contain no munitions configured with explosives or propellants, destruction of the latter items is not an element in the ATP. All test results will be evaluated independently by the Army

Materiel Systems Analysis Activity (AMSAA) for a Milestone I/II in-process review (IPR) decision.

This test report is specifically directed at the evaluation of biodegradation for the neutralization-biodegradation process for the chemical agent HD (mustard).

1.2 Objectives

The purpose of the test described herein is to collect data on biodegradation of HD hydrolysate through laboratory- and the bench-scale testing. Information obtained from this test will be used for further evaluation of the technology and to obtain process information, including operating and design data, necessary to support the pilot plant design. The intent of the test is to further develop the biodegradation process, and demonstrate scalability and stability of the process.

The specific objectives of the biodegradation test described herein are as follows:

a. to develop and demonstrate a stable biological treatment process that is capable of achieving performance objectives through laboratory- and bench-scale subtests

b. to collect process information necessary for design and demonstration of the process at the pilot scale.

1.3 Test Summary

The main component of the test system is an aerobic sequencing batch reactor (SBR), in which biodegradation reactions are allowed to occur by contacting biomass with the water hydrolysis product of HD.

For this test, 2.5- to 5-liter (L) fermenters for the laboratory-scale subtests and 80-L fermenters for the bench-scale subtests were operated as aerobic SBRs. The function of the SBR is to convert the organics present in the hydrolysate into carbon dioxide, water, inorganics (such as sulfate), and biomass. The general procedure for biodegradation testing consisted of feeding the HD hydrolysate (after pH adjustment and addition of nutrients) to the bioreactors daily. The bioreactors were initially seeded with activated sludge from the Back River Wastewater Treatment Plant, Baltimore, MD, or acclimated biomass from laboratory feasibility testing. The laboratory and bench bioreactors were operated using SBR conditions developed in feasibility tests. Sampling of the reactor liquid was conducted to monitor the operation of the SBR and assess performance. Operating data, such as pH, temperature, and dissolved oxygen (DO) of the SBR, were collected. Excess biomass was removed from the SBR daily and settled effluent was drawn from the reactor daily and stored. All streams and major units used in the process were sampled and analyzed for chemical characterization.

The testing series consisted of four laboratory-scale (2.5 to 5L) and three bench-scale (80L) biodegradation subtests. Four separate subtests were conducted at the laboratory scale. Three of these subtests (subtest nos. 1 A, 1 B, and 1 C) were conducted to test biodegradation of 1.27, 3.8, and 8.6 weight percent (wt %) HD/water hydrolysate using unacclimated biomass. The fourth laboratory subtest (subtest no, 2) collected data on the biodegradation of 8.6 wt % HD/water hydrolysate using acclimated biomass. Subtest nos. 3, 4A, and 4B were conducted to demonstrate the biodegradation process for 1.27, 4.1, and 8.6 wt % HD/water hydrolysate at the bench scale in 80-L reactors using unacclimated biomass,

1.4 Test Results

1.5 Conclusions and Recommendations

SECTION 2
BACKGROUND AND SUMMARY OF TESTING PERFORMED

2. BACKGROUND

This test report focuses on the biodegradation step of the neutralization-biodegradation process for demilitarization of HD. This test report provides the results of the testing intended to generate process data for the biodegradation of the water hydrolysis product of HD. Prior to the testing described herein, the information on the neutralization-biodegradation process consisted of feasibility testing conducted at the Edgewood Research, Development, and Engineering Center (ERDEC), which indicated that biodegradation of HD/water hydrolysate is feasible. The feasibility tests established that an organic removal efficiency (ORE) of approximately 88 percent can be achieved under aerobic conditions in an SBR using activated sludge as the seed biomass. The tests described herein enhance this state of knowledge by attempting to prove the biodegradation process and by collecting process data from the operation of laboratory- and bench-scale bioreactors.

2.1 Introduction

The test described in this report consists of laboratory-scale (2.5 to 5L) and bench-scale (80L) subtests. In these subtests, biodegradation of hydrolysate generated from HD loadings of 1.27, 3.8, 4.1, and 8.6 wt % was studied. Biodegradation of the water hydrolysis product generated at an HD loading of 1.27 wt % has been tested in feasibility experiments. Hydrolysis at a higher HD loading followed by biodegradation of the hydrolysate diluted to an organic concentration comparable to the 1.27 wt % hydrolysate is the design basis for the pilot facility. Therefore, hydrolysates obtained from different HD loadings were studied. The hydrolysate obtained contains dissolved iron, which precipitates (along with other metals) when the pH of the hydrolysate is

raised to a value above 7. In all subtests, the bioreactor feedstock consisted of the hydrolysate with the metals floc included from startup.

The testing series consists of four laboratory-scale and three bench-scale biodegradation subtests. The test matrix is shown in table 2-1. The test performance was determined by measuring the removal efficiency of organic compounds in terms of the concentration of thiodiglycol (TDG), total organic carbon (TOC), and chemical oxygen demand (COD).

At the laboratory scale, four separate subtests were conducted. Three of these subtests (subtest nos. 1 A, 1 B, and 1 C) were conducted for testing biodegradation of 1.27, 3.8, and 8.6 wt % HD/water hydrolysate using unacclimated biomass. The fourth laboratory subtest (subtest no. 2) collected data at the 8.6 wt % loading using acclimated biomass. Subtest nos. 3, 4A, and 4B involved demonstration of the biodegradation process for 1.27, 4.1, and 8.6 wt % HD/water hydrolysate at the bench scale in 80-L reactors. All of these subtests involved including the precipitated metals floc in the bioreactor feedstock.

The information to be gained from the completion of the laboratory- and bench-scale subtests is proof of biodegradation of hydrolysate produced at higher HD loadings. Additionally, biodegradation process data necessary for design and demonstration of the process at the pilot scale is to be collected. The test data to be generated from this testing are intended to meet the data generation requirements for the critical technical parameters identified in the Test and Evaluation Master Plan (TEMP) for the CSDP and the Laboratory- and Bench-Scale Test Plan for Biodegradation of Neutralized HD Mustard.

2.2 Test Objectives and Criteria

The purpose of the testing described herein is to demonstrate the biodegradation process for HD through laboratory- and bench-scale testing. Information obtained from this test will be used for further evaluation of the technology and to obtain process information, including operating and design data, necessary to support the pilot plant design. The intent of the test is to further develop, and demonstrate scalability and stability of the biodegradation process.

The specific objectives of the biodegradation test described herein are as follows:

a. to develop and demonstrate a stable biological treatment process that is capable of achieving performance objectives through laboratory- and bench-scale subtests

b. to collect process information necessary for design and demonstration of the process at the pilot scale.

Objective a. The overall objective is to develop and prove a stable biological treatment process that is capable of effectively degrading organic compounds present in the hydrolysate. Specifically, the objectives are as follows:

- to determine whether a stable biological treatment process for the hydrolysate prepared using HD loadings of 1.27, 3.8, and 8.6 wt % can be developed and to determine the ORE that can be achieved

- to obtain OREs in bench tests equal to or greater than that demonstrated in laboratory tests

- to meet the Chemical Weapons Convention (CWC) treaty requirements by demonstrating a TDG removal efficiency of greater than 99 percent

- to demonstrate that the mixed liquor suspended solids (MLSS) concentration can be maintained without washout conditions. Washout conditions will be defined as an uncontrolled, excessive loss of biomass from the reactor into the liquid effluent stream and will be defined in this test report as a greater than 1000-mg/L reduction in the MLSS over a period of 1 week.

Objective b. This objective is to collect design data and determine feed and effluent characteristics from the laboratory-scale and bench-scale aerobic SBRs.

The draft TEMP for the CSDP requires the generation of data for the following critical technical parameters: temperature, pressure, sampling time, reaction time, agent destruction efficiency (ADE), thermodynamics of reaction, reaction products, mixing requirements, material corrosion, and concentrations of hazardous pollutants in the air, liquid, and solids. For biodegradation, temperature, pressure, sampling time, reaction time, and mixing conditions used in the subtests will be stated. ADE is not required for biodegradation because agent destruction occurs in the hydrolysis step prior to biodegradation. The heat of reaction of biodegradation is applicable thermodynamic information and will be estimated from theoretical considerations as part of the pilot plant design. Material corrosion will be considered in materials of construction testing. Reaction products and concentrations of hazardous pollutants in the air, liquid, and solid streams will be determined under this objective. For this test: the following biodegradation data will be collected and/or calculated.

- operating data
- ORE
- organic loading

- biomass yield
- HRT
- solids retention time (SRT)
- oxygen consumption rate (OCR)
- settling characteristics
- chemical characteristics of feed and effluent streams.

The pass/fail criterion for the subtests is as follows: a subtest will be considered successful if the minimum quantitative data requirements are met for the duration of testing. These data are: ORE (in terms of TDG, TOC, and COD reduction), MLSS, effluent total suspended solids (TSS), OCR, and chemical characteristics of effluent streams. In addition, temperature, pressure, sampling time, reaction time, and mixing conditions should be recorded and chemical characteristics of the liquid effluent and excess biomass streams should be determined.

In addition, the following criteria will be used to evaluate the generated data. These criteria are related to the evaluation of the test performance.

a. The TDG removal efficiency will be calculated to determine if treaty requirements of greater than 99 percent removal efficiency have been met. This is not a pass/fail criterion, because further TDG removal may be obtained during posttreatment of the bioreactor effluents.

b. The ORE will be calculated and compared with that obtained in the feasibility tests (88 percent TOC-based ORE). Evaluation of the ORE will be qualitative. The OREs obtained for hydrolysates from different loadings may be different. Analysis of the data obtained will consist of a comparison of OREs for different loadings. Also, this is not a pass/fail criterion because further organic removal may be obtained during posttreatment of the bioreactor liquid effluent.

c. The data will be evaluated to determine whether the biodegradation process is stable. The evaluation will be conducted by calculating the range of OREs obtained during the test period of three HRTs or one SRT (whichever is longer). If the ORE profile shows a continuously decreasing trend and/or the OREs are not within approximately +/-5 percent deviation from the average value, then the test will be interpreted as being unstable.

d. The MLSS data will be evaluated to determine if the MLSS can be maintained in the bioreactors during the test period. The data will be evaluated qualitatively. A minimum MLSS of approximately 2500 mg/L should be maintained in the bioreactor. If the MLSS profile indicates a continuously decreasing trend and an uncontrolled decrease of more than 1000 mg/L in a period of 1 week, then the process will be interpreted as not viable for the conditions tested.

2.3 Summary of Tests

The parameters that distinguish the individual subtests are HD loading, scale of testing, and the type of seed biomass. Hydrolysate prepared from HD loadings of 1.27, 3.8, and 8.6 wt % were tested. Four of the subtests were conducted in laboratory-scale bioreactors (2.5 to 5L) and three subtests were conducted in bench-scale bioreactors (80L). In addition, one laboratory-scale test (subtest no. 2) used acclimated biomass while the remaining subtests used activated sludge as the seed biomass.

All of the subtests used a similar test setup and were conducted using essentially similar procedures. The biodegradation subtests consist of the following general steps: feed preparation, bioreactor operation, liquid effluent withdrawal and filtration, and excess biomass removal and digestion. During testing, all streams and major units used in the process were sampled and analyzed for chemical characterization. Descriptions of the test setup and test procedures for individual subtests are provided

in section 3. The general procedures followed are described in the following paragraphs.

The hydrolysate was received from the generation point and the pH verified. The pH of the hydrolysate was measured and adjusted to 11, with 2.5N sodium hydroxide (NaOH). The hydrolysate was manually transferred to a feed storage tank, which was maintained at 39°F (4°C). The hydrolysate was mixed with a laboratory mixer and transferred from the refrigerated feed storage tank into a feed day tank. Macronutrients (nitrogen and phosphorus) and micronutrients (trace metals) were added manually to the feed day tank. Approximately 1440 mg/L of ammonium chloride and 277.5 mg/L of potassium dihydrogen phosphate were used as the sources of nitrogen and phosphorus, respectively. These concentrations were adjusted during the test so that the effluent ammonia-nitrogen concentration was 5 to 10 mg/L and the orthophosphate-phosphorus concentration was 0 to 5 mg/L. Micronutrients were added to the hydrolysate in the form of a standard Wolin Salts solution. Sodium bicarbonate, at a concentration of 12 g/L, was added as a solid for buffering the reactor liquid contents.

Following reactor startup, the SBR was operated under the acclimation phase, followed by the test phase. The startup procedure involved calibration of pH and DO probes and preparation of the reactor for operation. This preparation included connecting the base supply, feed addition, aeration, and effluent withdrawal lines. After preparation, the reactor was seeded with activated sludge obtained from the Back River Wastewater Treatment Plant, Baltimore, MD. The SBR was operated by following the daily cycle consisting of Fill, React, Settle, and Draw periods. During the Fill period, the hydrolysate was added to the SBR over a 5-hour period. The aeration and agitation systems were turned on at the start of the Fill period. Aeration was provided at a rate sufficient to maintain a minimum DO concentration (as measured by the online DO probe) of 10 percent oxygen saturation during periods when the rate of oxygen consumption was the highest. The React period was initiated following the completion of the Fill period. Aeration and agitation were continued during the React period.

During the Fill and React periods, the pH of the mixed liquor was maintained between pH 6.8 and 8.5(+/-0.2) units. The React period was terminated by turning off agitation and mixing. The suspended solids were allowed to settle for 1 hour (Settle period). Liquid effluent was then drawn off (Draw period). Excess biomass was removed as mixed liquor from the SBR during the last hour of the React period to accomplish wasting and to maintain the desired SRT in the range of 15 to 60 days.

During the acclimation phase, the SBR was operated at HRTs in the range of 10 to 20 days. The HRT was adjusted based on an evaluation of the effluent TOC and COD, OCR, and DO profile data. If the effluent TOC and COD values and OCR values at the end of the React period continued to increase, and the DO profiles indicated less than 4 hours of excess treatment capacity (that is, the time period during the end of the React phase when the DO concentration is at its maximum), then the acclimation period was extended and the HRT increased. If organic compounds accumulated in the reactor, temporary periods of 1 day of no feed were used to allow for consumption of residual organic compounds. After stable TOC and COD values, stable effluent suspended solids concentrations, stable OCR values, and consistent DO profiles were obtained for a period of 1 week, the test period was initiated. During the test period, the SBR was operated at the target HRT for the duration of the test. During daily operation, the SBR liquid effluent was collected in an effluent day tank and filtered using a filtration apparatus. The excess biomass was transferred to a 1-L graduated glass cylinder for gravity settling. The supernatant was decanted and used as liquid effluent. For the bench-scale tests, the settled sludge was manually added to a continuously aerated aerobic digester tank, which served to store and digest the excess biomass.

Data was collected by sampling and analysis of the various streams and units used in the process. The type and frequency of data collection was specific to each subtest and is provided in detail in section 3. In general, the sampling and analysis strategy

consisted of collecting data on the feed characteristics, on SBR operating data, and on the liquid effluent and excess biomass (before and after digestion) characteristics.

SECTION 3
SUBTESTS

3. SUBTESTS

This section describes the objectives, test criteria, setup, procedures, results, and analysis of results for each of the subtests.

3.1 Subtest Nos. 1A, 1B, and 1C

3.2 Subtest No. 2

3.3 Subtest No. 3

In subtest no. 3, biodegradation of HD/water hydrolysate prepared at a 1.27 wt % loading was tested at the bench scale.

3.3.1 Specific Objectives of Subtest No. 3. The specific objectives of subtest no. 3 are:

a. to demonstrate biological treatment of HD/water hydrolysate (with the metals floc included) prepared at a 1.27 wt % loading

b. to collect process information necessary for design and demonstration of the process at the pilot scale.

3.3.2 Subtest No. 3 Criteria. Subtest no. 3 will be considered successful if the minimum quantitative data for the biodegradation test are met for the duration of testing (that is, the period of three HRTs or one SRT). These data are ORE (in terms of TDG, TOC, and COD reduction), MLSS concentration, effluent TSS concentration, OCR, and

chemical characteristics of effluent streams. In addition, temperature, pressure, sampling time, reaction time, and mixing conditions should be recorded and chemical characteristics of the liquid effluent and excess biomass streams should be determined.

In addition, the following criteria will be used to evaluate the generated data. These criteria are related to the evaluation of the test performance.

a. The TDG removal efficiency will be calculated to determine if treaty requirements of greater than 99 percent removal efficiency have been met.

b. The ORE will be calculated and compared with that obtained in the feasibility tests (88 percent TOC-based ORE). Evaluation of the ORE will be qualitative. The OREs obtained for hydrolysates from different loadings may be different. Analysis of the data obtained will consist of a comparison of removal efficiencies for different loadings. This is not a pass/fail criterion because further organic removal may be obtained during posttreatment of the bioreactor liquid effluent.

c. The data will be evaluated to determine whether the biodegradation process is stable. This evaluation will be done by calculating the range of OREs obtained during the testing period of three HRTs or one SRT (whichever is greater). If the ORE profile shows a continuously decreasing trend and/or the removal efficiencies are not within approximately +/-5 percent deviation from the average value, then the test will be interpreted as being unstable.

d. The MLSS data will be qualitatively evaluated to determine if the MLSS concentration can be maintained in the bioreactors during the test phase. A minimum MLSS concentration of approximately 2500 mg/L should be

maintained in the bioreactor. If the MLSS concentration profile indicates a continuously decreasing trend and a decrease of more than 1000 mg/L over a period of 1 week, then the process will be interpreted as not viable for the conditions tested.

3.3.3 Subtest No. 3 Setup. The main unit for this subtest was a BioFlo 5000 80-L fermenter manufactured by New Brunswick Scientific Co., Inc. (Edison, NJ). All other units were either polyethylene or glass containers, which were used for various material transfer and storage operations. These units either involved manual transfers or were interconnected by flexible PharMed® (polypropylene) tubing. Complete details on the physical configuration, specifications, and the associated instrumentation for the fermenter are available in the manufacturer's operating manual for the BioFlo 5000 model (New Brunswick Scientific Co., Inc., 1995). The physical setup of the fermenter and the required tubing and electrical connections for operation as an SBR are provided in the process Standing Operating Procedure (SOP) (Biodegradation of HD/Water Hydrolysate, CR9-2NP004). A brief description of the fermenter and a discussion of the system components relevant to operation as an SBR follows.

The fermenter is designed with microprocessor control and display of pH, DO, temperature, and agitation. The fermenter consists of a pressure vessel with a working volume of 60L. The pressure within the fermenter vessel is 1 atmosphere.

Aeration. A ring-type sparger, which enters the side wall of the fermenter, was used for supplying air to the reactor liquid. The ring is located beneath the impeller (diameter of 6 inches) and has a diameter of 4.5 inches. For this test, an aeration rate in the range of 6 to 7 L/minute was used.

Temperature Control. The temperature of the reactor liquid is measured by a resistance temperature device (RTD) probe and is controlled by circulation of water

through the vessel jacket. For this test, the temperature was maintained at a constant value between 77°F (25°C) and 86°F(30°C).

Agitation. Two axial flow impellers mounted on a shaft, driven by a direct current motor, were used for agitation. These impellers provide for low-shear mixing of the reactor liquid so that successful flocculation and settling of biomass can be achieved. An agitation speed of 250 revolutions per minute (rpm) was used.

Instrumentation. The pH of the reactor liquid was sensed by a glass pH probe placed through a port in the reactor wall. The pH was controlled in the range of 6.8 to 8.5(+/-0.2) units using the proportional pH control system of the fermenter. Since the biodegradation reaction is acidic, only the base supply was required. DO is measured using a polarographic probe placed through a port in the reactor wall.

Pumps. One of the three peristaltic pumps mounted on the reactor console was used for base addition. In addition, peristaltic pumps for feed addition to the reactor and withdrawal of liquid effluent were used.

3.3.4 Subtest No. 3 Procedures. The following paragraphs describe the procedures used for subtest no. 3. The test conditions are summarized in table 3-3-l.

a. *Bench-Scale Hydrolysa te Preparation*

The procedures used for the generation of hydrolysate by neutralization are described in the SOP, Small Quantity Agent Operations in the Chemical Transfer Facility. After completion of the reaction, NaOH was added to increase the pH of the hydrolysate to 11. The amount of NaOH added for a 12-L batch of hydrolysate was 84g (as solid).

Table 3-3-l. Summary of Conditions for Subtest No. 3

Parameter	Value
Feedstock	1.27 wt % HD/water hydrolysate, pH-adjusted to greater than 10
Ammonium chloride (NH_4Cl)	1440 mg/L or lower
Potassium dihydrogen phosphate (KH_2PO_4)	277.5 mg/L or lower
Micronutrients	Wolin Salts (10 mL/L)
Buffer	$NaHCO_3$ (12 g/L)
Reactor volume	60L
SBR cycle times	5-hour Fill (aerated), 17.5-hour React (aerated), 1 -hour Settle, 0.25-hour Draw, 0.25-hour Idle
Aeration	6-7 L/minute; fine bubble
PH	6.8-8.5
Agitation	250 rpm
Temperature	constant between 77°F (25°C) and 86°F (30°C)
Biomass source	Back River Wastewater Treatment Plant

b. *Hydrolysafe Transfer and Storage*

The hydrolysate was received from the source and the pH verified. The pH of the hydrolysate ranged from 1.2 to 2.5. The 2.5N NaOH or a combination of solid NaOH and 2.5N NaOH were added to raise the pH to 11. The hydrolysate was then manually transferred to a 30-gallon feed storage tank and was stored at 39°F (4°C).

For batches in which the iron floc was separated, approximately 50L of hydrolysate was added to a 15-gallon conical, polyethylene tank (iron floc tank). After 1 to 2 hours, the iron floc settled by gravity and the supernatant was drawn off using a peristaltic pump. An average of approximately 5L of iron floc was obtained from an average of 116L of hydrolysate.

c. *SBR Feed Preparation*

The hydrolysate was mixed with a laboratory mixer and transferred once a day from the refrigerated feed storage tank into a feed day tank (10-L polyethylene container for batches from which iron floc was removed and a 5-L glass container for other batches). Macronutrients (nitrogen and phosphorus) and micronutrients (trace metals) were added manually to the feed day tank. For each liter of feed, 10 mL of a 144 g/L stock solution of ammonium chloride and 10 mL of a 27.75 g/L stock solution of potassium dihydrogen phosphate were added. These concentrations were continually adjusted during the test (see paragraph 3.3.5 for details) so that the effluent ammonia-nitrogen concentration was approximately in the range of 5 to 10 mg/L and the orthophosphate concentration was in the range of 0 to 5 mg/L. Approximately 10 mL of a standard Wolin Salts

solution were added per liter of the SBR feed. Sodium bicarbonate, at a concentration of 12 g/L, was added as a solid for buffering capacity.

d. *SBR Operation*

Specific procedures for startup and operation of the SBR are described in the following paragraphs. Details of the instrument calibration procedures, the physical configuration of the fermenter, and the start-up procedures of the fermenter are available in the ERDEC process SOP, Biodegradation of HD/Water Hydrolysate, CR9-2NP004. The following steps were involved in the operation:

Step	Activity
A	Reactor Startup
B	Acclimation Phase
C	Test Phase

(1) *Step A - Reactor Startup.* The reactor was prepared for operation, as described in the manufacturer's operating manual and the process SOP (Biodegradation of HD/Water Hydrolysate, CR9-2NP004). The start-up procedure involved calibrations of pH and DO probes and preparation of the reactor for operation. This preparation included connecting the base supply, feed addition, aeration, and effluent withdrawal lines. The temperature of the reactor was maintained at a constant in the range between 77°F (25°C) and 86°F (30°C) using the automatic temperature control system (except for a period of approximately 8 hours when the temperature increased to 122°F (50°C) due to a system malfunction.

After preparation, the reactor was seeded with activated sludge obtained from the Back River Wastewater Treatment Plant, Baltimore, MD. The activated sludge was obtained from the return waste activated sludge line, with a concentration of approximately 10,000 mg/L of suspended solids. Approximately 30L of return activated sludge was added to the SBR and diluted using 30L of tap water. This procedure yielded a mixed liquor with a final MLSS concentration of 5,364 mg/L. The reactor was aerated for a day prior to the start of feed addition to remove any organics associated with the seed sludge. Prior to the first day of operation, 7L of mixed liquor were removed to lower the MLSS to approximately 4,500 mg/L.

(2) *Step B - Acclimation Phase.* The goal of this phase was to develop a microbial population, which would be capable of degrading the organic compounds in the hydrolysate at a target HRT. During this period, an initial HRT of 20 days was used. The 20-day HRT was based on conditions used in prior testing and is equivalent to a low organic loading. The HRT of the reactor was varied depending on reactor performance (described in paragraph 3.3.5). During acclimation, the SBR was operated as follows.

(a) *Fill (Acclimation* Phase). Initially, the feed pump was set at a flow rate to feed 3L of feed in 5 hours (Fill period) and activated at the start of the daily cycle. This Fill volume is equivalent to a 20-day HRT. For other HRTs, the Fill volume was calculated as the reactor volume divided by the HRT. The aeration and agitation systems were turned on at the start of the Fill period and the feed volume was added over the 5-hour Fill period. The impeller speed was set at

250 rpm. Aeration was provided in the range of 6 to 7 L/minute. During the acclimation phase, the iron floc was separated from the hydrolysate and the separated hydrolysate was used as the bioreactor feedstock.

(b) *React, Settle, and Draw (Acclimation Phase).* After the Fill period, aeration and agitation were continued during the React period. During the Fill and React periods, the pH of the mixed liquor was maintained between 6.8 and 8.5(+/-0.2) units. After 17.5 hours of the React period, agitation and mixing were turned off. The suspended solids were allowed to settle for 1 hour (Settle period). Liquid effluent was then drawn off during the 0.5-hour Draw period using a peristaltic pump. The volume of liquid effluent withdrawn was equal to the difference between the Fill volume and excess biomass volume removed. To accomplish wasting, excess biomass was removed as mixed liquor from the SBR during the last hour of the React period. The volume of excess biomass to be removed was calculated as the reactor volume divided by the desired SRT. The SRT used during the test is described in paragraph 3.3.5.

(3) *Step C - Test Phase.* After the acclimation phase, the SBR operation was continued. A 20-day HRT was maintained for the 60-day test phase. This HRT was determined to be acceptable for reactor operation and was maintained during the test phase. For the first 17 days of the test phase, the bioreactor feed consisted of the hydrolysate separated from the iron floc. For the remaining duration of the test (43 days), the iron floc was not separated and

the hydrolysate containing the iron floc was used as feed. Excess biomass was removed as described for the acclimation phase.

e. *Filtration and Storage of Liquid Effluent*

The SBR liquid effluent was collected in an effluent day tank and then filtered, using filtration apparatus, on a daily basis. The filter paper and solids were placed in a polyethylene, l-gallon container for analysis. The filtered effluent was transferred to the Treated Effluent Storage Tank (working volume of 30 gallons) for storage and analysis.

f. *Excess Biomass Processing*

The excess biomass removed from the SBR was transferred to a I-L graduated glass cylinder for gravity settling. The supernatant was decanted and used as liquid effluent. The settled sludge was manually added to the Aerobic Digester Tank, which served to store the excess biomass. The aerobic digester was a polyethylene tank with fine bubble aeration. Two batch tests were conducted for aerobic digestion. The purpose of the first batch was to collect excess biomass during the test and obtain operating data during the biodegradation test. To collect data on metals and organics of digested and filtered solids (which required a larger volume of excess biomass), a second batch was tested. This second batch used residual biomass from the SBR after completion of the test phase.

(1) *Aerobic Digester (First Batch).* Approximately 5L was accumulated in a 1 O-L aerobic digester, which was continuously aerated. After this level was reached, the temporary digester was operated for 10 days at a 1 O-day HRT. This was accomplished by removing,

from the digester, an amount equivalent to the daily excess biomass wasted from the bioreactor. The wasted excess biomass from the bioreactor was then added to the digester. The removed digester contents were stored in a separate container with aeration. The pH of the digester contents was checked daily and adjusted to pH 7 using 1 N NaOH. After 10 days, the digester contents were analyzed (see step g for parameters and paragraph 3.3.5 for results).

(2) *Aerobic Digester (Second Batch).* Following completion of the test, residual biomass from the SBR was settled and transferred to the Aerobic Digester Tank (___-L capacity) and aerated for 10 days. The volume of the excess biomass obtained from the SBR was approximately 16L. At the end of the 1 O-day aeration period, the digester was sampled for analysis (see step g for parameters and paragraph 3.3.5 for results), and then the digester contents were sent to a vendor for dewatering. The dewatered solids were analyzed (see step g for parameters and paragraph 3.3.5 for results). During the 10-day aeration period, the pH of the digester contents was checked and adjusted to 7 using 1 N NaOH when the pH was less than 7.

g. *Bench Subtest Data Requirements*

The type of data collected and the frequency of data collection is described here. Samples for chemical analysis were collected from the following streams and units.

- hydrolysate
- bioreactor unit

- bioreactor liquid effluent stream
- bioreactor offgas
- excess biomass stream
- aerobic digester
- aerobic digester offgas
- suspended solids from effluent filtration.

(1) *Analytical Procedures.* Analytical procedures used for the analysis of samples were from the following references:

- Standard Methods for the Examination of Water and Wastewater [American Public Health Association (APHA), American Water Works Association (AWWA), and Water Environment Federation (WEF), 18th edition, 1992];

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods [Environmental Protection Agency (EPA) publication SW-846, 3rd edition]; and,

- Methods for Chemical Analysis of Water and Wastes (EPA, EPA-600/4-79-020, revised March 1983).

The analytical methods used for each parameter are listed in table 3-3-2.

(2) Sampling *and Analysis Strategy.* The samples collected, sampling volume and frequency used, and the parameters analyzed are summarized in table 3-3-3. The sampling strategy is detailed as follows.

Table 3-3-3. Summary of Sampling Strategy

Sampling Point	Parameter	Sample Volume (mL)	Sampling Frequency
Hydrolysate	pH, TDS, TSS, Alkalinity	200	Batch"
	Organosulfur compounds	80	Batch
	svocs	1000	Batch
	VOCs	40	Batch
	TDG	80	Batch
	Metals	500	Batch
	TOC	40	Batch
	BOD	500	Batch
	COD	30	Batch
	Sulfate (SO_4)	25	Batch
	NH,-N, soluble ortho PO_4[b]	50	Monthly
Bioreactor Unit	MLSS	50	5/week
	MLVSS	[c]	2/week
	ssv, zsv	1000[d]	3/week or more
	OCR	30	[e]
Bioreactor Effluent'	TSS	250	2/week or more
	NH,-N, NO,-N, NO,-N, soluble ortho PO,	105	3/week
	TDS, VSS	[g]	Weekly
	Alkalinity	100	Weekly
	SO_4	25	Biweekly
	TOC	40	Daily
	COD (total)	30	Twice
	COD (soluble)	30	Daily
	BOD (total)	500	Weekly
	BOD (soluble)	500	Weekly
	HD	250	After each HRT
	Metals	500	a times
	TDG	80	a times
	VOCs	40	a times
	svocs	1000	a times

Table 3-3-3. Summary of Sampling Strategy (Continued)

Sampling Point	Parameter	Sample Volume (mL)	Sampling Frequency
	Organosulfur compounds	800	a times
	Organochlorine pesticides and PCBs	1000	Once
	TPH	1000	Once
	Total phosphorus	1000	Once
	Total cyanide	1000	Once
	Dioxin (TCDD)	1000	Once
Bioreactor Offgas	VOCs	SUMMA@ canisters	4 times[h]
Excess Biomass	TSS, VSS	125	After second and third HRT
	HD	250	
	Metals	500	
	TDG	80	
	VOCs	40	
	svocs	1000	
	Organosulfur compounds	800	
Aerobic Digester (First Batch)	TSS, VSS	100	2/week and end of batch'
	OCR	25	2/week
	COD (soluble)	30	2/week and end of batch
	HD	250	End of batch
	Metals	500	End of batch
	TDG	80	End of batch
	VOCs	40	End of batch
	svocs	1000	End of batch
	Organosulfur compounds	80	End of batch
	BOD (soluble)	500	End of Batch

Table 3-3-3. Summary of Sampling Strategy (Continued)

Sampling Point	Parameter	Sample Volume (mL)	Sampling Frequency
Aerobic Digester (Second Batch)	TSS, VSS	100	End of batch'
	OCR	25	
	COD (soluble)	30	
	Metals	500	
	VOCs	40	
	svocs	1000	
	Organosulfur compounds	80	
	TDG	80	
	BOD (soluble)	500	
Aerobic Digester Solids (From Second Batch)	TSS, VSS	5g	End of batch'
	TCLP, metals, VOCs, SVOCs	100g	
	VOCs	5g	
	TDG	10g	
Aerobic Digester Dewatered Solids Filtrate	TSS	100	
	VSS, TDS	9	
	TOC	40	
	COD	30	
	TDG	80	
	VOCs	40	
	svocs	1000	
	Metals	500	
Aerobic Digester Tank Offgas	VOCs	SUMMA@ canisters	Twice

Table 3-3-3. Summary of Sampling Strategy (Continued)

Sampling Point	Parameter	Sample Volume (mL)	Sampling Frequency
Suspended Solids (From Effluent Filtration)	HD	Filter paper and solids	Once

NOTES:

[a] Represents each batch of hydrolysate. In addition, once during the test phase, the hydrolysate was sampled within 24 hours of offgas sampling for organosulfur compounds, SVOCs, VOCs, TDG, and metals.

[b] N and P analysis samples were taken from the feed day tank after nutrient addition.

[c] MLVSS analysis was conducted using the MLSS sample.

[d] Sample was obtained from the excess biomass stream and transferred to the aerobic digester.

[e] OCR tests were conducted 1 hour before the end of Fill (weekly), 1 hour after the beginning of React (weekly), and 1 hour before the end of React (daily).

[f] Except for TSS, TDS, VSS, total COD, and total BOD, analyses were conducted on filtered effluent and data were reported as soluble values. In addition, twice during the test phase, the effluent was analyzed within 24 hours of offgas collection for organosulfur compounds, SVOCs, VOCs, TDG, and metals.

[g] TDS and VSS analyses were conducted using the TSS sample.

[h] See paragraph 3.3.4, step g for details on sampling frequency.

[i] These analyses were conducted at the end of one aerobic digester batch.

a. *Sampling and Analysis of Hydrolysate.* The hydrolysate was analyzed for pH, TDS, TSS, alkalinity, organics, metals, TDG, TOC, COD, BOD, and sulfate. Organic analysis included determination of organosulfur compounds, VOCs, and SVOCs. Metals analysis included all metals listed in the Universal Treatment Standards (UTS) (Federal Register (FR) §268.48, vol. 59, no. 180, 1994) and also trace metals required for biological treatment (calcium, cobalt, copper, iron, magnesium, manganese, molybdenum, nickel, potassium, selenium, sodium, and zinc). Each batch of hydrolysate received was analyzed. Samples for ammonia-nitrogen (NH_3-N) and soluble orthophosphate analysis were taken from the feed day tank after macronutrients had been added. These analyses were performed once a month to ensure that the correct amounts of nutrients were being added. In addition, once during the test phase the hydrolysate was sampled for analyses of organosulfur compounds, SVOCs, VOCs, TDG, and metals within 24 hours of offgas collection.

b. *Sampling and Analysis of Bioreactor Contents.* The pH, temperature, and DO of the bioreactor were monitored using the online probes and the data stored in the online computer. The pH measured by the online probe was verified using an external probe daily. The MLSS concentration was monitored daily during the workweek and the MLVSS concentration was measured once a week. Samples for analysis of MLSS and MLVSS concentrations were taken toward the end of the React period and at approximately the same time of day. The OCR was measured at the end of the React period five times per week. The OCR was measured 1 hour before the end of the Fill period and 1 hour after the start of the React period on a weekly basis. OCR

measurements were also conducted following addition of bioreactor feed to samples of mixed liquor to determine the extent of increases in OCR values.

In addition, microscopic examination of the biomass in the bioreactor was performed periodically to monitor any changes in the microbial population. During the Settle period, visual observations were made on the settling rate of the biomass and the relative clarity and appearance of the decanted effluent. The SSV and the ZSV also were measured daily at first and then three times per week to assess the settling characteristics of the biomass. The SVI was calculated from the SSV data.

c. *Sampling and Analysis of Bioreactor Offgas.* SUMMA@ canisters were used to collect three offgas samples during the test. Samples were collected during the Fill and React period. At the end of one HRT, samples were collected during the Fill period in l-hour intervals, during the beginning of the React period (1 and 2 hours after the start of the React period), and 1 hour before the end of the React period. Offgas samples were analyzed for VOCs, which included determination of hazardous air pollutants.

d. *Sampling and Analysis of Treated Effluent.* The bioreactor effluent was analyzed daily for TOC and COD (soluble). The TSS was measured five times per week initially; the frequency was reduced to two times per week. The effluent also was analyzed for NH,-N, nitrate-nitrogen (NO,-N), nitrite-nitrogen (NO,-N), and soluble orthophosphate three times per week. Weekly analysis included total and soluble BOD, VSS, TDS, sulfate, total COD, and TDG. Alkalinity was measured weekly. Analyses for HD, metals, VOCs,

SVOCs, and organosulfur compounds were conducted a total of eight times during the test. Analyses for all metals listed in the UTS and iron were conducted. Analyses for TSS, TDS, VSS, total COD, and total BOD were conducted directly on the liquid effluent from the SBR. Analyses for all other parameters were conducted on filtered liquid effluent. The collected data represent soluble values. Analysis of the effluent for organosulfur compounds, SVOCs, VOCs, TDG, and metals included three measurements within 24 hours of offgas sampling. For further characterization, the liquid effluent was analyzed for organochlorine pesticides and PCBs, TPH, total phosphorus, total cyanide, and TCDD. These analyses were conducted once, at the end of the test phase.

e. *Sampling and Analysis of Excess Biomass.* The excess biomass was analyzed for TDG, VOCs, SVOCs, organosulfur compounds, TSS, VSS, HD, UTS metals, and iron after the second and third HRT.

f. *Sampling and Analysis of Aerobic Digester.* For the first batch, after the maximum digester volume was achieved, the following analyses on the digester contents were conducted two times a week: COD (soluble), TSS, VSS before excess biomass feed, pH, OCR, and specific oxygen consumption rate (SOCR) before and after excess biomass feed. TSS and VSS analyses of the settled biomass added to the digester were conducted two times per week. After 10 days of operation at the maximum volume, the digester contents were analyzed for TSS, VSS, soluble COD, soluble BOD, HD, metals (including iron), TDG, VOCs, SVOCs, and organosulfur compounds.

For the second batch, after 10 days of aeration in the Aerobic Digester Tank, the digester contents were analyzed for TSS, VSS, soluble COD, soluble BOD, metals (including iron), VOCs, SVOCs, organosulfur compounds, and TDG. The OCR was measured twice during the 1 O-day period.

In the second batch, after 10 days of aeration, the digester contents were dewatered. The dewatered solids were analyzed for TSS, VSS, VOCs, and TDG. In addition, a TCLP extract of the solids was analyzed for metals, VOCs, and SVOCs. The dewatered solids filtrate was analyzed for TSS, VSS, TDS, TOC, COD, TDG, metals, VOCs, and SVOCs.

The filtrate from the dewatering of solids was analyzed for TSS, VSS, TDS, TOC, COD, TDG, metals, VOCs, and SVOCs.

g. *Sampling and Analysis of Aerobic Digester Tank Offgas.* The offgas from the second digester batch was collected in SUMMA@ canisters and analyzed for VOCs. Analysis was conducted twice during the 1 O-day digester test.

h. *Sampling and Analysis of Suspended Solids from Effluent Filtration.* The suspended solids obtained from the filtration of the effluent (along with the filter papers on which the solids are collected) were collected and analyzed for HD.

3.3.5 Subtest No. 3 Results. The analytical data collected for the various streams and units of the biodegradation process (as described in paragraph 3.3.4, step g) are summarized in the following paragraphs in terms of the bioreactor feed, bioreactor operation, bioreactor liquid effluent, excess biomass (before and after digestion),

bioreactor and digester offgas, and suspended solids from effluent filtration. The collected data is presented in appendix C-3. These data were collected over the 87-day duration of the subtest. The overall duration of testing consisted of 27 days of acclimation followed by 60 days of the test phase. During the acclimation phase, the SBR was operated under varying HRTs and the target HRT was determined to be 20 days. In the test phase, the target HRT of 20 days was maintained and the SBR operated for a period of three HRTs (60 days). During the first 44 days, HD/water hydrolysate with the iron floc separated was used as the bioreactor feedstock; the remaining testing period used hydrolysate with the iron floc included. For the various data reported, statistical significance was determined by conducting replicate analyses; average and standard deviation values for these data are presented in appendix C-S (table C-5-l). The list of analytes and the quantitation limits for organics and metals analyses are provided in table C-5-2.

a. Bioreactor Feed Data

Four batches of hydrolysate were used during the entire testing period. The analytical data collected for feed characterization is presented in appendix C-5 (table C-5-3).

(1) *General Characteristics.* Following NaOH addition, the pH of the feedstock was between 10 and 11. After sodium bicarbonate addition, the pH of the feedstock was ___. The feed TDS values ranged from 9.1 to 9.6 g/L. For batches with the metals floc removed, the average TSS was 8 mg/L, and for batches containing the metals floc, the average TSS was 125 mg/L. The feed TSS values ranged from below quantitation limits (BQLs) to 116 mg/L. The alkalinity of the feed (prior to sodium bicarbonate addition) ranged from 90 to 180 mg/L (as calcium carbonate).

(2) *Feed Organic Characteristics.* Organic characterization consisted of a determination of the VOCs, SVOCs, TDG, and organosulfur compounds present in the feed, and determination of organic concentration in terms of TOC, COD, and BOD.

VOCs detected in the feed included 1,2-dichloroethane, chloroethane, chloroform, trichloroethene, and vinyl chloride. The 1,2-dichloroethane was detected at the highest concentration and ranged from 8.8 to 105 mg/L. Except for 1,2-dicloroethane (average concentration of 56.5 mg/L) and chloroform (average concentration of 7.1 mg/L), all other VOCs were detected at concentrations below 1 mg/L. No SVOCs were detected in the feed above their respective detection limits. Two organosulfur compounds (other than TDG) were detected; 1,4-dithiane was detected at an average concentration of 65 mg/L and 1,4-oxathiane was detected at an average concentration of 7.3 mg/L.

Table 3-3-4 summarizes the TDG, TOC, and COD data for the different feed batches. The average TOC and COD values for the four batches were 4,233 and 18,340 mg/L, respectively. The TDG values ranged from 8.1 to 8.4 g/L. The BOD data obtained was highly variable and was inconsistent with the TOC and COD characteristics; therefore, BOD data is not included here.

(3) *Feed Inorganic Characteristics.* Inorganic characterization included determination of metals, inorganic sulfate, nitrogen, and phosphorus concentrations.

Table 3-3-4. Bioreactor Feed TOC, COD, and TDG Data

Feed Batch ID	Days of Use	TOC (mg/L)	COD (mg/L)	TDG (g/L)
P-3-l (without iron floc)	1 to30	4,130	19,260	8.2
P-3-2 (without iron floc)	31 to 44	4,730	19,540	8.4
P-3-3 (with iron floc)	45 to 69	4,410	16,160	8.1
P-3-4 (with iron floc)	70 to 87	3,660	18,400	8.2
Average		4,233	18,340	8.2

Metals detected in the feed included antimony, arsenic, barium, calcium, chromium, copper, iron, lead, magnesium, manganese, molybdenum, nickel, selenium, sodium, thallium, and zinc. Sodium was detected at an average concentration of 3735 mg/L and was the metal with the highest concentration. The average concentration of iron in the feedstock batches with the iron floc removed was 1.3 mg/L, compared to 34.5 mg/L in batches with the iron floc included. Other metals, such as chromium, nickel, and zinc, were also present at higher concentrations in batches with the iron floc included, compared to batches with the iron floc removed.

The average sulfate concentration in the feed was 28.6 mg/L. The nitrogen concentration (measured as ammonia-nitrogen) and the phosphorus concentration (measured as orthophosphate) measured in the feed following nutrient addition were 297.4 and 77.4 mg/L (average).

b. *Bioreactor Operating Data*

Reactor operating data collected included the feed and sludge wasting volumes and measured values of MLSS, MLVSS, SVI, ZSV, and OCR.

The bioreactor operating data is shown in table C-5-4. During the acclimation phase, the SBR was operated under varying HRTs ranging from 14 to 20 days. The daily feed volume was calculated as the ratio of the reactor volume to the desired HRT. Periods of no feed were also used during the acclimation phase. The test phase was initiated from the 28th day of operation using a 20-day HRT. The variation of HRT as a function of days of operation is shown in figure 3-3-l.

The daily wasting volume (that is, the volume of mixed liquor removed from the SBR prior to the end of the React period) was calculated as the ratio of reactor volume to the desired SRT. The volume of mixed liquor removed ranged from 0.5 to 2L per day.

The initial MLSS concentration obtained in the reactor on the first day of operation was 4570 mg/L. A graph of the MLSS concentrations during the overall testing period is shown in figure 3-3-2. The trend of the average MLSS concentrations (5-day rolling average) is also shown. In general, four trends were distinguished. First, during the initial 20 days of the test, the average MLSS concentration decreased from the initial value of 4570 mg/L to approximately 3500 to 3700 mg/L. Second, the MLSS concentration was fairly stable in this range between days 21 and 51 of the test. Third, a decreasing trend was observed between days 51 and 58, during which time the MLSS decreased to approximately 3200 mg/L. Fourth, the MLSS was fairly stable (in the range of 3100 to 3300 mg/L) for the remaining duration of the testing period. The MLVSS concentration trend is similar to the MLSS trend. At the end of the test, the MLVSS to MLSS ratio had increased from 0.61(+/-0.02) to 0.73(+/-0.02). The average MLSS and MLVSS concentrations during the test phase were 3350(+/-281) and 2290(+/-90) mg/L, respectively.

The SVI of the biomass varied from approximately 70 to 110 mL/g over the first 64 days of the test. The SVI values ranged from 125 to 286 mL/g during the remaining portion of the test and showed an increasing trend. The ZSV values ranged from 0.18 to 4.3 cm/minute. Variation of ZSV was inversely proportional to the SVI values.

The OCR data collected consisted of the OCR values measured at the end of the React period (hereafter referred to as end-of-React OCR), at 1 hour before the end of the Fill period, 1 hour after the start of the React period, and after 24 hours of additional aeration of a mixed liquor sample. In addition to direct measurement of OCR, samples were also spiked with the bioreactor feed and the OCR was measured again. The OCR data is presented in appendix C-5 (see table C-5-5). Figure 3-3-3 shows the end-of-React OCR values and those measured during the Fill period.

During the acclimation phase, the end-of-React OCR values were highly variable. During the test phase, the OCR values were stable in the range of 25 to 35 mg/L/h. For a brief period, between days 52 and 65, the OCR values decreased; however, by the end of the test phase, the OCR values increased to the original range of 25 to 35 mg/L/h. The OCR values measured after spiking with the bioreactor feed indicated an increase of 20 to 40 mg/L/h over the baseline rate (that is, the OCR value measured before spiking). The OCR values measured during the Fill period and during the start of the React period were obtained to determine the maximum OCR of the bioreactor. The maximum OCR value obtained was 134 mg/L/h; the corresponding SOCR value was 43 mg/g/h. The average OCR value measured was approximately 82 mg/L/h during the Fill period and was approximately 84 mg/L/h during the start of the React period. The corresponding SOCR values were 22.6 mg/g/h and 23.1 mg/g/h.

c. *Bioreactor Liquid Effluent Data*

Bioreactor liquid effluent data is presented in appendix C-5 (table C-5-6).

(1) *General Characteristics.* The effluent pH varied between 6.8 and 7.6. The effluent TDS values gradually increased during the first 50 days of the test and leveled off at a concentration of approximately 20.8 g/L. The variation of effluent TSS values is shown in figure 3-3-4.

Effluent TSS values were generally below 100 mg/L. However, for a period of approximately 20 days, the effluent TSS profile indicated a temporary increase with a maximum TSS of 370 mg/L. The average effluent VSS concentration was approximately 73 percent of the effluent TSS value. The alkalinity of the effluent ranged from 70 to 224 mg/L (as calcium carbonate), except for a single measurement of 644 mg/L (as calcium carbonate).

(2) *Liquid Effluent Organic Characteristics.* Organic characterization consisted of a determination of the VOCs, SVOCs, HD, TDG, and organosulfur compounds present in the liquid effluent, and determination of organic concentration in terms of TOC, COD, and BOD.

Neither VOCs nor SVOCs were detected in the effluent above their respective detection limits. HD was not detected in the liquid effluent at concentrations below 0.2mg/L. The two organosulfur compounds (other than TDG) that were detected in the feed (1,4-dithiane and oxathiane) were also detected in the effluent. The 1,4-dithiane was detected at an average concentration of

3.3 mg/L and 1,4-oxathiane was detected at an average concentration of 1.3 mg/L.

The effluent TDG concentration was less than 5 mg/L and, in a majority of the samples, was below the TDG detection limit of 2 mg/L. For the test phase, the average TDG concentration was below the detection limit. The trends of effluent TOC and COD values are shown in figure 3-3-5. During the acclimation phase, the effluent COD values temporarily increased. Following operation at the 20-day HRT, the effluent COD values were stable in the range of 1300 to 1550 mg/L except for the period between days 52 and 60 when the effluent COD temporarily increased to a maximum of 2085 mg/L. The average COD of the effluent during the test phase was 1557(+/-226) mg/L. The average total COD (measured using unfiltered effluent) was 1734 mg/L. No major differences from the COD trend were noted for the effluent TOC profile. The average TOC of the effluent during the test phase was 444(+/-68) mg/L. Limited effluent BOD data were collected.

Neither pesticides nor PCBs were detected in the liquid effluent above their respective quantitation limits. The TPH concentration was measured to be 0.533 mg/L. *Dioxin data*

(3) *Effluent Inorganic Characteristics.* Inorganic characterization included determination of metals, inorganic sulfate, nitrogen, and phosphorus concentrations.

Metals detected in the effluent included antimony, arsenic, barium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, molybdenum, nickel, selenium, silver,

sodium, vanadium, and zinc. Sodium was detected at a maximum concentration of 9750 mg/L and was the metal with the highest concentration.

The sulfate concentration in the liquid effluent gradually increased at the start of testing and leveled off at approximately 7000 mg/L after 50 to 60 days. The nitrogen concentrations (measured as ammonia-nitrogen, nitrate-nitrogen, and nitrite-nitrogen) and the phosphorus concentration (measured as orthophosphate) of the liquid effluent were variable and were related to the amounts of nitrogen and phosphorus added to the bioreactor feed. The maximum concentrations of ammonia-nitrogen, nitrate-nitrogen, nitrite-nitrogen, and orthophosphate concentrations measured in the effluent were 108, 120, 40, and 68.6 mg/L, respectively. However, effluent nitrate-nitrogen and nitrite-nitrogen values were generally less than 5 mg/L during the test phase.

Total cyanide concentration of the liquid effluent was 0.008 mg/L and total phosphorus concentration was 2.32 mg/L. Since the reported cyanide concentration is only slightly above the method quantitation limit, this result may be an artifact.

d. *Excess Biomass Data*

Excess biomass data is presented in appendix C-5 (table C-5-7).

(1) *Excess Biomass General Characteristics.* The TSS and VSS of excess biomass were measured to be 11.6 and 7.9 g/L, respectively.

(2) *Excess Biomass Organic and inorganic Characteristics.* The 1,2-dichloroethane was the only VOC-detected (0.025 mg/L) in the excess biomass stream. No SVOCs were measured above the quantitation limits. The 1,4-dithiane was detected twice at an average concentration of 2.34 mg/L. The 1,4-oxathiane was detected once at a concentration below 1 mg/L. TDG was not detected above its quantitation limit. HD was also not detected below the 0.2 mg/L level. Metals detected in excess biomass were antimony, arsenic, barium, cadmium, chromium, iron, lead, mercury, nickel, silver, sodium, vanadium, and zinc. Sodium was the metal with the highest concentration detected (6520 mg/L). Iron was detected in one sample of excess biomass at a concentration of 652 mg/L.

e. *Aerobic Digester Data*

Aerobic digester data is presented in appendix C-5 (table C-5-7).

(1) *Aerobic Digester (First Batch).* The average TSS concentration of the aerobic digester contents was 25.9 g/L. The VSS concentration measured 30 days after digester operation began was 11.7 g/L and is equivalent to 45 percent of the TSS. The OCR of the aerobic digester was 31.8 mg/L/h on the day the maximum value was reached. After 9 days of aerobic digester operation, the OCR was 23.6 mg/L/h. The corresponding SOCR values were 1.23 and 0.91 mg/g/h. The soluble COD fraction of the aerobic digester contents was 3773 mg/L.

The aerobic digester contents from the first batch were characterized for organics and metals. No VOCs were detected

above the quantitation limits. No SVOCs were measured above the quantitation limits. The 1,4-dithiane was detected at a concentration of 0.776 mg/L and 1,4-oxathiane was detected at a concentration of 0.172 mg/L. TDG was not detected above its quantitation limit, Sodium and iron were the principal metals. *HD analytical results to be added.*

(2) *Aerobic Digester (Second Batch).* The TSS concentration of the aerobic digester contents was measured to be 10.3 g/L at the end of the batch. The corresponding VSS concentration was 7.2 g/L and was equivalent to 70 percent of the TSS concentration. The OCR of the aerobic digester was 52.8 mg/L/h at the beginning, 34.0 mg/L/h after 2 days of operation, and 19.1 mg/L/h at the end of the batch. The corresponding SOCR value at the end of the batch was 1.86 mg/g/h. The soluble COD fraction of the aerobic digester contents was 1210 mg/L.

The aerobic digester contents from the second batch were characterized for organics and metals. No VOCs were detected above the quantitation limits. No SVOCs were measured above the quantitation limits. TDG was not detected above its quantitation limit. *HD analytical results to be added.*

(3) *Aerobic Digester Dewatered So/ids (Second Batch).* The TSS and VSS concentrations of the dewatered solids were measured to be 152 and 99 g/L.

The aerobic digester dewatered solids from the second batch were characterized for organics and metals. The only VOC detected in the solids was methylene chloride at a concentration of 0.3 mg/L.

The presence of methylene chloride may have been the result of laboratory contamination. A TCLP extract of the solids was analyzed for VOCs, SVOCs, and UTS metals. TCLP VOCs and SVOCs were BQL. The TCLP metals detected were barium and silver. TDG was detected at a concentration of 87.8 mg/kg.

(4) *Aerobic Digester Dewatered Solids Filtrate.* The TSS, VSS, and TDS concentrations of the filtrate were measured to be 37.5 mg/L, 13 mg/L, and 23.2 g/L.

The filtrate was also characterized for organics and metals. TOC and COD values were measured to be 252 and 1014 mg/L. The filtrate contained TDG at a concentration of 2.7 mg/L. Neither VOCs nor SVOCs were detected above the quantitation limits. Metals detected included barium, cadmium, calcium, cobalt, copper, iron, magnesium, manganese, sodium, and zinc. Sodium and iron were the metals detected with the highest concentrations at 8650 and 111 mg/L, respectively.

f. *Bioreactor and Digester Offgas Data*

The VOCs detected in the bioreactor offgas were 1,2-dichloroethane, chloroform, methylene chloride, 4-methyl-2-pentanone, and 1,2,4-trichlorobenzene. Analytical data on the bioreactor offgas is presented in table C-5-8. Consistent with the feed characteristics, 1,2-dichloroethane was the VOC with the highest concentration detected in the offgas. The average concentration of 1,2-dichloroethane measured during the Fill period was greater than 32.9 µg/L. (NOTE: Due to sample dilution constraints, the reported values for VOCs are estimates only and the actual values may be greater.) These data are average values for the

duration of offgas collection. Average offgas concentrations were measured during the Fill and React periods. As expected, the average concentrations in the offgas collected during the Fill period were greater than those measured during the React period. The 4-methyl-2-pentanone and 1,2,4-trichlorobenzene were not detected in the feed and their source is questionable. These compounds were detected at very low concentrations and in only one or two of several samples. These two VOCs are likely laboratory contaminants.

Chloroform was the only VOC detected in the aerobic digester offgas (table C-5-8).

24-hour analysis of feed and effluent streams

g. *Data on Suspended Solids from Effluent Filtration*

Data to be added

3.3.6 Analysis of Subtest No. 3 Results

Feed Characteristics. The bioreactor feedstock consisted of TDG as the primary organic compound with smaller amounts of 1,4-dithiane, 1,4-oxathiane, and chlorinated VOCs. The average TOC and COD of the feedstock were 4,233 and 18,340 mg/L, respectively. During the test phase, the average TOC and COD of the feedstock were 4,152 and 17,257 mg/L, respectively. Based on a theoretical oxygen demand of 1.84 grams per gram of TDG, the fraction of TDG contribution of the total feed COD was approximately 83.5 percent. The corresponding TOC contribution was approximately 80 percent. Sodium and iron were the principal metals in the feedstock.

Bioreactor Performance. The overall testing approach consisted of SBR operation using an acclimation phase followed by a test phase. During the acclimation phase, the SBR was operated by increasing the loading (that is, by decreasing the HRT) with the goal of reaching a 10-day HRT. Initially, the SBR was operated at a 20-day HRT. During this period, the effluent COD rapidly increased to approximately 2400 mg/L, indicating buildup of organics in the reactor. To allow for biodegradation of residual organics, the SBR was not fed on day 6. Following continued operation at a 20-day HRT, the SBR performance improved with lower effluent COD values. The HRT was decreased to 16 days and then to 14 days over the next 6 days. Although the effluent COD values increased, the rate of COD increase was not as rapid as observed for the first 4 days. However, continued operation at the 14-day HRT resulted in a rapid buildup of organics with effluent COD reaching 3268 mg/L. Again, periods of no feed were used to allow for biodegradation of residual organics. In all cases, the strategy of not feeding the reactor allowed the system to consume residual organics and resulted in decreased effluent COD.

During acclimation, reactor performance was also monitored by analyzing the DO profile for each cycle. By day 7, a DO profile representative of normal SBR operation developed. A typical DO profile observed in the SBR is shown in figure 3-3-6. This DO profile consisted of three trends: first, a deceasing trend during the Fill period and part of the React period indicating rapid DO consumption; second, a constant trend in which the DO is at a minimum indicating a period of high rate of oxygen consumption; and third, a constant trend in which the DO increases and plateaus to a maximum (referred to as the endogenous phase). During the endogenous phase, biodegradation reactions are assumed to be essentially complete and the oxygen consumption rate (which is assumed to correspond with the endogenous respiration rate) is at a minimum during the cycle. (NOTE: In practice, a true endogenous OCR is determined following an additional 24 hours of aeration of a mixed liquor sample,)

During the acclimation phase, DO profiles with decreasing endogenous periods were obtained as a result of decreasing the HRT. In addition, during days when the effluent COD values were high, the DO profile did not appear to indicate periods of oxygen consumption; instead, the DO was generally constant or decreased at a much lower rate than normally observed. Following operation of the SBR without feed resulted in recovery of the normal DO profile. These observations were corroborated with end-of-React OCR measurements. The end-of-React OCR and SOCR values increased during periods of buildup of COD (see figure 3-3-5). Increasing end-of-React SOCR values are indicative of incomplete biodegradation reactions within each cycle. During the test phase, constant or decreasing SOCR values along with normal DO profiles, which are both representative of satisfactory operation, were obtained.

The average HRT used in the acclimation phase was 25 days. Since decreasing HRTs resulted in buildup of organics and reduced ORE, it appeared that a lengthy period of acclimation may be required for operation at a 10-day HRT. Therefore, it was decided to operate the SBR at a lower organic loading than the IO-day HRT. Based on the data obtained, it appeared that stable bioreactor performance could be obtained at a 20-day HRT. Operation of the SBR was changed to a 20-day HRT. As a result, the effluent COD decreased to the range of 1300 to 1500 mg/L. The end-of-React OCR values also stabilized suggesting that the biodegradation reactions were complete within the 17.5hour React period and residual organics were not accumulating in the SBR.

The SBR was operated from day 28 to day 87 at a 20-day HRT (except for day 35 when the SBR was not fed as a precautionary measure to prevent overloading of organics). During this test phase, effluent COD values continued to be stable. Both the DO profile and the end-of-React OCR data indicated acceptable reactor performance with at least 8 hours of excess capacity.

During the acclimation phase, the MLSS concentration decreased by approximately 800 to 1000 mg/L. This decrease in MLSS concentration is likely due to two reasons.

First, the digestion of organic solids present in the seed sludge can result in MLSS reduction. Second, death and lysis of microorganisms incapable of using organic compounds present in the hydrolysate as a carbon and energy source can also reduce the MLSS concentration. Following the initial decrease, the MLSS concentration was fairly constant for approximately 30 days. During this period, the average wasting rate was approximately 0.7L of mixed liquor. This means that the increase in MLSS concentration due to growth approximately equaled the suspended solids wasted from the SBR. The temporary decrease in MLSS concentration for a brief period after the temperature increase in the reactor can be partly explained by the lysis of microorganisms exposed to temperatures above their optimum range for growth. Decreases in MLSS concentrations were accompanied by increased effluent TSS values, which are also indicative of poor settling biomass and cellular debris. During the period of high TSS values, a temporary increase in cloudiness of the liquid effluent was also visually noted. However, bioreactor conditions returned to normal with the MLSS concentration being stable for the last 20 days of the test and the effluent TSS values decreasing to less than 70 mg/L.

The average wasting rate used during the test phase was approximately 1 L per day. The corresponding SRT was calculated to be 56 days (see table C-5-9). This SRT is greater than SRTs typical of biological treatment plants where SRTs of 20 to 30 days are common. At the SRT used, the MLSS was fairly stable.

Liquid Effluent Characteristics. ORE results are presented in appendix C-5 (table C-5-4) and are graphically shown in figure 3-3-7. ORE values were calculated as the efficiency of COD removal. During the period when residual organics were accumulating, the ORE values showed a continuously decreasing trend; the ORE decreased from greater than 90 percent to 81 percent.

For a brief period prior to and after a temporary increase in the reactor temperature [to 122°F (50°C)], the ORE decreased to 85 percent; however, within 1 week, the ORE

had increased to the normal range of 89 to 91 percent. The temporary decrease in ORE corresponded with increased cloudiness and high effluent TSS values. It was determined that the increased effluent COD was due to the COD associated with suspended solids in the liquid effluent. The average COD-based ORE obtained during the test phase was 91 percent. The COD-based ORE values were stable and ranged from 85 to 93 percent during the 60-day period at the 20-day HRT.

Effluent TDG values (see table C-5-6) were less than 5 mg/L throughout the testing period. The TDG removal efficiency averaged greater than 99.9 percent. Since TDG represents only 82 percent of the organics present in the bioreactor feed (on a COD basis) and the average ORE is 91 percent, it appears that non-TDG organics are also degraded by approximately 50 percent. Both 1,4-dithiane and 1,4-oxathiane appeared to be biodegraded to a significant extent (greater than 74 percent). The buildup of organics observed during the acclimation phase is likely due to a slower rate of or no biodegradation of non-TDG organics. It should be noted that TDG removal efficiency was high even during periods of high effluent COD. Inorganic sulfate concentrations measured in the effluent are shown in figure 3-3-8. Inorganic sulfate concentrations gradually increased during the course of testing and leveled off at approximately 7000 mg/L. Assuming stoichiometric conversion of the sulfur in TDG to inorganic sulfate as a result of biodegradation, the sulfate concentration in the effluent would be 6482 mg/L (based on an average feed TDG concentration of 8.25 g/L). Since the actual sulfate concentration measured in the effluent is very comparable (94 percent) to the theoretical estimate, biodegradation can be concluded as the removal mechanism for TDG.

By the end of the testing period, the MLVSS to MLSS ratio had increased to 0.73 from the original value of 0.61 for the biomass seed. This increase in the MLVSS fraction represents an increase in the viable fraction of the biomass as a result of acclimation and the SRT used in the test. The increase in the MLVSS fraction along with the ability

to sustain the MLSS concentration is representative of a successful biological treatment process in which the influent organics support biological growth.

In general, the settling characteristics of the biomass developed during this subtest were acceptable. The SVI of the biomass was less than 110 mL/g for most of the testing period. A range of 100 to 200 mL/g is considered acceptable and an SVI of less than 100 mL/g represents good settling characteristics. Although the SVI data showed an increasing trend toward the end of the test, the biomass settled adequately within the l-hour Settle period and the effluent TSS values were low. No bulking (extremely poor settling) biomass was observed during the course of the test.

Microscopic examination of the mixed liquor was conducted periodically during the acclimation phase and the test phase to monitor the types of microorganisms present. Initially, the mixed liquor consisted primarily of bacteria with few flagellated protozoa present. Over the course of testing, the mixed liquor population became more diverse. Bacteria were still the predominant microorganisms; however, there appeared to be more protozoa and rotifers present. The presence of a diverse group of microorganisms, including bacteria, protozoa, and rotifers is an indicator of good sludge quality. On day 72, a type of water mite was observed in the bioreactor. Upon further microscopic examination, there appeared to be a large number of water mites present in the mixed liquor. Mites were likely present at low concentrations in the original biomass sample obtained from the wastewater treatment plant. Similar mites have been observed by plant operators at the Back River Wastewater Treatment Plant. The presence of mites did not seem to be detrimental to reactor operation. Both the MLSS concentration and the ORE appeared to be unaffected by the presence of mites.

Excess Biomass and *Aerobic Digestion.* The suspended solids concentration of the settled excess biomass obtained (12 g/L) is typical of that obtained after gravity settling of biomass. During operation of the first batch of the aerobic digester, considerable water loss occurred due to evaporation. As a result, the organics and solids were

concentrated and the measured TSS in the first batch was higher than the excess biomass concentration. Since the second batch was operated for 10 days only, no major loss of water occurred. Therefore, the TSS concentration measured in the second batch is more typical of normal operation. For the same reason, the soluble COD measured in the first batch is higher than that measured in the second batch. The extent of TSS and VSS reduction was approximately 10 percent following 10 days of aerobic digestion.

Nutrient and *Buffer* Requirements. Nitrogen and phosphorus concentrations in the feed were continually adjusted so that the effluent ammonia-nitrogen and orthophosphate concentrations were less than 5 mg/L. However, effluent nitrogen and phosphorus were not controlled at low concentrations but were added in excess of microbial requirements. Therefore, nitrogen and phosphorus requirements were less than the planned amounts of 1440 mg/L of ammonium chloride and 277.5 mg/L of potassium dihydrogen phosphate; however, the exact requirements for long-term operation were not determined. An excess of nitrogen and phosphorus in the liquid effluent did not affect bioreactor performance. No significant nitrification (conversion of ammonia to nitrate and nitrite) was observed except during the first 3 weeks of operation.

Sodium bicarbonate (12 g/L) was used as the buffer and was found to sufficiently maintain the pH within a range of 6.8 to 7.6. Typically, the pH of the bioreactor contents decreased during the Fill and initial parts of the React period. During this period, additional NaOH was required to maintain the pH above 6.8. The pH increased during the latter part of the React period from 6.8 to 7.4. The pH of the liquid effluent was typically in the range of 7.2 to 7.6.

The results obtained from subtest no. 3 are summarized in table 3-3-5. The following biodegradation data were determined for the subtest.

Table 3-3-5. Summary of Subtest No. 3 Results

Parameter	Average Value
Effluent TOC	444(+/-68)[a] mg/L
Effluent COD	1557(+/-226) mg/L
MLSS	3350(+/-28 1)mg/L
Effluent TSS	92(+/-100) mg/L
ORE (COD-based)	91 (+/-I .8) percent
ORE (TOC-based)	89(+/-1.7) percent
TDG Removal Efficiency	99.9 percent
Biomass Yield	0.29g (+/-0.005) of MLSS per gram of TOC
HRT	20 days
SRT	56 days
OCR Measured During Fill	82(+/-27) mg/L/h
End-of-React OCR	26(+/-7) mg/L/h
SVI	132(+/-55) mL/g

NOTE:

[a] Values in parentheses indicate standard deviations calculated for the test phase.

a. *Bioreactor Operating Data*. These data consist of pH, DO, temperature, effluent TOC and COD, MLSS, and effluent TSS. A typical profile of the DO and pH is shown in figure 3-3-6. The temperature of the reactor was held constant at 77°F (25°C) except for a brief increase to 131 °F (55°C). The average effluent TOC was 444(+/-68) mg/L and the average effluent COD was 1557(+/-226) mg/L. Profiles of TOC and COD are presented in figure 3-3-5. The average MLSS concentration during the test phase was 3350(+/-281) mg/L and the average effluent TSS concentration was

92(+/-100) mg/L. Profiles of MLSS and TSS are shown in figures 3-3-2 and 3-3-4.

b. ORE. A graph of COD-based ORE as a function of time over the course of the test is shown in figure 3-3-7. During the test phase, the average ORE values based on removal of TDG, TOC, and COD were 99.9, 89, and 91 percent, respectively. The ORE based on BOD data was not determined.

c. Organic *Loading.* The average organic loading during the test phase was calculated to be 0.06(+/-0.01)g of TOC per gram of MLSS per day and 0.09(+/- 0.01)g of TOC per gram of MLVSS per day (see table C-5-10 for calculations). In terms of COD, the loading values are 0.26(+/-0.01)g of COD per gram of MLSS per day and 0.39(+/-0.01)g of COD per gram of MLVSS per day.

d. *Biomass Yield.* The average biomass yield was calculated to be 0.29(+/-0.005)g of MLSS produced per gram of TOC consumed. The yield calculation is based on the average values of MLSS, TOC, and wasting volumes, and is shown in appendix C-5 (table C-5-9). On a COD basis, the average biomass yield was 0.07g of MLSS produced per gram of COD consumed. Yield values obtained were lower than typical (0.6 to 0.7 g of MLSS per gram of TOC). For well-acclimated systems with lower SRTs, higher yields are generally obtained.

e. *HRT.* The HRT used during the test phase was 20 days and the average HRT of the acclimation phase was 25 days.

f. *SRT.* The average SRT during the test phase was 56 days.

g. OCR. The maximum OCR value measured was 134 mg/L/h and the corresponding SOCR value was 43 mg/g/h. The average OCR value measured during the end of the Fill period and the start of the React period was approximately 72 mg/L/h; the corresponding SOCR values were 19.6 and 20.1 mg/g/h. The average end-of-React OCR value was 26 mg/L/h and the SOCR was 7.7 mg/g/h.

h. *Settling Characteristics.* The average SVI during the test phase was 132(+/-55) mL/g. In general, the average SVI is indicative of well-settling biomass. The average ZSV was measured to be 2.5 cm/minute.

i. *Chemical Characteristics of Feed and Effluent Streams.* The chemical characteristics of the feed and effluent streams were determined in terms of the concentrations of organics (VOCs, SVOCs, and organosulfur compounds), TDG, and metals, and the data is presented in appendix C-5 (tables C-5-2 and C-5-5).

Comparison of Results With Test Criteria. The minimum quantitative data for ORE (in terms of TDG, TOC, and COD reduction), MLSS, effluent TSS, OCR, and chemical characteristics of effluent streams were fully obtained. In addition, temperature, pressure, sampling time, reaction time, and mixing conditions were recorded. Chemical characteristics of the liquid effluent and excess biomass streams were determined in terms of VOCs, SVOCs, organosulfur compounds, and metals. The results obtained from this subtest successfully meet the pass/fail test criterion.

The biodegradation data generated from this subtest were also evaluated based on the test criteria established in paragraph 3.3.3.

a. The TDG removal efficiency obtained in this subtest meets the treaty requirements of greater than 99 percent removal efficiency. TDG removal

of 99.9 percent was accomplished in the bioreactor and the TDG concentration in the liquid effluent was below the quantitation limit of 2 mg/L. Therefore, posttreatment of the bioreactor effluent for additional TDG removal would not be required.

b. The average biodegradation OREs based on COD and TOC were 91 and 89 percent, respectively. These values compare very well with the value obtained in the feasibility tests (88 percent TOC-based ORE).

c. The COD-based OREs obtained during the test phase ranged from 85 to 93 percent. For a majority of the test phase, the ORE data was very stable in the range of 88 to 90 percent and did not show more than a 2 percent variation. Moreover, ORE data did not exhibit a continuously decreasing trend during the test phase. For a brief period, the ORE decreased to 85 percent; however, the ORE increased to 90 percent within a week. Stable ORE data (that is, less than 5 percent deviation from the average) were obtained for the entire duration of three HRTs (which was greater than the period of one SRT used in this subtest). These data are evidence of a stable biodegradation process.

d. During the test phase, the MLSS concentration (5-day average) in the bioreactor ranged from 3500 to 3700 mg/L. During the 87-day testing period, the MLSS was maintained above the desired minimum MLSS of 2500 mg/L while biomass was continuously wasted. No major decreases in MLSS concentrations or continuously decreasing MLSS trends were observed. For the conditions tested, it can be concluded that the process is viable in terms of supporting biomass growth and maintaining biological treatment.

3.4 Subtest No. 4

DRAFT

SECTION 4
SUMMARY OF TEST RESULTS

SECTION 5
CONCLUSIONS

APPENDIX A
ACRONYMS/ABBREVIATIONS

APPENDIX A
ACRONYMS/ABBREVIAITONS

ADE	agent destruction efficiency
AMSAA	Army Materiel Systems Analysis Activity
APHA	American Public Health Association
AR	Army Regulation
ATP	Alternative Technology Program
AWWA	American Water Works Association
BOD	biochemical oxygen demand
BQL	below quantitation limit
COD	chemical oxygen demand
CSDP	Chemical Stockpile Disposal Program
c w c	Chemical Weapons Convention
DAB	Defense Acquisition Board
DO	dissolved oxygen
EPA	Environmental Protection Agency
ERDEC	Edgewood Research, Development, and Engineering Center
FR	Federal Register
HD	mustard agent
HRT	hydraulic retention time
IPR	in-process review

MLSS	mixed liquor suspended solid
MLVSS	mixed liquor volatile suspended solid
NRC	National Research Council
OCR	oxygen consumption rate
ORE	organic removal efficiency
PCB	polychlorinated biphenyls
PMCD	Program Manager for Chemical Demilitarization
R&D	research and development
rpm	revolutions per minute
RTD	resistance temperature device
SBR	sequencing batch reactor
SOCR	specific oxygen consumption rate
SOP	Standing Operating Procedure
SRT	solids retention time
ssv	settled sludge volume
SVI	sludge volume index
svoc	semivolatile organic compound
TCDD	2,3,7,8-tetrachlorodibenzo-p-dioxin
TCLP	Toxicity Characteristic Leaching Procedure
TDG	thiodiglycol
TDS	total dissolved solids
TEMP	Test and Evaluation Master Plan
TOC	total organic carbon
TPH	total petroleum hydrocarbons

TSS	total suspended solids
USATHAMA	U.S. Army Toxic Hazardous Materials Agency
UTS	Universal Treatment Standards
VOC	volatile organic compound
v s s	volatile suspended solid
v x	nerve agent
WEF	Water Environment Federation
ZSV	zone settling velocity

APPENDIX B
DRAWINGS

APPENDIX B
DRAWINGS

(NOT APPLICABLE)

16407

APPENDIX C

DATA

APPENDIX C
DATA

APPENDIX C-1

(TO BE SUPPLIED)

APPENDIX C-2
(TO BE SUPPLIED)

APPENDIX C-3
ANALYTICAL DATA
(TO BE SUPPLIED)

APPENDIX C-4
(TO BE SUPPLIED)

APPENDIX C-5
SUBTEST NO. 3 DATA

TABLE C-5-2. DETECTION LIMITS OF ANALYTICAL METHODS

ANALYSIS	METHOD NUMBER	DETECTION LIMITS	TCLP DETECTION LIMITS
TDC, mg/L	AEC UW-22	2.00	
VOC's, µg/L	SW-846 # 8260		
1,1,1-Trichloroethane		5.0	
1,1,2,2-Tetrachloroethane		5.0	
1,1,2-Trichloroethane		5.0	
1,1-Dichloroethane		5.0	
1,1-Dichloroethene		5.0	100
1,2-Dichloroethane		5.0	100
1,2-Dichloropropane		10.0	
2-Butanone		10.0	100
2-Chloroethylvinyl ether		10.0	
2-Hexanone		10.0	
4-Methyl-2-pentanone		10.0	
Acetone		10.0	
Benzene		5.0	100
Bromodichloromethane		5.0	
Bromoform		5.0	
Bromomethane		10.0	
Carbon disulfide		5.0	
Carbon tetrachloride		5.0	100
Chlorobenzene		5.0	100
Chloroethane		10.0	
Chloroform		5.0	100
Chloromethane		10.0	
cis-1,3-Dichloropropene		5.0	
Dibromochloromethane		5.0	
Ethylbenzene		5.0	
Methylene chloride		5.0	
Styrene		5.0	
Tetrachloroethene		5.0	100
Toluene		5.0	
trans-1,2-Dichloroethene		5.0	
trans-1,3-Dichloropropene		5.0	
Trichloroethene		5.0	100
Vinyl acetate		10.0	
Vinyl chloride		10.0	100
Total xylenes		5.0	
1,4-Dichlorobenzene			100
VOC's, µg/L (offgas)	TO-14		
1,1,1-Trichloroethane		2.0	
1,1,2,2-Tetrachloroethane		2.0	
1,1,2-Trichloroethane		2.0	
1,1-Dichloroethane		2.0	
1,1-Dichloroethene		2.0	
1,2-Dichloroethane		2.0	
1,2-Dichloropropane		2.0	
2-Butanone		2.0	

TABLE C-5-2. DETECTION LIMITS OF ANALYTICAL METHODS

ANALYSIS	METHOD NUMBER	DETECTION LIMITS	TCLP DETECTION LIMITS
VOC's, µg/L (offgas), continued			
2-Chloroethylvinyl ether		2.0	
2-Hexanone		2.0	
4-Methyl-2-pentanone		2.0	
Acetone		2.0	
Benzene		2.0	
Bromodichloromethane		2.0	
Bromoform		2.0	
Bromomethane		2.0	
Carbon disulfide		2.0	
Carbon tetrachloride		2.0	
Chlorobenzene		2.0	
Chloroethane		2.0	
Chloroform		2.0	
Chloromethane		2.0	
cis-1,3-Dichloropropene		2.0	
Dibromochloromethane		2.0	
Ethylbenzene		2.0	
Methylene chloride		2.0	
Styrene		2.0	
Tetrachloroethene		2.0	
Toluene		2.0	
trans-1,2-Dichloroethene		2.0	
trans-1,3-Dichloropropene		2.0	
Trichloroethene		2.0	
Vinyl acetate		2.0	
Vinyl chloride		2.0	
Total xylenes		2.0	
1,2-Dichloroethene (total)		2.0	
1,2,4-Trichlorobenzene		2.0	
Dichlorodifluoromethane		2.0	
Freon 114		2.0	
cis-1,2-Dichloroethene		2.0	
1,2-Dibromoethane		2.0	
m,p-Xylene		2.0	
o-Xylene		2.0	
1,3-Dichlorobenzene		2.0	
1,4-Dichlorobenzene		2.0	
1,2-Dichlorobenzene		2.0	
1,2-Dibromo-3-chloropropane		2.0	
SVOC's, µg/L	SW-846 # 8270		
1,2,4-Trichlorobenzene		20.0	
1,2-Dichlorobenzene		20.0	
1,3-Dichlorobenzene		20.0	
1,4-Dichlorobenzene		20.0	50
2,4,5-Trichlorophenol		100.0	50
2,4,6-Trichlorophenol		20.0	50
2,4-Dichlorophenol		20.0	

TABLE C-5-2 DETECTION LIMITS OF ANALYTICAL METHODS

ANALYSIS	METHOD NUMBER	DETECTION LIMITS	TCLP DETECTION LIMITS
SVOC's, µg/L, continued			
2,4-Dimethylphenol		20.0	
2,4-Dinitrophenol		100.0	
2,4-Dinitrotoluene		20.0	50
2-Chloronaphthalene		20.0	
2-Chlorophenol		20.0	
2-Methylnaphthalene		20.0	
2-Methylphenol		20.0	50
2-Nitroaniline		100.0	
2-Nitrophenol		20.0	
3,3'-Dichlorobenzidine		40.0	
3-Nitroaniline		100.0	
4,6-Dinitro-2-methylphenol		100.0	
4-Bromophenyl-phenylether		20.0	
4-Chloro-3-methylphenol		20.0	
4-Chloroaniline		20.0	
4-Chlorophenyl phenyl ether		20.0	
4-Methylphenol		20.0	50
4-Nitroaniline		100.0	
4-Nitrophenol		100.0	
Acenaphthene		20.0	
Acenaphthylene		20.0	
Anthracene		20.0	
Benzoic acid		100.0	
Benzo[a]anthracene		20.0	
Benzo[a]pyrene		20.0	
Benzo[b]fluoranthene		20.0	
Benzo[g,h,i]perylene		20.0	
Benzo[k]fluoranthene		20.0	
Benzyl alcohol		20.0	
bis(2-Chloroethoxy) methane		20.0	
bis(2-Chloroethyl) ether		20.0	
bis(2-Chloroisopropyl) ether		20.0	
bis(2-Ethylhexyl) phthalate		20.0	
Butyl benzyl phthalate		20.0	
Chrysene		20.0	
di-n-Butylphthalate		20.0	
di-n-Octylphthalate		20.0	
Dibenzofuran		20.0	
Dibenz[a,h]anthracene		20.0	
Diethylphthalate		20.0	
Dimethyl phthalate		20.0	
Fluoranthene		20.0	
Fluorene		20.0	
Hexachlorobenzene		20.0	50
Hexachlorobutadiene		20.0	50
Hexachlorocyclopentadiene		20.0	50
Hexachloroethane		20.0	
Indeno[1,2,3-cd]pyrene		20.0	

TABLE C-5-2. DETECTION LIMITS OF ANALYTICAL METHODS

ANALYSIS	METHOD NUMBER	DETECTION LIMITS	TCLP DETECTION LIMITS
SVOC's, µg/L, continued			
Isophorone		20.0	
N-nitroso-di-n-propylamme		20.0	
N-nitrosodiphenylamine		20.0	
Napthalene		20.0	
Nitrobenzene		20.0	
Pentachlorophenol		100.0	250
Phenanthrene		20.0	
Phenol		20.0	
Pyrene		20.0	
Pyridine			50
Organosulfur Cmpds, µg/L	AEC UL-04		
1,4-Dithiane		25.0	
1,4-Oxathiane		50.0	
Dimethyldisulfide		25.0	
p-Chlorophenylmethylsulfide		75.0	
Benzothiazole		75.0	
p-Chlorophenylmethylsulfoxide		75.0	
p-Chlorophenylmethylsulfone		75.0	
Metals, **µg/L**	SW-846 #		
antimony	7041	0.70	
arsenic	7060	0.80	100
barium	6010	17.20	42.9
beryllium	6010	0.90	
cadmium	6010	2.60	9.60
calcium	6010	65.20	
chromium	6010	9.80	11.5
cobalt	6010	7.90	
copper	6010	14.90	
iron	6010	31.20	
lead	7421	0.30	68.4
magnesium	6010	18.50	
manganese	6010	4.30	
mercury	7470	0.100	0.100
molybdenum	6010	11.70	
nickel	6010	22.80	
potassium	6010	330.0	
selenium	7740	0.70	76.2
silver	7761	0.20	9.40
sodium	7770	72.00	
thallium	7841	2.00	
vanadium	6010	26.40	
zinc	6010	6.30	
TPH, **mg/L**	SW 846 # 8015		
Diesel fuel		0.250	
Gasoline		0.250	

TABLE C-5-2. DETECTION LIMITS OF ANALYTICAL METHODS

ANALYSIS	METHOD NUMBER	DETECTION LIMITS	TCLP DETECTION LIMITS
TPH, mg/L, continued			
Heavy oil		1.25	
Jet fuel		0.250	
Kerosene		0.250	
Mineral oil		0.250	
Naphtha		0.250	
Paint thinner		0.250	
Stoddard solvent		0.250	
Total unknown		0.250	
Pesticides/PCB's, µg/L	SW-846 # 8080		
4,4'-DDD		0.110	
4,4'-DDE		0.040	
4,4'-DDT		0.120	
Aldrin		0.040	
alpha-BHC		0.030	
Aroclor 1016		0.500	
Aroclor 1221		0.500	
Aroclor 1232		0.500	
Aroclor 1242		0.650	
Aroclor 1248		1.00	
Aroclor 1254		1.00	
Aroclor 1260		1.00	
beta-BHC		0.060	
Chlordane		0.140	
delta-BHC		0.090	
Dieldrin		0.020	
Endosulfan I		0.140	
Endosulfan II		0.040	
Endosulfan sulfate		0.660	
Endrin		0.060	
Endrin aldehyde		0.230	
gamma-BHC (Lindane)		0.040	
Heptachlor		0.030	
Heptaclor epoxide		0.050	
Methoxychlor		1.76	
Toxaphene		2.40	
Dioxin, µg/L	SW-846 # 8280 modified	0.010	
Total cyanide, µg/L	335.2	5.00	
Total phosphorus, mg/L	365.2	0.200	
TOC, mg/L	415.1	10.0	
BOD, mg/L	405.1	2.00	
Sulfate, mg/L	375.4	50.0	

Table C-5-l 0. Organic Removal Efficiency and Organic Loading Calculations

Organic removal eff iciencv (ORE)

$$ORE = \frac{(C_f - C_e)}{C_f} \times 100$$

where:

C_f = average feed organic concentration during test phase, mg/L
C_e = average liquid effluent organic concentration during test phase, mg/L

Parameter	Feed Concentration (mg/L) (average)	Effluent Concentration (mg/L) (average)	ORE (percent)
COD	17,727	1,557	91
TOC	4,152	444	89
TDG	8,260	5	99.9

Organic loading

$$\text{Organic loading} = \frac{Q_f \times C_f}{X \times V}$$

where:

Q_f = Fill volume added per day, 3 L/d
C_f = average feed organic concentration during test phase, mg/L
X = average concentration of biomass (3350 mg/L as MLSS or 2290 mg/L as MLVSS), mg/L
V = reactor volume. 60L

Parameter	Feed Concentration (mg/L) (average)	Organic Loading (g/g of MLSS-day)	Organic Loading (g/g of MLVSS-day)
COD	17,727	0.26	0.39
TOC	4,152	0.06	0.09