

JV Task 18 – Assessment of the Subsurface Fate of Monoethanolamine – Phase III

Final Report

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JV TASK 18 – ASSESSMENT OF THE SUBSURFACE FATE OF MONOETHANOLAMINE – PHASE III

EXECUTIVE SUMMARY

Burial of amine reclaimer unit sludges and system filters has resulted in contamination of soil at the CanOxy Okotoks decommissioned sour gas-processing plant with amines, amine byproducts, and salts. A three-phase research program was devised to investigate the natural attenuation process that controls the subsurface transport and fate of these contaminants and to apply the results toward the development of a strategy for the remediation of this type of contamination in soils.

Phase I experimental activities examined interactions between monoethanolamine (MEA) and sediment, the biodegradability of MEA in soils at various concentrations and temperatures, and the biodegradability of MEA sludge contamination in a soil slurry bioreactor. The transport and fate of MEA in the subsurface was found to be highly dependant on the nature of the release, particularly MEA concentration and conditions of the subsurface environment, i.e., pH, temperature, and oxygen availability. Pure compound biodegradation experiments in soil demonstrated rapid biodegradation of MEA under aerobic conditions and moderate temperatures (>6°C).

Phase II landfarming activities confirmed that these contaminants are readily biodegradable in soil under ideal laboratory conditions, yet considerable toxicity was observed in the remaining material. Examination of water extracts from the treated soil suggested that the toxicity is water-soluble. Phase II activities led to the conclusion that landfarming is not the most desirable bioremediation technique; however, an engineered biopile with a leachate collection system could remove the remaining toxic fraction from the soil.

Phase III was initiated to conduct field-based experimental activities to examine the optimized remediation technology. A pilot-scale engineered biopile was constructed at a decommissioned gas-sweetening facility in Okotoks, Alberta, Canada. On the basis of a review of the analytical and performance data generated from soil and leachate samples, the biopile operation has successfully removed all identified amines and removed significant amounts of organic nitrogen and organic carbon. Salts initially present in the soil and salts generated during the biodegradation of contaminants remain to be flushed from the soil. Laboratory data show that these salts are readily removable with a simple soil leach.

JV TASK 18 – ASSESSMENT OF THE SUBSURFACE FATE OF MONOETHANOLAMINE – PHASE III

INTRODUCTION

Research Program Rationale

Alkanolamines are commonly used by the natural gas industry to remove hydrogen sulfide, carbon dioxide, and other acid gases from the natural gas in which they occur (“sour” gas if hydrogen sulfide is present). Sour gas makes up a significant portion of the natural gas produced in both the United States and Canada. In Canada, about 40% of the annual net production is sour. Alberta produces approximately 83% of the total Canadian gas and is the largest producer of sour gas in North America. There are at least 90 gas sweetening-plants in Canada that use alkanolamines (Oilweek, 1994). The use of natural gas in general has increased significantly over the last decade, a trend that is expected to continue well into the future. As North America’s reserves of sweet gas are depleted, the use of sour gas will increase, which in turn will require a corresponding increase in the use of gas-sweetening facilities. At sour gas-processing plants, as at all plants that use alkanolamines for acid gas removal (AGR), spills and on-site management of wastes containing alkanolamines and associated reaction products have occasionally resulted in subsurface contamination that is presently the focus of some environmental concern.

Research Program Objectives

A three-phase program was initiated at the Energy & Environmental Research Center (EERC) to investigate the natural attenuation process that controls subsurface transport and fate of MEA- (monoethanolamine)-related wastes through development of data and insights under both laboratory and field conditions.

Phase I experimental activities examined interactions between MEA and sediment, the biodegradability of MEA in soils at various concentrations and temperatures, and the biodegradability of MEA sludge contamination in a soil slurry bioreactor. Phase II landfarming activities were undertaken to determine if these contaminants are readily biodegradable in soil under laboratory conditions. Phase III activities aimed to obtain key treatability data that could be used to select and design a remediation option.

Project Support and Participants

Funding for this research program was provided by Canadian Occidental Petroleum Ltd. (CanOxy), the Canadian Association of Petroleum Producers (CAPP), the U.S. Department of Energy (DOE), GRI, Environment Canada, and the National Energy Board of Canada.

BACKGROUND INFORMATION

Alkanolamines

Alkanolamines can be considered organic derivatives of ammonia and are classified as primary, secondary, or tertiary alkanolamines. The classifications are based on the number of substituent groups attached to the nitrogen atom. The molecular formula for ammonia is NH_3 . Substitution of an organic group, R, for one of the hydrogen atoms gives a primary amine, represented as RNH_2 . Similarly, the substitution of two and three hydrogen atoms by organic groups results in secondary (RNHR) and tertiary (RNRR) alkanolamines, respectively (Solomons, 1988). MEA is classified as a primary amine and is considered to be the most basic of the alkanolamines, therefore the most reactive toward acid gases. Diethanolamine (DEA), methyldiethanolamine (MDEA), and diisopropanolamine (DIPA) are also commonly used for gas-sweetening operations (Skinner and others, 1995). The alkanolamines are also completely miscible in water. Typical concentrations for MEA usage fall in the range of 15–20 wt%, although concentrations as high as 35% may be required under conditions of high acid gas content (Gagliardi and others, 1989).

In gas-sweetening systems, alkanolamines can be involved in a variety of reactions that produce chemical species that may have a role in alkanolamine-related subsurface contamination. The products formed by these reactions range from simple breakdown products (for example ammonia, water, and hydrogen gas) to complex nitrogenous organic compounds, referred to here as thermal/oxidative (T/O) reaction products, about which little is known concerning their chemical identity and properties. Some of the T/O reaction products commonly associated with MEA include *N*-(hydroxyethyl) imidazolidone (HEI); *N*-(hydroxyethyl)-1,2-ethylenediamine (HEED); *N,N*-bis-(hydroxyethyl) ethylenediamine (BHEED); and 2-oxazolidinone (OX). These are produced by MEA reactions with CO_2 and/or COS (Skinner and others, 1995). These T/O reaction products typically occur in sludges that accumulate in the reboiler tanks and filters of amine-based AGR units.

The operation of AGR units creates a variety of wastes that may be introduced to the environment. The managed and unmanaged waste streams may be composed of spent alkanolamines, sludges from process unit tank bottoms, and process system filters. Unmanaged waste streams, particularly spills during changeover operations (the process of exchanging spent chemical for fresh chemical), may also include fresh alkanolamines. The T/O reaction products discussed above are typically present in the waste streams in varying concentrations, depending on the process conditions. Additional components of the wastes include additives such as corrosion inhibitors and anti-foaming agents, benzene, and trace metals (Sorensen and others, 1998).

Okotoks Site Description

A decommissioned sour gas-processing plant located near Okotoks, Alberta, owned by CanOxy was the source of samples and field data for the laboratory-based experimental work. This site was selected to be the location for the field-based efforts.

The decommissioned Okotoks sour gas-processing plant has the following characteristics: 1) the geologic and hydrologic setting at Okotoks is representative of a number of other Alberta sour gas-processing sites; 2) MEA, which is used at more than one-third of Alberta sour gas-processing plants, was used exclusively at the site for the duration of its active life; 3) a substantial amount of information concerning MEA use at the site and the management of MEA-related wastes was readily available prior to the study; 4) extensive site assessment activities prior to the study resulted in a reasonably complete understanding of the geologic setting and hydrologic system of the site and also provided a general delineation of the MEA-related contamination; 5) an extensive groundwater-monitoring system was already in place; and 6) a variety of activities being undertaken at the site by other groups offered the opportunity for cooperation that would lead to increased efficiency and cost-effectiveness. Additional factors that made the Okotoks plant a favorable site for research were its proximity to Calgary, the presence of unused facilities that could be made available for use by on-site investigators, and its inactive status, which eliminated a variety of safety concerns.

The Okotoks Gas Plant is located approximately 25 km south of Calgary and 1 km east of the town of Okotoks in south central Alberta (see Figure 1). The town of Okotoks has a population of approximately 7200 people and is located in an area where farming and ranching are the primary land use activities. The gas plant is bordered to the south by pasture lands and the Sheep River, to the east by crop and pasture lands, to the north by croplands, and to the west by urban industrial and residential properties.

The climate of the Okotoks area is semiarid to moist, with an average annual precipitation of 488.2 mm/year. The frost-free period for the area is between 75 and 90 days. Average temperatures in the region are typically below 10°C 8 to 9 months of the year.

The Okotoks site can be physiographically divided into two areas, the terrace uplands, in which the plant facilities, landfill, and MEA sludge disposal pits are located, and the river valley in the area of the sulfur block, south of the plant. The contaminated soil samples used in the laboratory-based research activities described below were taken from the terraced upland portion of the site.

The near-surface geology of the site as a whole is dominated by unstratified glacial sediments comprising clay-rich glacial till with intercalated gravels, sands, and silts. The entire glacial till sequence in the area averages approximately 30 m in thickness. The tills are, by definition, extremely heterogeneous in texture and anisotropic in stratigraphic distribution.

Nature and Extent of Soil Contamination

Up to four former amine disposal pits were identified at the Okotoks site from aerial photographs (Shoal Environmental Consultants, 1994). The pits appear to have been simple dozer trenches advanced in the field in the northeastern portion of the plant property on the upper terrace of the site. These pits were mainly used to store tank-bottom sludges from the amine regeneration process. The four sludge disposal pits had been constructed sequentially from 1967 to 1974. By the time the pits were closed in 1984, they contained an estimated 2000 to 3000

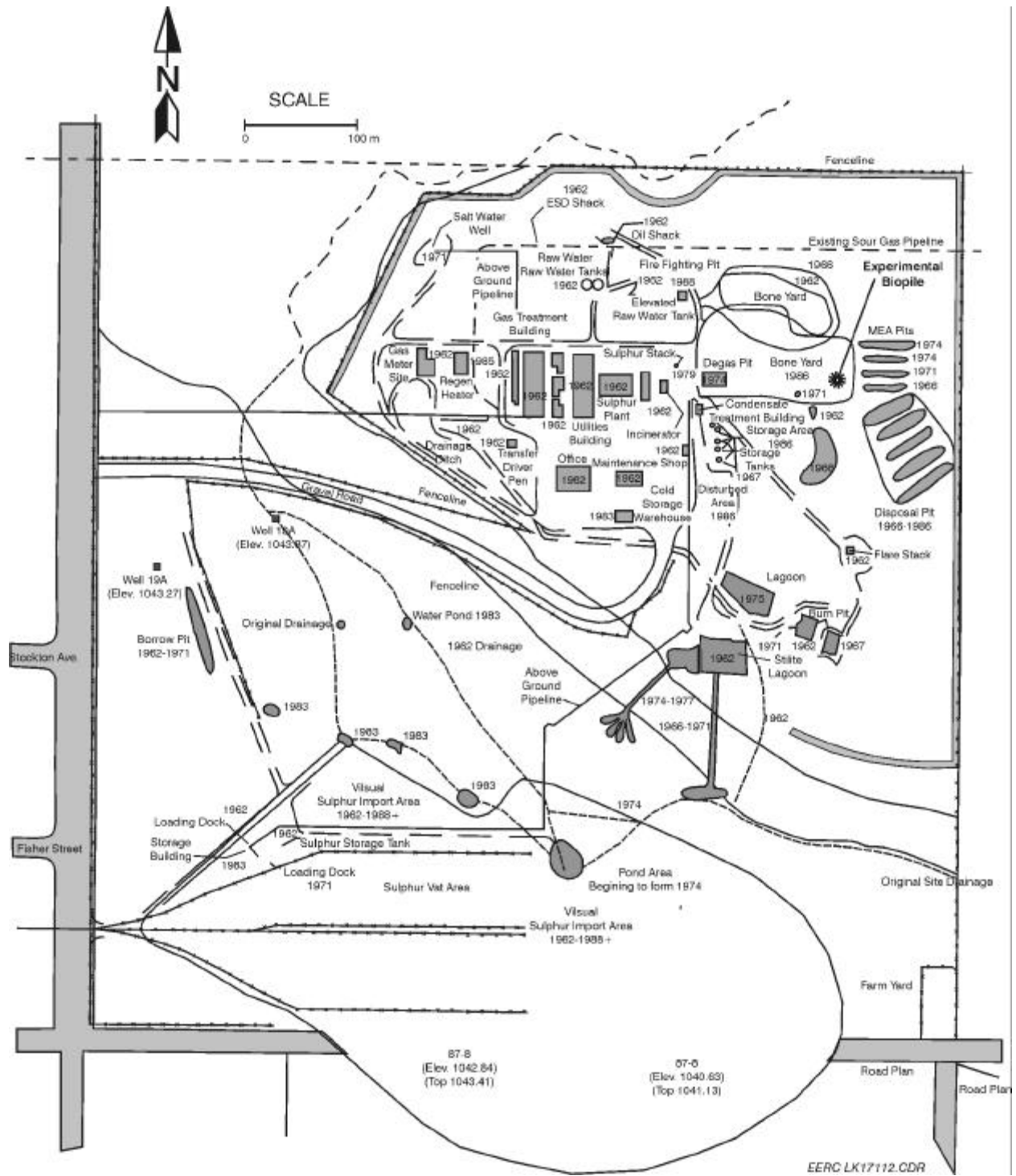


Figure 1. Location of the decommissioned Okotoks sour gas-processing plant.

barrels of MEA process waste. Upon closure, the liquid and sludge contents of the pits were removed. The liquids were injected into the site's saltwater disposal well, and the sludges were spread on the firebreaks around the plant and on the sulfur basepad. The pits were then backfilled and reclaimed.

The results of an environmental assessment conducted in 1992 as part of the plant's decommissioning process revealed the presence of a layer of sludge-contaminated material at a depth of 2 to 3 m in the area of the former amine sludge disposal pits. A strong ammoniacal odor in the sediments and elevated total organic nitrogen concentrations suggested that the contamination was MEA-related.

Extensive site assessment activities performed by other environmental consulting groups prior to this investigation indicated the presence of MEA-related subsurface contamination in the area of reclaimed pits that had been used for the disposal of wastes from the gas-sweetening system. Although the process-derived sludge had been removed, as noted, prior to pit closure, the location and character of this sludgelike material indicate a relationship with the original sludge, probably residual MEA-related contamination derived from that sludge. The conclusion that the contamination was MEA-related was based on elevated ammonia and total Kjeldahl nitrogen (TKN) concentrations detected in soils and groundwater in the area of the pits. Geophysical techniques provided good delineation of the extent of the subsurface impact extending from the pits themselves (Komex, 1996). Indications of MEA-related subsurface soil contamination were also present in the area of the sulfur block where sludges had been spread, as well as the area of the plant where MEA was unloaded and stored for use.

The lithology at the former amine pit locations indicated a grayish brown, moist, firm, clayey silt fill with no odor or staining to approximately 2 m below ground level (bgl). The sediment became wet at 1.4 m bgl. This fill covered the pit sediment, which consisted of very dark gray, wet, loose, silt, with a strong amine (ammoniacal) odor. At approximately 3.5 m bgl, a yellowish brown (weathered), moist, stiff, silty clay till was encountered with a slight odor and gray staining in fractures to a depth of 7.6 m bgl.

The areal extent of soil contamination associated with the amine pits, on the basis of a suite of test holes and EM31 geophysical survey data, was estimated at approximately 4250 m². The in situ volume of this impacted soil is estimated at 8000 m³ to 12,000 m³ (Komex, 1996).

A variety of soil samples were collected at Okotoks for organic and inorganic characterization of the amine-related contamination. Contaminated materials from the MEA pit were sampled at depths of up to 3.5 m, depending on where the contaminated material was encountered, which was primarily determined by the presence of an ammonia smell that is generally associated with MEA. A backhoe was used to dig out the samples at each collection location. The samples were then placed into 1- and 4-liter glass jars, which were packed in ice and shipped to the EERC for extensive analysis and use in a variety of laboratory-based experimental activities.

Qualitative gas chromatography–mass spectroscopy (GC–MS) analysis of a soil sample from the MEA pit revealed numerous peaks, including naphthalene, C2-naphthalenes, dibenzothiophene, and a significant amount of HEI, an MEA T/O reaction product. Additional compounds were tentatively identified as benzothiazole, methylthiophenes, and methylbenzothiophenes. Dilute acid extracts from Okotoks amine pit soil were also analyzed quantitatively by ion chromatography (IC). The IC analysis detected an MEA concentration of approximately 300 mg/kg in the MEA pit sediment sample extract.

PHASE I RESEARCH

Phase I of the research program was designed to evaluate the primary natural attenuation processes that control the subsurface transport and fate of MEA, biodegradation, and interactions between MEA and sediment. Experimental activities were designed to provide estimates of 1) the time required to initiate the biodegradation, 2) the rates of degradation of the contaminants, 3) the degree of difficulty in operation, and 4) the composition of the material remaining after slurry treatment. Detailed discussion of Phase I research activities is provided in Gallagher and others, 1996.

Results of Phase I Slurry Experiments

Soil contaminated by MEA-related sludge biodegraded well for the first 15 days, but metabolism was slower thereafter. Operation of the bioreactors for 24 days reduced the chemical oxygen demand (COD, a measure of organic carbon) and organic nitrogen concentration 79% and 59%, respectively, at 8% soil concentration. COD removals were approximately the same at all three slurry concentrations. TKN reductions indicated toxicity that resulted in reduced removals at higher slurry concentrations. The complete data set from these experimental activities is provided in Gallagher and others (1996).

While the data on MEA-contaminated material are limited, it was clear that the contaminants, as measured by COD, will degrade sufficiently and rapidly in a slurry bioreactor, although between 20% and 40% of the COD and organic nitrogen appeared to be recalcitrant. In general, the results of the Phase I bioslurry experiments suggested that land treatment of the MEA-contaminated soils may be a technically viable remediation technique.

PHASE II RESEARCH

Phase II research included landfarming experiments designed to examine the effectiveness of land treatment for reducing MEA-related contamination and toxicity in soil. The key variables chosen to be evaluated in these tests are the following: 1) phosphorus dose, 2) tillage frequency, 3) loading rate of contaminated soil, and 4) pH adjustment. The experimental design chosen to evaluate these variables is a 2^{4-1} factorial design. Eleven separate trials were run simultaneously and treated identically except for perturbations dictated by the variable levels. The variables of phosphorus dose, tillage frequency, and soil loading were given as high, low, and middle

concentrations. The concentrations were selected to bracket the practical range that may be used in a full-scale landfarming operation. The laboratory-scale landfarms were covered (with small holes for air exchange) and incubated statically in the dark at room temperature. Core samples were taken from each landfarm for each week of the study period, which lasted 120 days. The soil samples were analyzed for moisture, pH, ammonia, nitrate plus nitrite, and microbial counts. Samples from the beginning and end of the study period were analyzed for toxicity, COD, and TKN. Detailed descriptions of the experimental designs and methods are provided in Gallagher and Sorensen, 1997.

Results of Phase II Landfarm Experiments

Landfarm experiments showed that the most important variable affecting bioremediation was soil loading. Increases in phosphorus resulted in increases in ammonia, suggesting that bioactivity increased. Tillage was not observed to be a significant variable in these experiments, although all landfarms were tilled a minimum of once per week, so the effect of zero tillage is not known. With regard to contaminant removal, the most effective landfarm removed 51% of the initial COD, 68% of the initial TKN, and 66% of the organic nitrogen. The data showed that toxicity of some components in the contaminated soil resulted in reduced bioactivity, longer lag times, and reduced removals. On the basis of the bacterial luminescence test (Microtox[®]), toxicity in all landfarms at the lower soil loadings (10 and 17.5 wt%) was reduced to zero. However, the results of additional assessments via seed emergence, root elongation, and earthworm survival suggested that significant toxicity remained even in the lower soil-loading conditions. Further testing showed that the toxic fraction of the treated soil is extractable in both water and methanol, which suggests that those components may be leachable from the soil. The identity of these toxic components is not known. The complete set of data from the Phase II experimental activities are provided in Gallagher and others (1997). Phase II results indicate landfarming is not the most desirable bioremediation technique. Because the remaining toxic fraction is water-extractable, a design including a leachate collection system, such as an engineered biopile, may be a viable and effective alternative to landfarming. The biodegradation of the contaminated material in a biopile would be expected to be very similar to that in a landfarming cell, and leachate could easily be collected for further abiotic treatment or disposal. The use of engineered biopiles for the bioremediation of sludges and contaminated soils is widely practiced in the oil and gas industry as an inexpensive and effective means of removing contaminants. Phase I and II results indicated the operation of an engineered biopile is a logical and economically viable method for the bioremediation of alkanolamine sludge-contaminated soil.

PHASE III RESEARCH

Phase III research activities are field based investigations designed to evaluate the effectiveness of an engineered biopile for remediation of MEA-contaminated soils.

Biopile Design and Operation

In the spring of 1998, a demonstration-scale, or pilot, biopile operation was designed by the EERC and Hazco Environmental Services of Calgary. In the summer of 1998, Hazco constructed the biopile, and operation was initiated on August 5. A majority of the monitoring and sampling activities were conducted by Matrix Solutions, while routine analyses were provided by Norwest Laboratories of Calgary and Edmonton. Analyses for amines and T/O reaction products were performed by the EERC.

The biopile containment cell measures 40 m long by 10 m wide by 1.5 m deep. Above a 25-mil reinforced polyethylene (RPE) liner is a thin layer of crushed gravel. A filter fabric caps the gravel layer and lies directly beneath the treatment soils. The soil layer is gently mounded and enclosed by a 25-mil RPE cover. Figure 2 is a plan view of the biopile showing the layout of the irrigation and aeration systems, leachate sump unit, leachate collection tank, air blower unit, electrical supply, freshwater supply tank, and water-pumping system. Figure 3 is a cross-sectional view of the biopile.

Approximately 450 m³ of contaminated soils and 50 m³ of straw are housed within the constructed cell. Soil additives include 2.58 m³ of calcium chloride (CaCl₂) as well as 10–34–00 (percent nitrogen–phosphorus–potassium) liquid fertilizer (2036 kg). The fertilizer addition estimate was based on the mean organic carbon content of the amine-contaminated soil using a ratio of COD to phosphorus of 100:1. Fertilizer was added as a nutrient source for the microbial

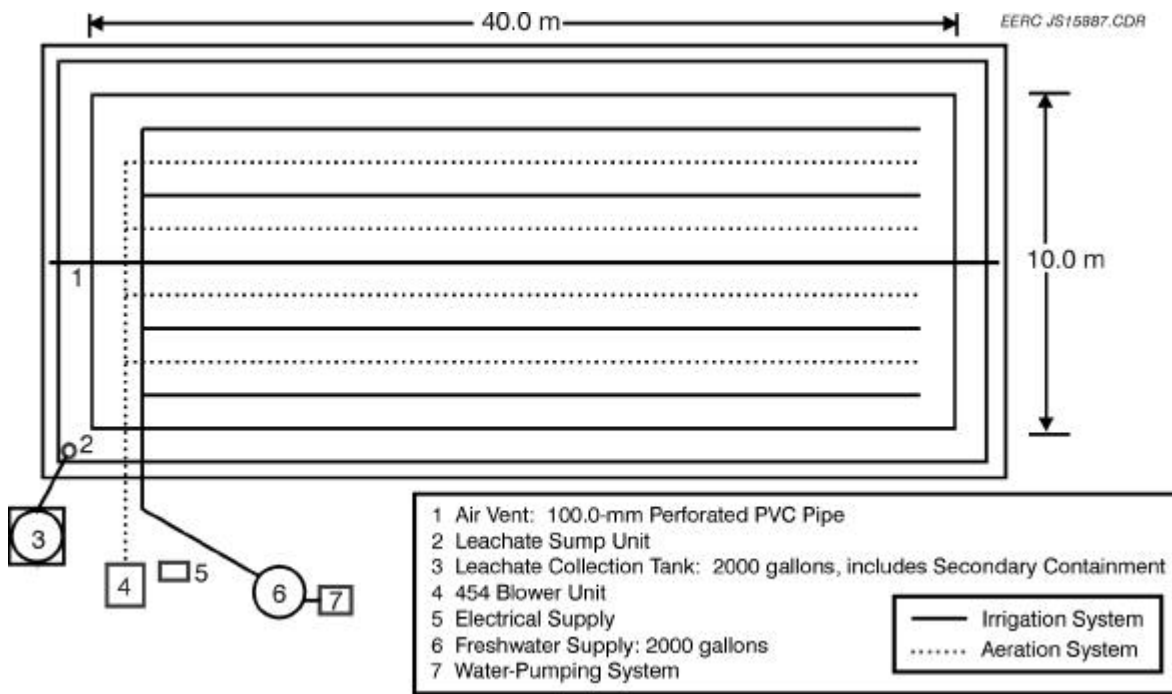


Figure 2. Plan view of the biopile showing the layout of the aeration and irrigation system.

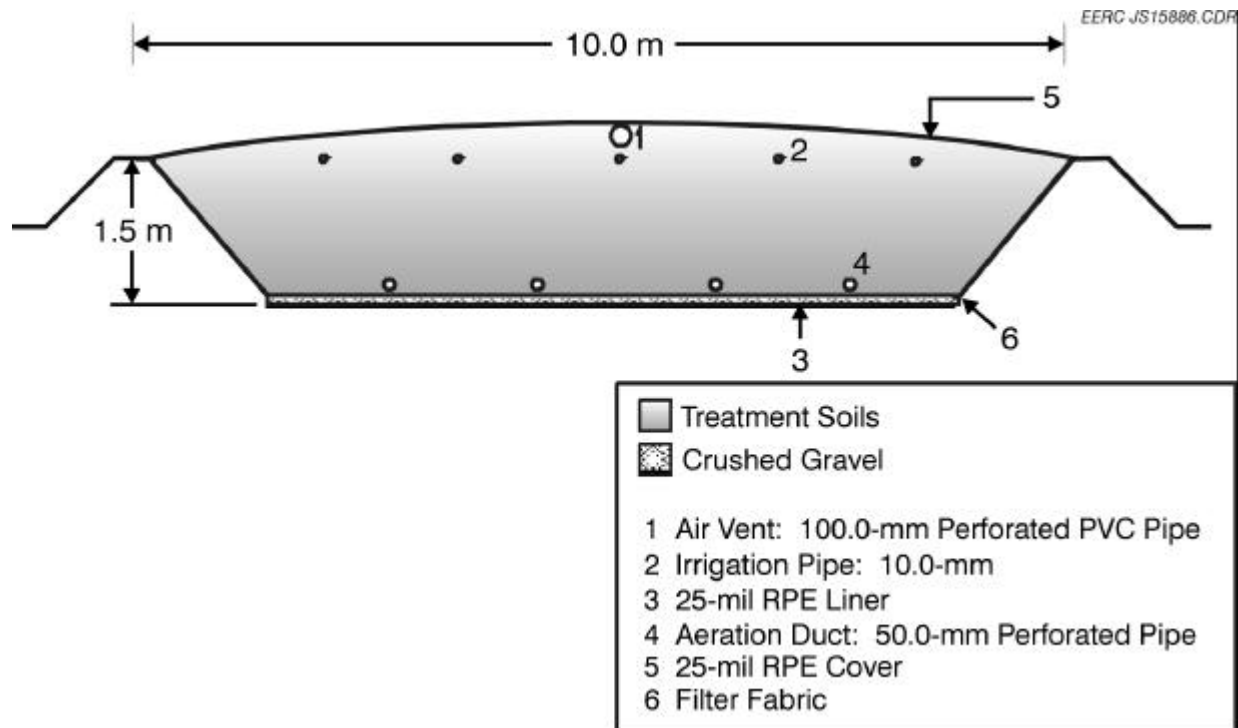


Figure 3. Cross-sectional view of the biopile.

population to improve the biodegradation rate. Straw was added to act as a bulking agent, thereby increasing the porosity and permeability of the biopile. The addition of CaCl_2 effectively increases the permeability of the soil, allowing water to move more easily through the biopile. The calcium addition rate was estimated from laboratory tests with Okotoks soil as the minimum dose to achieve the maximum hydraulic conductivity.

Aeration, irrigation, and leachate collection systems are the dynamic components of the structure. Aeration is performed to supply oxygen to the microbial population, which in turn enhances biodegradation. The aeration system consists of a large air vent (100-mm perforated PVC pipe) coming from the blower to a manifold where four equally spaced flow ducts (50-mm perforated pipe) run the entire 40-m length of the biopile. The aeration piping was installed in the biopile horizontally at a level about one-third the height of the soil. At the distal end of each aeration leg, a riser leads to the top of the soil. The windspeed of air moving through this pipe can be estimated by removing the plug in this riser. The flow ducts are powered by an external blower unit. The blower was sized to supply 1 to 1½ pore volumes of air through the pile per day.

Adequate soil moisture is also necessary to obtain appropriate microbial activity, although if the soil is saturated or nearly so, aeration will be inhibited. Therefore, water is periodically added to the biopile using an irrigation system. The timing and amount of water application is determined by soil moisture measurements and the need to leach the soil. The irrigation system comprises five semipermeable hoses, equally spaced, which also run the full length of the structure on the soil surface. The irrigation system water is pumped from the on-site well to an external

freshwater supply tank (2000 imperial gallons) and from that tank to the irrigation hoses. Leachate moves via gravity to a sump situated below the biopile grade, where it is pumped to an external tank (2000 imperial gallons) by a float-activated sump pump. The leachate generated at the Okotoks biopile is disposed of in an on-site injection well.

Temperature of the soil was monitored by placement of three equidistant thermocouples at a depth of about 0.75 m in a line down the long axis of the pile. The leads to these thermocouples ran to the near edge of the pile, allowing for attachment to a handheld pyrometer.

Construction of the biopile was initiated and completed during July of 1998. The aeration system was activated and the first 2000 gallons of water were added to the biopile at the beginning of August, and forced aeration was initiated on August 5, 1998. Another 2000 gallons of water were incorporated into the biopile at the end of September. Aeration and pumping were suspended for the winter in mid-November, and the pile was mixed at the end of November to break up channels that may have formed during summer and fall operation. No sampling or monitoring activities were performed during the winter months. Reactivation of the aeration and irrigation system occurred on July 23, 1999. In mid-August, 1200 gallons of water were incorporated into the biopile. On September 22, 1999, forced aeration was stopped and the operation of the biopile was shifted to leaching mode. Aeration was halted because an evaluation of the data suggested that active biodegradation had reached a plateau. Laboratory activities in previous phases showed that once a plateau is attained, limited additional benefit accrues from bioactivity. Approximately 10,900 gallons of water were incorporated into the biopile during 1999 operations. Figure 4 shows the quantity of water incorporated and leachate generated during operations. Operations were suspended November 2, 1999, because of freezing weather.

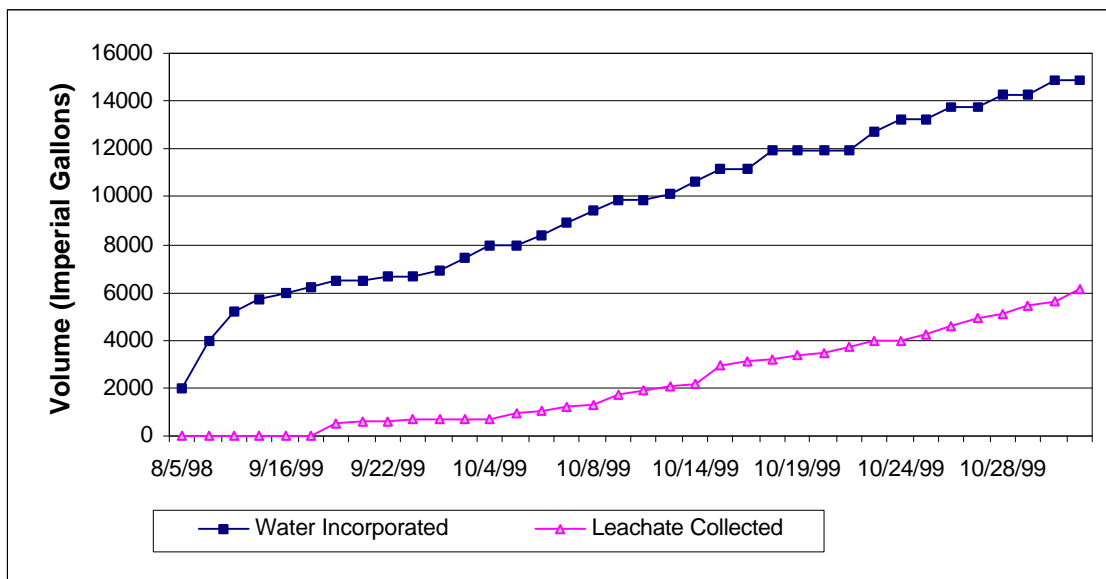


Figure 4. Cumulative water incorporation and leachate generation during the operation of the biopile.

Biopile Sampling and Analysis

A sampling plan was prepared during the start-up period. This plan involved an imaginary division of the pile into eight equal-sized blocks of approximately 10×5 m labeled A through H, as shown in Figure 5. At each sampling period, three preselected blocks were sampled using a hand-operated coring device. For routine analytical parameters, separate upper (the upper 0.75 m) and lower (bottom 0.75 m) cores were taken, for a total of six samples. For initial characterization and for toxicity assessments, upper and lower cores from each of the three grids were composited. The blocks were chosen in a pseudorandom manner to ensure representativeness. Samples were collected by Matrix Solutions staff and submitted promptly to the appropriate laboratory. In addition to the soil sampling, the temperature of the soil and the windspeed at the riser of each leg of the aeration system were measured and the moisture was estimated using a handheld meter.

Upon completion of construction and immediately prior to the beginning of active operation (wetting and aeration), samples of biopile material were collected for a baseline characterization. A composite sample was submitted to a commercial laboratory in Calgary for the following analyses as required by the Alberta Energy and Utilities Board (EUB): hydrometer sediment size analysis, salinity, pH, conductivity, sodium adsorption ratio, bulk density, moisture, soluble salts, soil organics, and metals. Separate samples were collected and analyzed for total organic carbon (TOC), ammonia ($\text{NH}_3\text{-N}$), and nitrate + nitrite-N ($\text{NO}_x\text{-N}$). Start-up and shutdown samples were evaluated for their toxic effects on earthworm survival, lettuce seedling emergence, lettuce root elongation, and bacterial luminescence (using Microtox[®]).

Once the wetting and aeration operations began, soil samples were collected on a biweekly basis. The biweekly samples were analyzed for pH, conductivity, TKN, TOC, $\text{NH}_3\text{-N}$, and $\text{NO}_x\text{-N}$, which are considered the key soil character parameters for evaluating the general activity of the biopile over the course of the study period. Moisture by wet weight percent was also determined to indicate how often to wet the biopile.

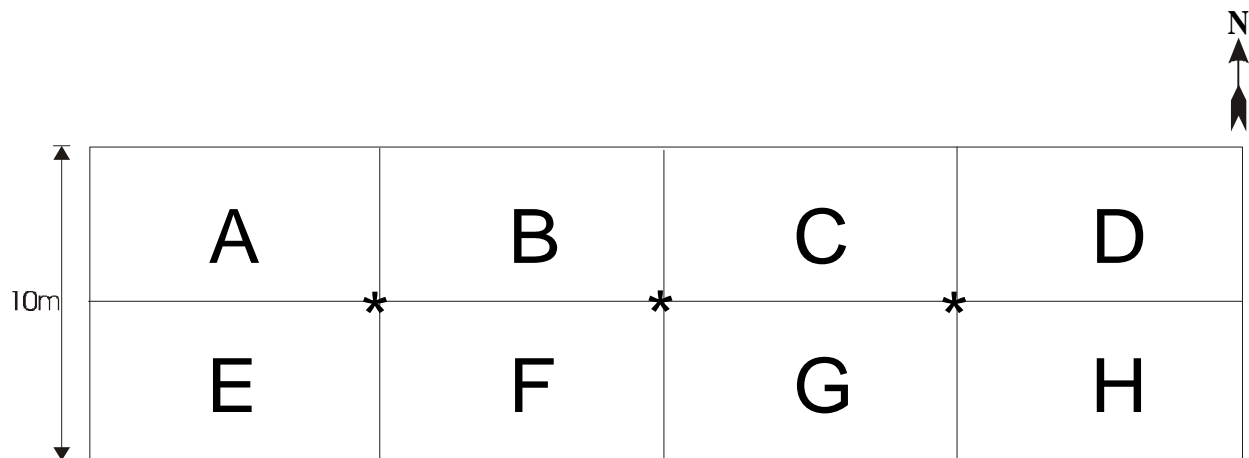


Figure 5. Block layout for sampling of the biopile (* indicate thermocouple locations).

Samples of leachate generated during 1998 operation were collected after the first week of operation (August 1998) and just prior to winter shutdown (late October 1998). During 1999, leachate samples were collected biweekly, concurrently with soil-sampling events. Leachate samples were analyzed for pH, conductivity, TKN, ammonia-N (NH₃-N), NO_x-N and COD.

The complete soil and leachate sampling schedule is given in Table 1.

Phase III Results

An initial characterization of the soil in the biopile is shown in Tables 2 and 3. These data show that the soil is of moderate salinity (EC of 7.66 dS/cm), high in organic nitrogen (5770 mg/kg), and moderately high in organic carbon (28,600 mg/kg). The sodium adsorption ratio (SAR) indicated a soil with high sodium. Initial soil toxicity results are shown in Table 4. IC₂₅ and IC₅₀ are concentrations inhibiting the control results by 25% and 50%, respectively. The toxicity data show that at the beginning of treatment the soil was toxic-to-highly toxic, depending on the test species. When toxicity was reassessed at the end of the first season of treatment (after about 90 days of treatment), toxicity for most species declined significantly. Much of the toxicity remaining during the November sampling is likely due to salinity. Additional toxicity testing should be performed once the salinity of the biopile soil has been reduced to target levels.

TABLE 1

Soil and Leachate Sampling Schedule

Soil Sample Date	Soil Grids Sampled	Leachate Sample Date
17 Jul 98	A C H	14 Aug 98
14 Aug 98	B D G	25 Sep 98
28 Aug 98	A E F	09 Oct 98
11 Sep 98	B C H	23 Jul 99
25 Sep 98	D F G	17 Aug 99
09 Oct 98	A C F	08 Sep 99
27 Oct 98	B D H	27 Sep 99
09 Nov 98	H	12 Oct 99
26 Nov 98	A E G	25 Oct 99
23 Jul 99	B D G	
17 Aug 99	A E F	
08 Sep 99	D E G	
27 Sep 99	A C F	
12 Oct 99	B D H	
25 Oct 99	A E G	

TABLE 2

Baseline Soil Characterization

Hydrometer Sediment Analysis			
Sand, %	34.3		
Silt, %	28.6		
Clay, %	37.1		
Particle Size:	Clay Loam		
Salinity		Elemental Composition, mg/kg	
pH	7.3	Arsenic	5.8
Conductivity, dS/m	7.37	Barium	196
Saturation, %	53	Beryllium	0.559
Sodium Adsorption Ratio	2.9	Cadmium	0.522
		Chromium	15.5
Physical		Cobalt	6.9
Bulk Density, gm/cm ³	1.44	Copper	18
Moisture, wt%	16.9	Lead	11.5
		Mercury	0.03
Soluble Salts		Molybdenum	0.38
Calcium, mg/kg	398	Nickel	21.6
Magnesium, mg/kg	111	Selenium	1.13
Sodium, mg/kg	183	Thallium	0.4
Potassium, mg/kg	214	Vanadium	19.1
Sulfate-S, mg/kg	110	Zinc	69.8
Chloride, mg/kg	587		

TABLE 3

Initial Soil Characterization

Analyte	Concentration, mg/kg
Total Kjeldahl Nitrogen	6250
Ammonia-Nitrogen	480
Nitrate + Nitrite-Nitrogen	130
Organic Nitrogen	5770
Total Organic Carbon	28600
Dean Stark Oil	1200
Total Purgeables	2.1
Total Extractables	124
Extractable Organic Halides	<0.3

TABLE 4

Toxicity Assessment of the Amine Waste-Contaminated Soil		
	July 1998	November 1998
Bacterial Luminescence		
IC25	NA ¹	>91
IC50	NA	>91
Earthworm Survival		
LC25	4.9	27
LC50	16	34
Root Elongation		
IC25	22	24
IC50	43	50
Seed Germination		
IC25	62	>100
IC50	>100	>100
Seedling Emergence		
IC25	24	39
IC50	40	60

¹ The bacterial luminescence test conducted on the July sample was performed only at the screening level, and IC25 and IC50 values were not determined. The results of the July bacterial luminescence tests are provided in Appendix D.

Figure 6 shows the mean concentrations of nitrogen species in the biopile soil, which includes TKN, organic nitrogen (calculated as the TKN-nitrogen minus ammonia-nitrogen), NO_x-N, and total nitrogen (the sum of the TKN-nitrogen, ammonia-nitrogen, and NO_x-N) during the operation of the biopile. These data show that the ammonia concentration increased quickly, fell, and increased again. Increases in ammonia are due to biodegradation of amines which in turn releases ammonia. Decreases in ammonia are due to volatilization, which is expected to be minor at the soil pHs, through leaching and through biological oxidation to nitrate or nitrite. Of these routes, the biological oxidation to nitrate and nitrite is probably the most important. Biological oxidation of ammonia is mediated by autotrophic bacteria that have slow growth rates and that are very sensitive to toxic components and to the presence of organic matter.

Nitrate + nitrite-N did not become significant until the end of the first and into the second season. This behavior of the NO_x-N is consistent with the slow development of a population of bacteria that can mediate ammonia oxidation. Losses of NO_x-N are generally through leaching and through biological denitrification. Denitrification does not occur significantly when oxygen is present. Thus, the major route of NO_x-N loss is through leaching. Organic nitrogen generally fell from the starting value of 5770 mg/kg to about 3800 mg/kg, where it has remained. Organic nitrogen can be lost by volatilization and through biological degradation. Volatilization, while occurring at some low level (evidenced by the amine smell to the contaminated soil), was not expected to be significant. Despite the high water solubility of the amines, leaching is not expected to be a major route of their loss (see Gallagher and others, 1996). The data show that the soil was heterogenous, making results more difficult to interpret.

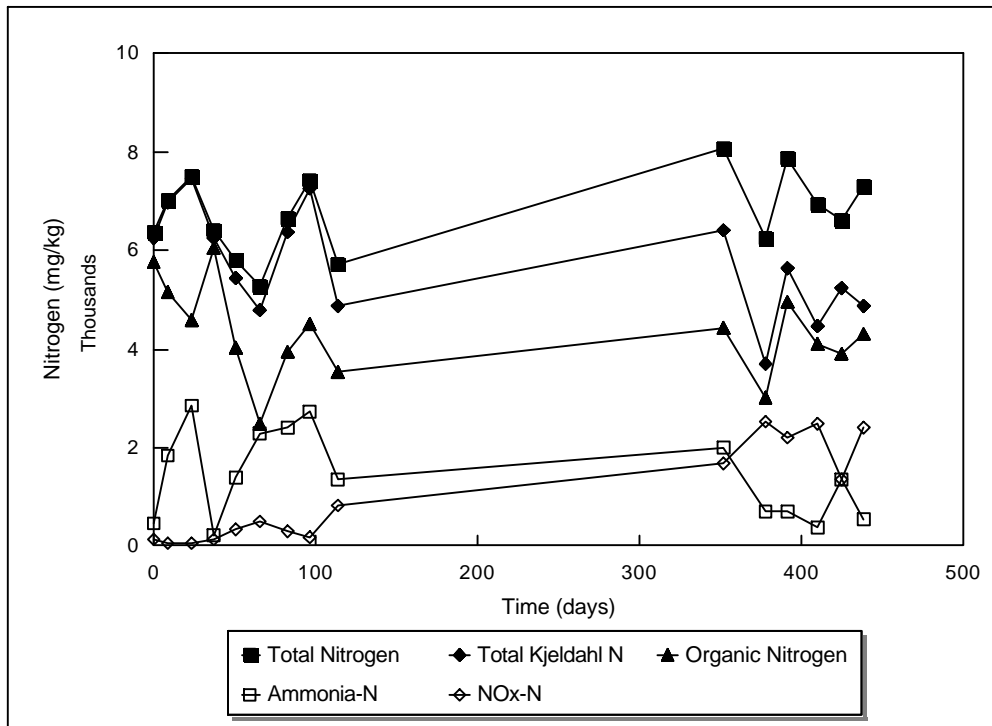


Figure 6. Variation in concentrations of nitrogen species during the operation of the biopile.

Figure 7 shows the concentrations of TOC during the operation of the biopile. The initial TOC was 28,600 mg/kg, and the concentrations generally fell through the first season to about 18,000 mg/kg, where, with considerable bouncing, it has remained. As the TOC includes all organic carbon, the interpretation of this parameter is confounded by natural soil organic matter and by the addition of straw to the soil. The observed TOC data are consistent with a rapid biodegradation of readily biodegradable organic matter. The plateau in TOC shows that the remaining organic matter is resistant to biodegradation.

Electrical conductivity (EC) is a measure of the concentration of dissolved ions present in the soil. The initial EC of 7.66 dS/m rose quickly, then more slowly through the operation of the biopile, with a small decrease and increase again at the end of operation (Figure 8). Increases in conductivity were expected as a result of conversion of the organic amines to their mineral components (carbonate and ammonia). Additionally, as the ammonia is oxidized to $\text{NO}_x\text{-N}$, the EC would be expected to increase for two reasons: first, $\text{NO}_x\text{-N}$ is a better conductor than ammonia and, second, ammonia may occur in a charged (NH_4^+) state, which would contribute conductivity, and a uncharged state (NH_3), which occurs as a dissolved gas. Conductivity is removed from the soil only through leaching.

The concentration of the soil moisture in the biopile during the operating period is shown in Figure 9. Soil moisture began high, with a mean of 18.5%, showed a sharp decline about Day 50, then recovered to around 20%. Soil moisture remained high throughout the remainder of

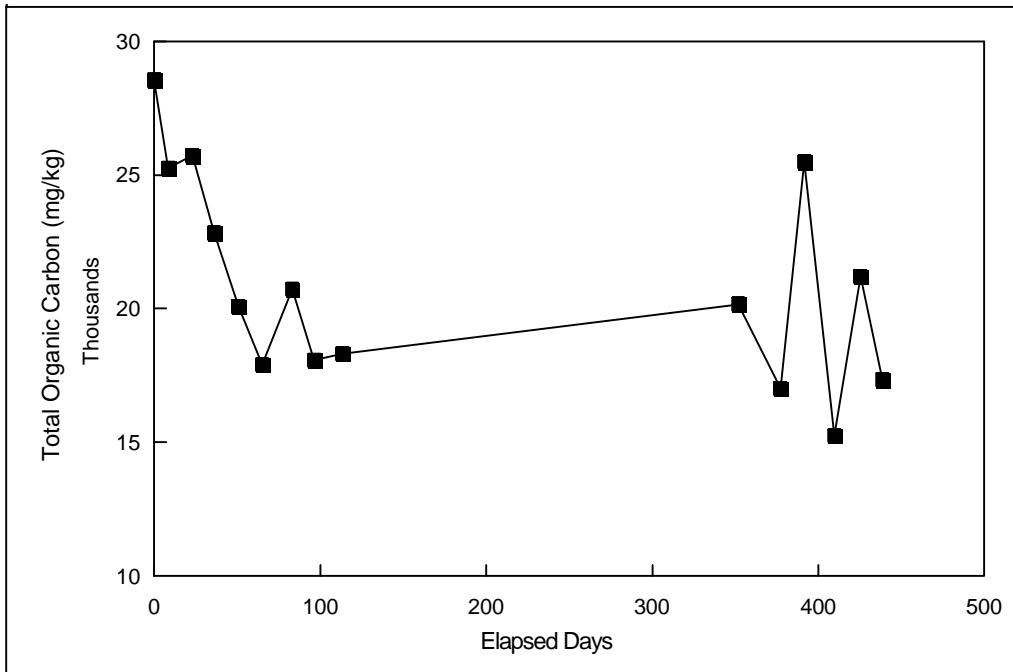


Figure 7. Variation in the concentration of TOC during the operation of the biopile.

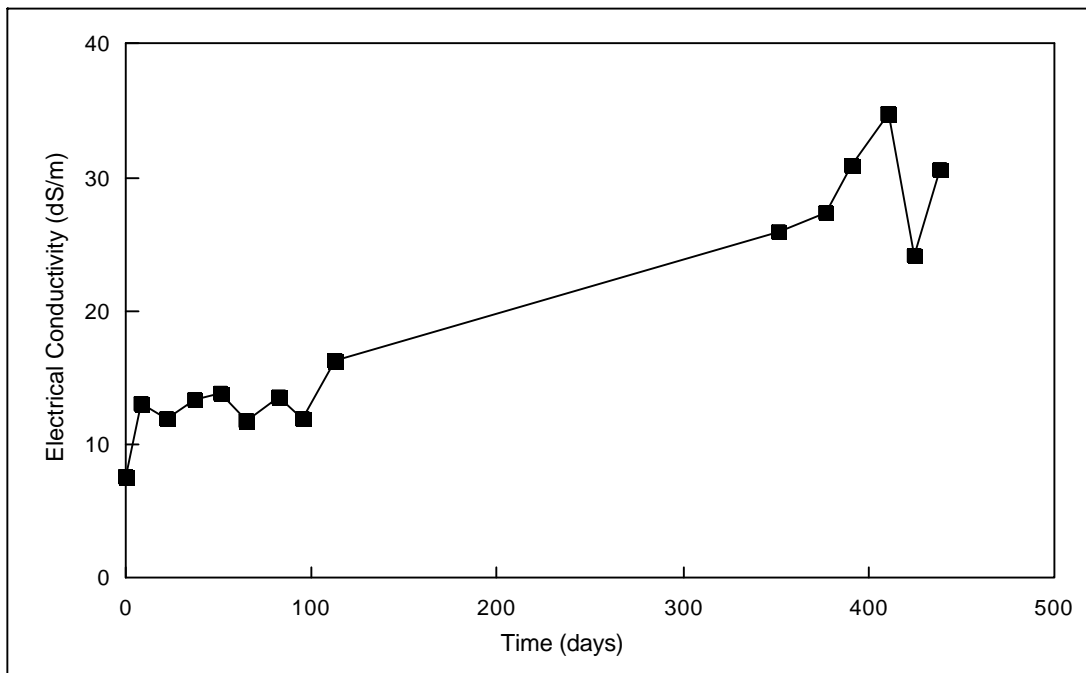


Figure 8. Variation in the EC of the soil during operation of the biopile.

operation. Soil moisture is generally optimal for bioactivity when it is around 60% to 80% of the holding capacity of the soil. The estimated moisture-holding capacity was about 20%–22%. Losses of moisture could occur through leaching and through the drying action of the forced air. The data suggest that moisture was probably not a limiting factor for any significant time during the operation of the biopile.

Figure 10 shows the variation in soil pH during operation of the biopile. The initial soil pH was 7.3, with the pH increasing by about 0.5 units during the first season and decreasing to about 6.8 in the second season. Interpretation of the pH is somewhat complicated. The pH would be expected to rise slightly as amines (basic in pH) are degraded, but may increase because the resultant ammonia is even more basic than most amines. The pH should show significant decreases as the ammonia is oxidized due to loss of ammonia and to acid produced during its oxidation. The pH remained within the range of suitability for bioactivity during the entire time.

The temperature of the biopile was monitored to show when it was suitable for bioactivity. Temperature effects on bioactivity are similar to those for other chemical reactions—activity doubles for every 10°C increase in temperature. At temperatures below about 10°C, bioactivity is slow. At temperatures below 0°C, bioactivity is near zero. The mean temperature of the biopile during operation is shown in Figure 11. Temperatures increased rapidly at the onset to around 45°C. Temperatures then fell rapidly through the first season. Temperatures did not increase much above ambient in the second season. Generally, the temperature of the biopile would be expected to reflect the ambient temperature, with some additional heating due to solar incidence. Additional increases in temperature, such as noted at the onset of the biopile operation, can occur as a result of the heat of metabolism.

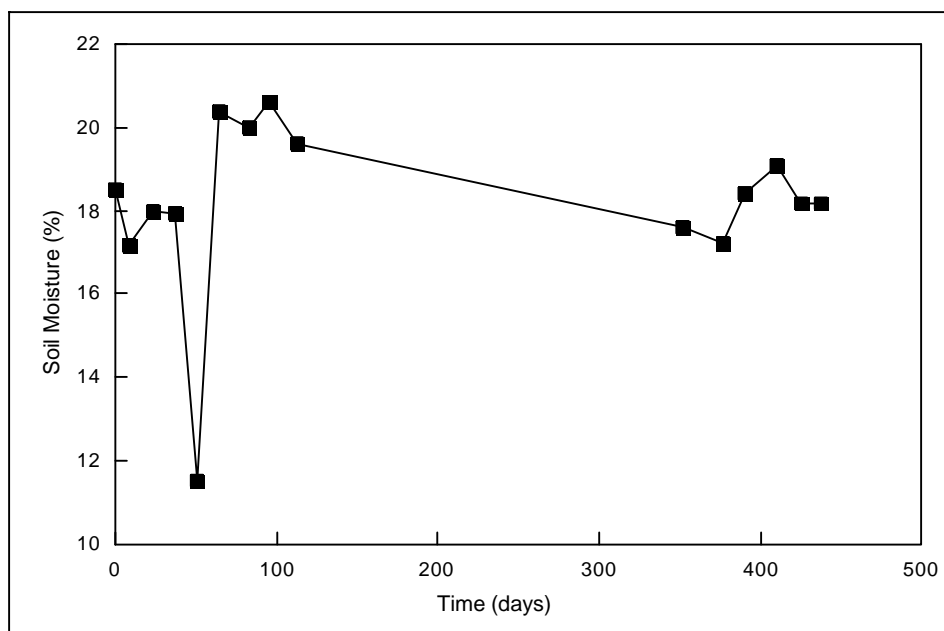


Figure 9. Soil moisture concentrations during the operation of the biopile.

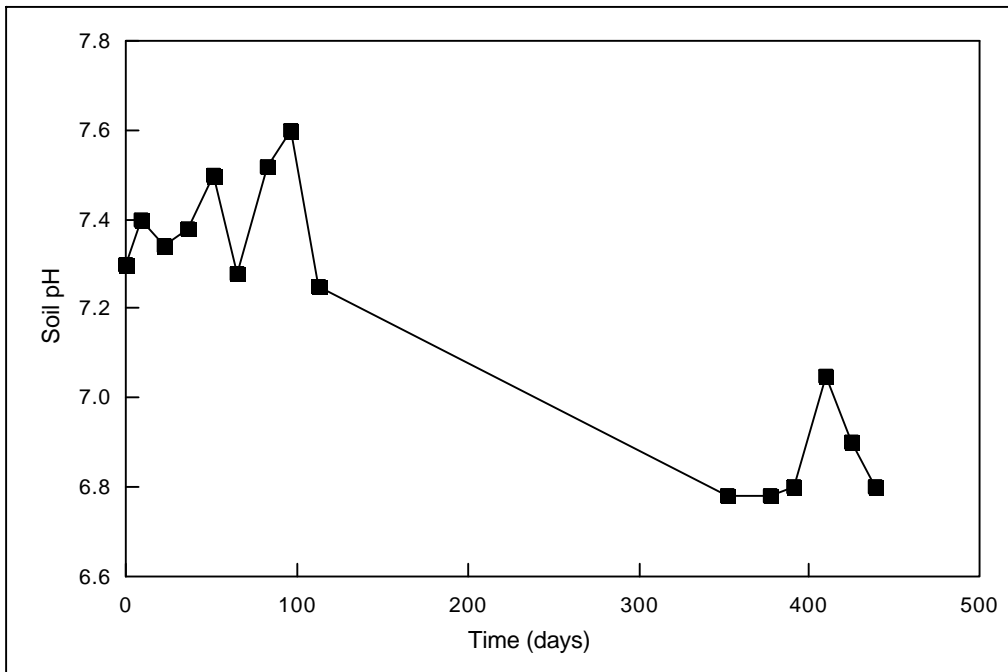


Figure 10. Variation in the soil pH during the operation of the biopile.

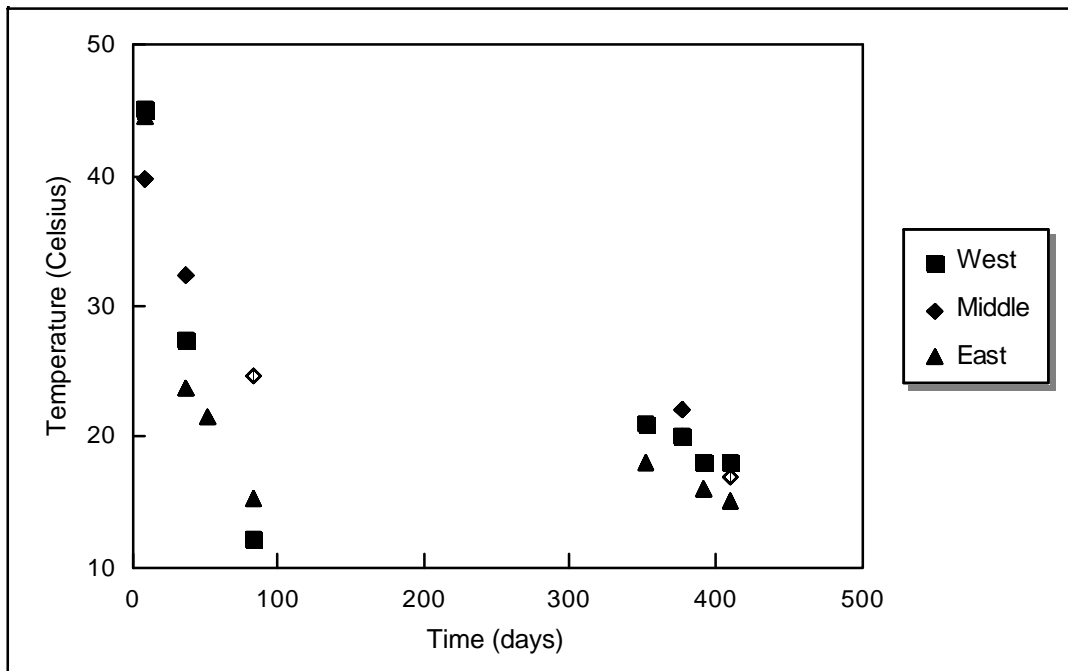


Figure 11. Variation in soil temperature during the operation of the biopile.

Analysis of amines and amine breakdown products was conducted at the EERC at four times throughout the operating period. The results of these analyses are summarized in Table 5. MEA, HEI, and OX are the principal amines in the soil. The concentration of these amines shows some surprising changes over time. For example, at the start of operations, the mean OX concentration was 4300 mg/kg, with a high of 6000 mg/kg. Four months later, two samples showed OX concentrations of 21,000 mg/kg. Most of these large changes in concentration are readily attributed to heterogeneity of the soil. In addition, some unusual results indicate the breakdown of larger, complex amine compounds to form identifiable amines. The final result of the amine analysis is that all identifiable amines were reduced to below the detection limit. Unfortunately, the detection limit for these compounds is from 100 to 200 mg/kg.

Tables 6 and 7 show the initial and final characterization of the soil treated in the biopile. Moisture remained about the same, EC increased by nearly four times, and pH dropped by about 0.5 units. The TKN dropped by 22% to a final concentration of 4880 mg/kg. Ammonia nitrogen increased by a small amount, while NO_x-N increased dramatically. Organic nitrogen dropped by 25% to a final concentration of 4330 mg/kg. Organic carbon as TOC, dropped by the largest amount of 38.1% to a final concentration of 17,700 mg/kg. Although the key parameters of contaminant measurement—TKN, organic nitrogen, and TOC—remain relatively high, this does not mean that successful remediation has not been achieved. Note that an uncontaminated soil may well have similar or higher values for these parameters. Additionally, the concentration of contaminant species that were monitored by these measures may have been completely removed. It is common for contaminant species to become incorporated into the humus material of soil. Generally, literature has shown that these contaminants incorporated into soil humus are not of further concern with respect to toxicity or mobility.

TABLE 5

Analysis of Amines and Amine Breakdown Products in Soil from the Biopile, mg/kg

Analyte	July 17, 1998	Nov 11, 1998	Sept 27, 1999	Oct 22, 1999
MEA	800	6700	190	BDL
HEI	10,000	BDL ¹	BDL	BDL
OX	4300	<100 to 21,000	100 to 200	BDL
HEED	trace	BDL	BDL	BDL
BHEED	trace	BDL	BDL	BDL
TEHEED	trace	BDL	BDL	BDL
Unknown B	10 to 100	BDL	BDL	BDL
Unknown C	10 to 100	BDL	BDL	BDL
Unknown D	100–1000	BDL	BDL	BDL

¹ BDL = below detection limit. The detection limit was from 100 to 200 mg/kg.

TABLE 6

Initial and Final pH, EC and Moisture Levels in the Biopile Soil,
plus or minus the standard deviation

	pH	Electrical Conductivity, dS/cm	Moisture, %
Initial	7.3	7.66 ± 1.57	18.5 ± 1.4
Final	6.8	30.5 ± 3.1	18.2 ± 0.8

TABLE 7

Initial and Final Concentrations of Key Analytes and Percent Removal Achieved,
plus or minus the standard deviation

	TKN	Ammonia-N	Organic Nitrogen	NO_x-N	TOC
Initial, g/kg	6250 ± 1120	480 ± 210	5770	130 ± 37	28,600 ± 6500
Final, mg/kg	4880 ± 910	550 ± 770	4330	2418 ± 490	17,700 ± 4500
% Removed	22.0	-14.6	25.0	-1760	38.1

Leachate was analyzed to estimate the amount of contaminants lost by that route. In fact, it proved difficult to make sense of the contaminant balance from the leachate. This is due to the fact that leachate analysis was not performed on each lot of leachate that was generated, leachate was not well mixed, and contaminants were further oxidized or reduced in the leachate collection system. For example, it was noted on one occasion early in the process that the leachate storage tank was effervescing vigorously. This was probably the result of denitrification of the nitrate in the leachate. Figure 12 shows the NO_x-N and ammonia-N in the leachate for some samples. The data show that ammonia was generally low, around 600 to 1000 mg/L. The leachate ammonia concentrations do not appear correlated with soil ammonia concentrations. The concentration of NO_x-N was low until early in the second season of treatment (around Day 390), when it increased rapidly to over 3000 mg/L. The increase in NO_x-N in the leachate correlates with the highest NO_x-N concentrations in the soil. This demonstrates that the nitrate is readily leachable.

The variation in conductivity of the leachate is shown in Figure 13. Conductivity was around 6000 to 14,000 μS/cm until early in the second season (around Day 390), when it rapidly increased to more than 20,000 μS/cm. The conductivity of the leachate generally follows that of the soil, but the relative increase in conductivity of the leachate is much larger than was noted in the soil. Additionally, the increase in leachate conductivity correlates well with the increases in NO_x-N. Again, these data suggest that NO_x-N is readily leachable and accounts for the major portion of the conductivity.

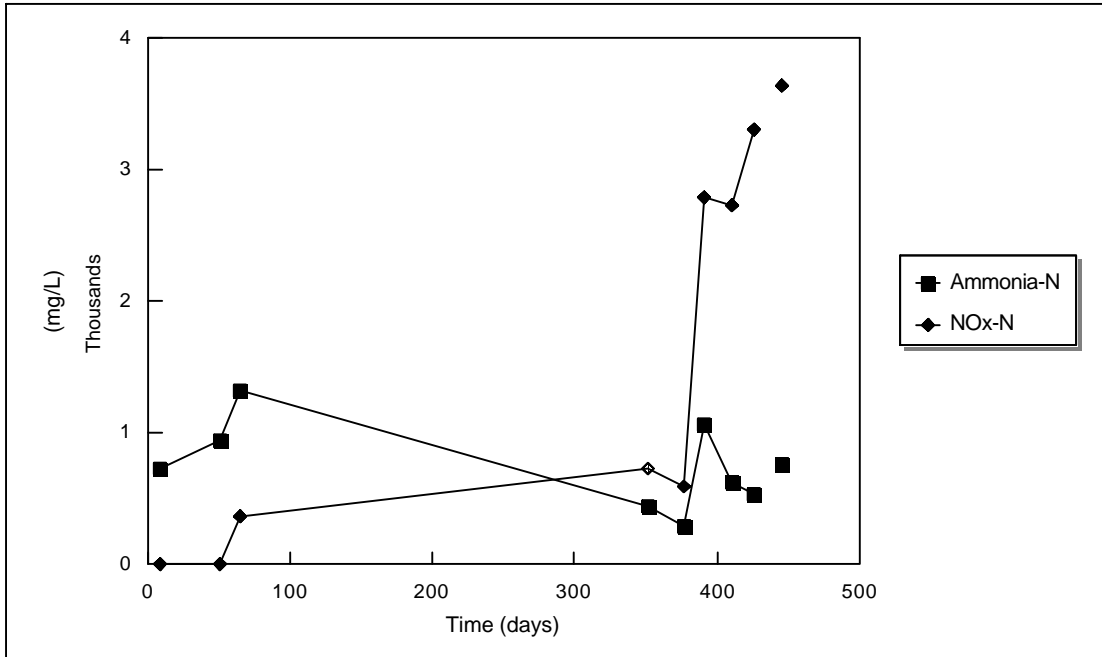


Figure 12. Concentrations of NO_x-N and ammonia-N in the leachate from the biopile.

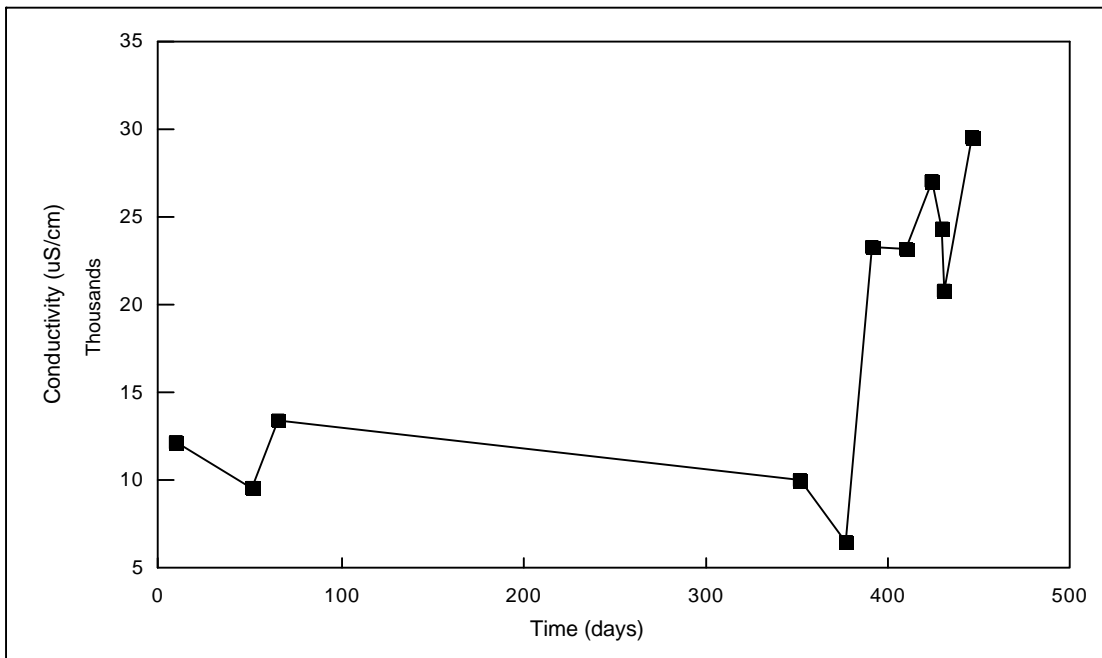


Figure 13. Variation in conductivity of the leachate from the biopile.

The pH of the leachate throughout the biopile treatment is shown in Figure 14. The pH of the leachate was initially higher than the corresponding soil pH. The leachate pH fell throughout the operating period to about 6.9. The higher initial pH probably reflects the presence of ammonia in a poorly buffered water. Later in the operating period, the leachate pH fell as a result of the slight acidification of the soil.

Although the biopile was lined to prevent leaching of contaminants to the groundwater, it was noted that an existing well was located immediately adjacent to the biopile. This well, designated 95-44A, was sampled quarterly by Komex as a part of the site-monitoring plan. A review of the water quality data available on this well, which included EC, pH, DKN (dissolved Kjeldahl nitrogen), chloride, DOC (dissolved organic carbon), and total dissolved solids (TDS, calculated), shows that the biopile did not impact the groundwater. The well completion and sampling data are located in Appendix F.

CONCLUSIONS

Analytical results indicate that soil toxicity was reduced from project initiation (July 1998) to the close of Year 1 operations (November 1998.) IC25 and IC50 values increased markedly in every case. This indicates reduced toxicity because higher concentrations of the aqueous extract are necessary to inhibit growth percentages. Toxicity testing was not performed on soil from the 1999 operating season because it would be impossible to separate the toxic effects of the salts from other related toxicity. High soil conductivity indicated that toxicity had likely increased over the 1999 season, although the leaching process had not been fully utilized to remove the water-extractable byproducts of bioactivity.

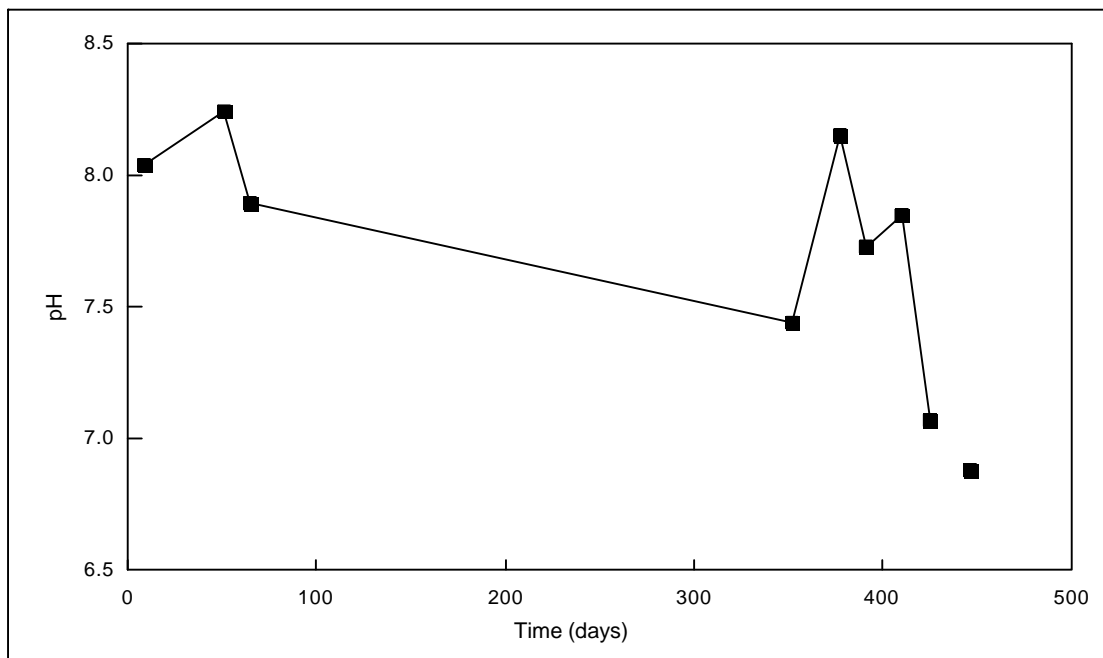


Figure 14. Variation in the pH of the leachate from the biopile.

Soil testing data show that amine compounds as measured by organic nitrogen are decreasing, while $\text{NO}_x\text{-N}$ is increasing with time. This general pattern is expected during biodegradation of the nitrogenous organic compounds (i.e., amines and T/O reaction products). Some additional biodegradation of organic matter in this soil can be expected to occur unaided. This may result in a small increase in salinity over time.

Analysis of specific amine compounds in the biopile soil showed that all identifiable amines have been removed to below detection limits. It is recommended that the biopile soil be leached to achieve an EC of less than 6 dS/cm. Once that EC value has been achieved, an additional round of toxicity testing should be performed to verify that the toxic components have been removed.

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NORWEST ANALYTICAL SOIL DATA

APPENDIX A

**Soil Analysis Report
17 Jul 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/m)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN ¹ (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Grid A - Upper	7.3	5.6	5610	3.62	18.1	497	124	-	-	-
60+cm Grid A - Lower	7.3	7.2	7200	2.41	19.6	238	218	-	-	-
0-60cm Grid C - Upper	7.0	10.3	10300	2.41	17.1	218	110	-	-	-
60+cm Grid C - Lower	7.2	6.9	6910	2.03	18.0	528	73	-	-	-
0-60cm Grid H - Upper	7.4	7.6	7620	3.38	20.9	719	166	-	-	-
60+cm Grid H - Lower	7.3	8.4	8350	3.28	17.5	680	149	-	-	-
Mean²	7.2	7.1	7665	2.86	18.5	480	140	-	-	-
Standard Deviation	0.1	0.9	1439	0.59	1.3	194	46	-	-	-

¹ TKN test omitted by Norwest Labs.

² mean pH is calculated using the geometric mean.

**Soil Analysis Report
14 Aug 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot D	7.7	10.5	10500	2.94	14.6	1720	101	7100	5380	7201
60-100cm Plot D	7.6	9.7	9650	2.29	19.7	1290	26	7800	6510	7826
0-60cm Plot B	7.6	15.8	15800	2.22	14.5	2360	33	6100	3740	6133
60-100cm Plot B	6.8	17.1	17100	2.20	24.6	1610	8	6600	4990	6608
0-60cm Plot G	7.5	13.3	13300	2.70	14.4	2080	80	6700	4620	6780
60-100cm Plot G	7.3	12.3	12300	2.84	15.3	1860	43	7500	5640	7543
Mean	7.41	13.1	13108.33	2.53	17.2	1820	49	6967	5147	7015
Standard Deviation	0.30	2.7	2665.59	0.30	3.8	341	32	571	860	573

**Soil Analysis Report
28 Aug 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot A	7.7	12.1	12100	2.14	16.4	2840	125	6400	3560	6525
60-100cm Plot A	7.5	7.3	7330	2.55	19.1	3280	15	6200	2920	6215
0-60cm Plot E	7.5	13.1	13100	3.10	16.9	1700	104	8500	6800	8604
60-100cm Plot E	6.9	10.4	10400	2.25	18.5	2930	22	7700	4770	7722
0-60cm Plot F	7.4	14.4	14400	2.80	19.4	3350	25	8100	4750	8125
60-100cm Plot F	7.1	14.8	14800	2.61	17.6	3100	6	7800	4700	7806
Mean	7.35	12.0	12021.67	2.58	18.0	2867	49	7450	4583	7499
Standard Deviation	0.27	2.6	2555.58	0.32	1.1	551	47	854	1211	852

**Soil Analysis Report
11 Sep 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot B	7.4	17.6	17600	1.88	17.7	277	305	5700	5423	6005
60-100cm Plot B	7.4	13.8	13800	2.40	18.4	216	63	6200	5984	6263
0-60cm Plot C	7.3	14.2	14200	1.93	17.2	195	355	5700	5505	6055
60-100cm Plot C	7.4	13.0	13000	2.08	18.0	213	56	5900	5687	5956
0-60cm Plot H	7.5	12.0	12000	2.66	17.9	220	93	7100	6880	7193
60-100cm Plot H	7.3	9.6	9550	2.76	18.5	133	32	7000	6867	7032
Mean	7.38	13.4	13358.33	2.29	18.0	209	151	6267	6058	6417
Standard Deviation	0.07	2.4	2428.03	0.34	0.4	42	129	579	603	503

**Soil Analysis Report
25 Sep 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot E	7.3	17.9	17900	2.26	16.5	1280	402	7000	5720	7402
60-100cm Plot E	7.2	15.7	15700	2.96	7.4	1480	61	7600	6120	7661
0-60cm Plot D	7.3	16.3	16300	-	16.5	207	978	3100	2893	4078
60-100cm Plot D	7.5	14.0	14000	2.01	14.7	952	634	4800	3848	5434
0-60cm Plot F	7.8	8.9	8870	1.75	9.8	2170	8	4900	2730	4908
60-100cm Plot F	7.7	10.9	10900	1.92	11.6	2360	35	5200	2840	5235
Mean	7.46	13.9	13945.00	2.18	12.8	1408	353	5433	4025	5786
Standard Deviation	0.22	3.1	3143.74	0.42	3.4	726	360	1491	1394	1306

**Soil Analysis Report
09 Oct 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot A	7.2	14.4	14400	1.71	17.0	1660	666	4600	2940	5266
60-100cm Plot A	7.1	14.2	14200	1.45	17.6	983	634	4800	3817	5434
0-60cm Plot C	7.0	18.1	18100	2.08	19.8	2540	1370	5800	3260	7170
60-100cm Plot C	7.6	9.8	9840	1.87	17.8	2370	43	4900	2530	4943
0-60cm Plot F	7.4	6.9	6870	1.64	25.0	3110	237	3800	690	4037
60-100cm Plot F	7.4	7.4	7350	2.00	25.1	3130	16	4800	1670	4816
Mean	7.28	11.8	11793.33	1.79	20.4	2299	494	4783	2485	5278
Standard Deviation	0.20	4.1	4085.72	0.22	3.4	769	468	584	1038	955

**Soil Analysis Report
27 Oct 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot B	7.6	12.0	12000	1.89	21.1	2730	281	5900	3170	6181
60-100cm Plot B	7.6	11.8	11800	1.72	20.1	2770	87	5300	2530	5387
0-60cm Plot D	7.3	17.7	17700	1.55	16.8	2220	857	5300	3080	6157
60-100cm Plot D	7.4	14.9	14900	2.36	19.3	2320	154	7400	5080	7554
0-60cm Plot H	7.5	14.1	14100	2.45	21.7	1900	394	6900	5000	7294
60-100cm Plot H	7.7	10.8	10800	2.49	20.9	2470	75	7300	4830	7375
Mean	7.52	13.6	13550.00	2.08	20.0	2402	308	6350	3948	6658
Standard Deviation	0.13	2.3	2322.89	0.37	1.6	300	270	886	1044	797

**Soil Analysis Report
09 Nov 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot H	7.6	12.0	12000	1.89	21.1	2730	281	7800	5070	8081
60-100cm Plot H	7.6	11.8	11800	1.72	20.1	2770	87	6700	3930	6787
Mean	7.60	11.9	11900.00	1.81	20.6	2750	184	7250	4500	7434
Standard Deviation	0.00	0.1	100.00	0.09	0.5	20	97	550	570	647

**Soil Analysis Report
26 Nov 98**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot A	7.3	17.1	17100	0.72	19.8	283	1100	4000	3717	5100
60-100cm Plot A	7.0	20.3	20300	2.58	20.5	5	1460	4000	3995	5460
0-60cm Plot E	7.2	12.7	12700	1.53	18.4	3	888	3300	3297	4188
60-100cm Plot E	7.1	19.9	19900	1.64	18.4	1460	1350	4300	2840	5650
0-60cm Plot G	7.4	12.1	12100	1.99	22.7	3290	144	6400	3110	6544
60-100cm Plot G	7.5	15.1	15100	2.52	17.6	3040	75	7200	4160	7275
Mean	7.25	16.2	16200.00	1.83	19.6	1347	836	4867	3520	5703
Standard Deviation	0.17	3.2	3203.64	0.64	1.7	1378	545	1419	475	992

**Soil Analysis Report
23 July 99**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot B	6.8	26.0	26000	1.19	16.3	1560	1710	5600	4040	7310
60-100cm Plot B	6.7	29.1	29100	1.53	17.0	1220	1960	5300	4080	7260
0-60cm Plot D	6.7	28.8	28800	1.84	15.4	2060	2090	6900	4840	8990
60-100cm Plot D	6.9	25.1	25100	2.98	22.1	2630	1730	6600	3970	8330
0-60cm Plot G	6.7	26.7	26700	2.11	18.0	1990	1890	7000	5010	8890
60-100cm Plot G	6.9	20.0	20000	2.48	16.6	2540	619	7100	4560	7719
Mean	6.78	26.0	25950.00	2.02	17.6	2000	1667	6417	4417	8083
Standard Deviation	0.09	3.0	3020.35	0.59	2.2	499	486	706	410	700

**Soil Analysis Report
17 Aug 99**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot A	6.7	25.4	25400	1.38	16.3	318	2470	6600	6282	9070
60-100cm Plot A	6.6	27.6	27600	1.83	18.9	198	2800	2700	2502	5500
0-60cm Plot E	6.8	27.5	27500	1.66	15.9	438	2830	3300	2862	6130
60-100cm Plot E	6.8	28.6	28600	1.36	17.5	192	2750	2000	1808	4750
0-60cm Plot F	6.9	29.0	29000	2.09	16.8	1410	2290	1600	190	3890
60-100cm Plot F	6.9	26.0	26000	1.92	17.9	1590	1970	6100	4510	8070
Mean	6.78	27.4	27350.00	1.71	17.2	691	2518	3717	3026	6235
Standard Deviation	0.11	1.3	1290.67	0.27	1.0	580	312	1942	1942	1810

**Soil Analysis Report
08 Sep 99**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0-60cm Plot E	6.7	30.9	30900	1.67	17.6	20	2520	3900	3880	6420
60-100cm Plot E	6.7	32.8	32800	1.78	16.5	164	2720	4400	4236	7120
0-60cm Plot D	6.6	35.2	35200	5.04	19.0	186	3080	5200	5014	8280
60-100cm Plot D	7	27.0	27000	2.56	20.6	1290	1410	3900	2610	5310
0-60cm Plot G	6.8	32.5	32500	2.52	18.3	802	2010	6700	5898	8710
60-100cm Plot G	7	26.9	26900	1.74	18.3	1800	1460	6800	5000	8260
Mean	6.80	30.9	30883.33	2.55	18.4	710	2200	5150	4440	7350
Standard Deviation	0.15	3.1	3051.46	1.17	1.3	656	626	1212	1039	1199

**Soil Analysis Report
27 Sep 99**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0–60cm Plot F	7.3	30.6	30600	1.61	23.7	441	2160	5600	5159	7760
60–100cm Plot F	7.1	36.3	36300	1.89	20.2	892	2460	5400	4508	7860
0–60cm Plot A	6.9	43.2	43200	1.63	17.4	316	3190	4500	4184	7690
60–100cm Plot A	7	33.8	33800	1.26	17.2	160	2340	3800	3640	6140
0–60cm Plot C	7.1	31.9	31900	1.37	17.9	389	2280	4000	3611	6280
60–100cm Plot C	6.9	32.8	32800	1.41	18.3	89	2420	3500	3412	5920
Mean	7.05	34.8	34766.67	1.53	19.1	381	2475	4467	4086	6942
Standard Deviation	0.14	4.2	4160.40	0.21	2.3	259	334	791	608	836

**Soil Analysis Report
12 Oct 99**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN ¹ (mg/kg)	ON (mg/kg)	TN (mg/kg)
0–60cm Plot B	6.5	27.4	27400	0.95	19.6	48	2140	–	–	–
60–100cm Plot B	6.8	26.2	26200	1.67	18.3	794	1590	–	–	–
0–60cm Plot D	7.0	20.1	20100	2.68	17.0	1430	941	–	–	–
60–100cm Plot D	7.2	20.2	20200	2.28	17.1	2260	722	–	–	–
0–60cm Plot H	7	24.1	24100	2.45	18.9	1600	1430	–	–	–
60–100cm Plot H	7	27.4	27400	2.69	18.2	1960	1400	–	–	–
Mean	6.91	24.2	24233.33	2.12	18.2	1349	1371	–	–	–
Standard Deviation	0.22	3.1	3090.13	0.63	0.9	739	456	–	–	–

¹ TKN test omitted by Norwest Labs.

**Soil Analysis Report
25 Oct 99**

Sample	pH	Conductivity (dS/m)	Conductivity (µS/cm)	TOC (%)	Moist.Wet Wt. (%)	Ammonium - N (mg/kg)	Nitrate and Nitrite (mg/kg)	TKN (mg/kg)	ON (mg/kg)	TN (mg/kg)
0–60cm Plot A	6.6	32.1	32100	1.38	19.0	21	2570	4100	4079	6670
60–100cm Plot A	6.5	30.2	30200	1.40	19.5	6	2540	4200	4194	6740
0–60cm Plot E	6.6	30.6	30600	2.16	17.4	111	2530	4200	4089	6730
60–100cm Plot E	6.8	33.1	33100	1.13	17.8	150	2660	4500	4350	7160
0–60cm Plot G	7	33.1	33100	2.33	17.2	920	2860	8000	7080	10860
60–100cm Plot G	7.3	24.2	24200	2.01	18.0	2120	1350	6300	4180	7650
Mean	6.79	30.6	30550.00	1.74	18.2	555	2418	5217	4662	7635
Standard Deviation	0.28	3.1	3051.09	0.45	0.8	767	491	1458	1085	1482

NORWEST ANALYTICAL LEACHATE DATA

APPENDIX B

Date	8/14/98	9/25/98	10/9/98	7/23/99	8/9/99	9/8/99	9/27/99	10/12/99	10/25/99
pH	8.04	8.24	7.89	7.44	8.15	7.73	7.85	7.07	6.88
Conductivity ($\mu\text{S/cm}$)	12200	19610	13400	9900	6500	23300	23200	27000	29500
TKN (mg/L)	3160	2160	1960	*	502	11.5	297	478	455
NO₄-N (mg/L)	721	942	1320	429	292	1060	611	533	756
NO_x-N (mg/L)	1.11	0.38	355	730	594	2780	2730	3300	3630
NO (mg/L)	2439	1218	640	*	210	-1048.5	-314	-55	-301
COD (mg/L)	122	7750	6260	2090	1320	4880	4580	3700	392

* Test omitted by Norwest Labs

**NORWEST STANDARD METHODS REFERENCE
DATA**

APPENDIX C

Parameter	Reference	Method Number
pH	McKeague	3.14
Sodium Adsorption Ratio	McKeague	3.26
Ammonium - N	McKeague	4.35
Ammonia - N	McKeague	4.35
Nitrate & Nitrite	McKeague	4.311
TKN	McKeague	3.62
Saturation %	McKeague	2.41
Moist Wet Wt. %	McKeague	2.41
Particle Size Distribution	Carter	47.3
Bulk Density	McKeague	2.2
Oil, Dean Stark	Alberta	29.7
Calcium	McKeague	3.26
Magnesium	McKeague	3.26
Sodium	McKeague	3.26
Theo Gypsum Req	Ashworth	–
Conductivity	EPA	3202
Total Organic Carbon	EPA	3280
Chloride	APHA	4500
Phosphate-P	APHA	4500
Sulfate-S	EPA	3207
Arsenic	EPA	3051
Barium	EPA	3051
Beryllium	EPA	3051
Cadmium	EPA	3051
Chromium	EPA	3051
Cobalt	EPA	3051
Copper	EPA	3051
Lead	EPA	3051
Mercury	EPA	3051
Molybdenum	EPA	3051
Nickel	EPA	3051
Potassium	EPA	6010
Selenium	EPA	3051
Thallium	EPA	3051
Vanadium	EPA	3051
Zinc	EPA	3051

1. Alberta, G-58 Oilfield Waste Management Requirements, Oil, Dean Stark, Part F, 19.7.
2. APHA (American Public Health Authority), 1998, Standard methods for the examination of water and waste water, 20th edition.
3. Ashworth, Keyes, Crepin, Canada Journal of Soil Science, theo gypsum req, 79, pp. 449–455.
4. Carter, M.R., 1993, Soil sampling and methods of analysis, Canadian Society of Soil Science.
5. McKeague, J.A., 1978, Manual on soil sampling and methods of analysis, Canadian Society of Soil Science.
6. EPA – United States Environmental Protection Agency
 - A. Test Methods for Evaluating Solid Waste, 1986, Physical and Chemical Methods SW-846, 3rd edition.
 - B. Methods for Chemical Analysis of Water and Wastewater, 1983.

HYDROQUAL TOXICITY DATA

APPENDIX D



HydroQual
Laboratories Ltd.

#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 98759
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CLIENT INFORMATION

Client: Energy and Environmental Research Centre
 Operation: University of North Dakota, Grand Forks
 Address: 15 North 23rd Street, PO Box 9018
 City: Grand Forks
 Province/State: North Dakota
 Country: USA
 Postal/ZIP Code: 58203
 Billing Information:
 Contact: Jim Sorenson
 Tel: 701-777-5000
 Fax: 701-777-5181

SAMPLE INFORMATION

Sample Type: Amine Biopile Composite
 Collected On: 98/07/17 At: not given
 Collected By: not given
 Shipped On: 98/07/17
 Shipped By: dropped off Prepaid: Collect:
 Received On: 98/07/17 At: 1200
 Received By: P. Reimer
 Container: 1 plastic bag
 Seals: none
 Initials on Seals: not applicable
 Frozen: no

INITIAL CHEMISTRY

pH (units)	not done	ammonium (mg-N/L)	not done
Conductance (uS/cm)	not done	residual chlorine (mg/L)	not done
Dissolved Oxygen (mg/L)	not done	Colour:	black
Temperature (°C)	not done	Odour:	organic
Alkalinity (mg-CaCO3/L)	not done		
Hardness (mg-CaCO3/L)	not done		

COMMENTS

SAMPLE HISTORY

Storage Conditions: 4 ± 2°C
 Disposed On: n/a by n/a Method: n/a

TEST LOG

Test Type	BL(S)	RE(D)	EW(D)	SE			
Number	981535	981539	981540	981538			
Started	98/07/22	98/07/31	98/07/31	98/07/31			
Ended	98/07/22	98/08/04	98/08/14	98/08/07			
Reported	98/08/06	98/08/06	98/08/17	98/08/04			
Faxed	98/07/28						

NOTES: TR, trout; FM, fathead minnows; DA, Daphnia; CD, Ceriodaphnia; AG, Selenastrum;

BL, bacterial luminescence; D, definitive test; S, screening test; n/a, not applicable

REVISED BY CG ON 96/04/04

FILE: REP-01.XLS

WRITTEN BY SG ON 95/05/12



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 98759	Test: 981535
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Test Method: Bacterial Luminescence Test (screening test, one treatment level plus a control)
Reference: Biological Test Method: Toxicity Test Using Luminescent Bacteria (*Vibrio fischeri*), 1992. Environment Canada, EPS 1/RM/24.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

Sample Information:

Description: Amine Biopile Composite
 Collected On: 98/07/17 At: not given By: not given
 Received On: 98/07/17 At: 1200 By: P. Reimer

Test Information:

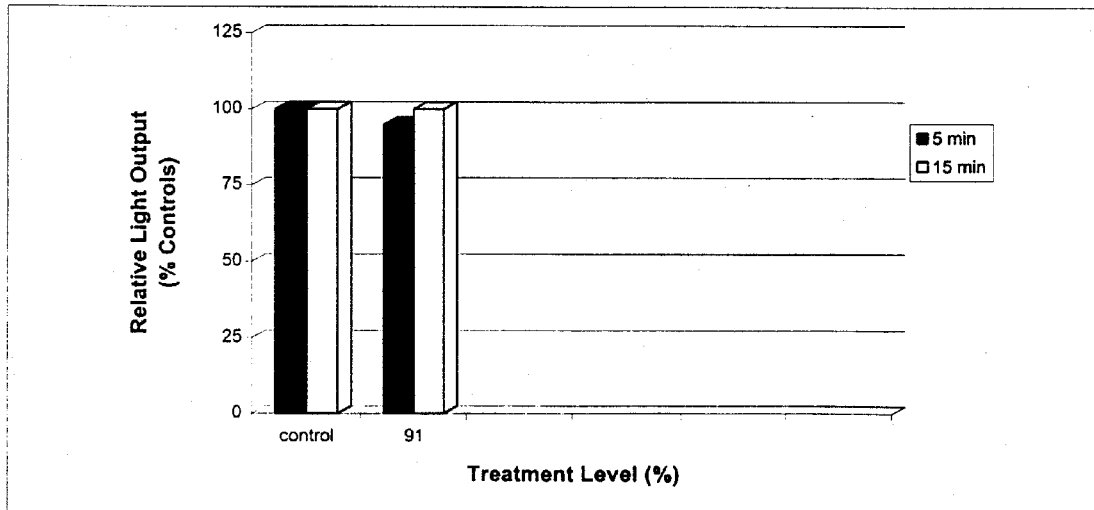
Started On: 98/07/22 At: PM By: JFH
 Ended On: 98/07/22 At: PM By: JFH
 Reported On: 98/08/06 By: CG

Test Result:

Aqueous Extract: 100% expressed as a percentage of the control.

Methanol Extract: 64% expressed as a percentage of the control.

Graph of Light Output Relative to Controls at 5 and 15 Minutes





HydroQual
Laboratories Ltd.

#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

TEST DATA

Client: 98025 Sample: 98759 Test: 981535

SAMPLE PRETREATMENT

pH adjustment	not required
preaeration	not required
turbidity	centrifuged for 10 minutes
other	none

4:1 Aqueous Extract

LIGHT READINGS

Treatment Level (%)	Time (min)		
	0	5	15
control	95	97	94
91	86	92	94

INHIBITION (%CTLS)

5 min	15 min
100	100
95	100

4:1 Methanol Extract (5%)

LIGHT READINGS

Treatment Level (%)	Time (min)		
	0	5	15
control	94	96	96
91	71	58	61

INHIBITION (%CTLS)

5 min	15 min
100	100
60	64

COMMENTS

All criteria have been met for a valid test and the test data and results are verified correct.

Signature 98108/20



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

QUALITY ASSURANCE INFORMATION

Test Method: Bacterial luminescence Test (IC_{50} , four or more treatments plus a control)
Reference: Biological Test Method: Toxicity Test Using Luminescent Bacteria (*Vibrio fischeri*), 1992. Environment Canada, EPS 1/RM/24.

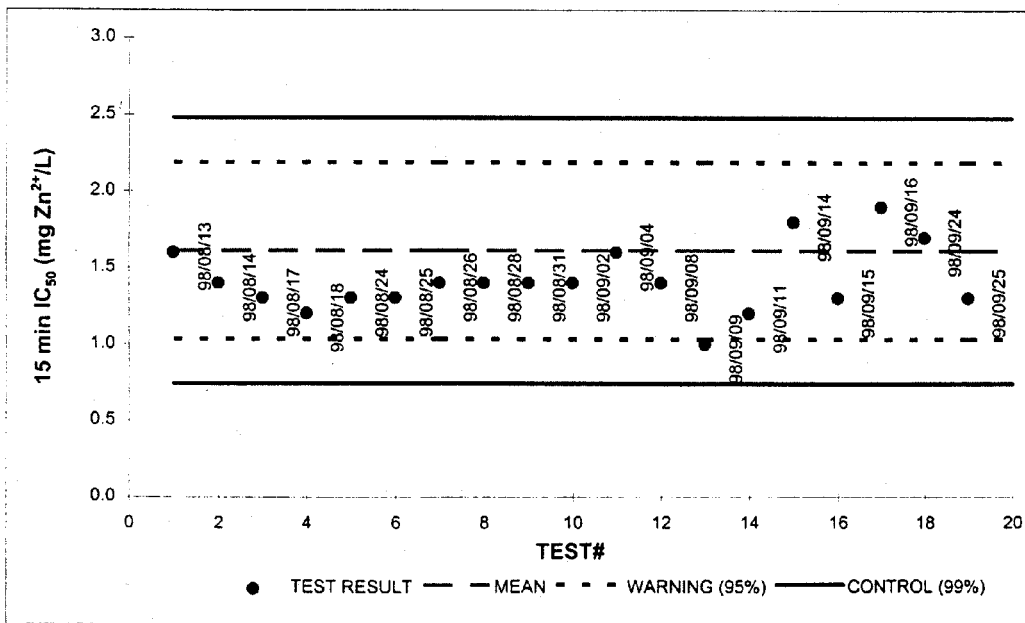
TEST ORGANISM		TEST DESIGN	
test species	<i>Vibrio fischeri</i>	storage temp.	-20 to -25°C
batch number	ACV012-3	analyzer	Model 500
date obtained	98/07/07	test temperature	15°C
expires	99/07/01		

NOTE: NEW REAGENT LOT 98/07/09

Quality Assurance Information:
 All criteria have been met for a valid test and the test data and result are verified correct.

WARNING CHART

TOXICANT: zinc ($ZnSO_4 \cdot 7H_2O$)
CURRENT TEST: started: 98/09/25 ended: 98/09/25
RESULT: 1.3 (0.76-2.2) mg Zn^{2+} /L 95% confidence limits are in brackets
HISTORICAL MEAN: 1.6 std.dev: 0.3 CV(%): 17.9
CHART LIMITS: warning: 1.0 2.2 control: 0.7 2.5
 95% , two standard deviations 99% , three standard deviations





#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942
 Client: 98025 Sample: 98759 Test: 981539

Test Method: 120h Root Elongation Test, Lettuce (*Lactuca sativa*)

Reference: Greene et al., 1989. Protocols for Short Term Toxicity Screening of Hazardous Waste Sites. EPA 600/3-88-029.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

Sample Information:

Sample: Amine Biopile Composite

Collected On: 98/07/17 At: not given By: not given
 Received On: 98/07/17 At: 1200 By: P. Reimer

Test Information:

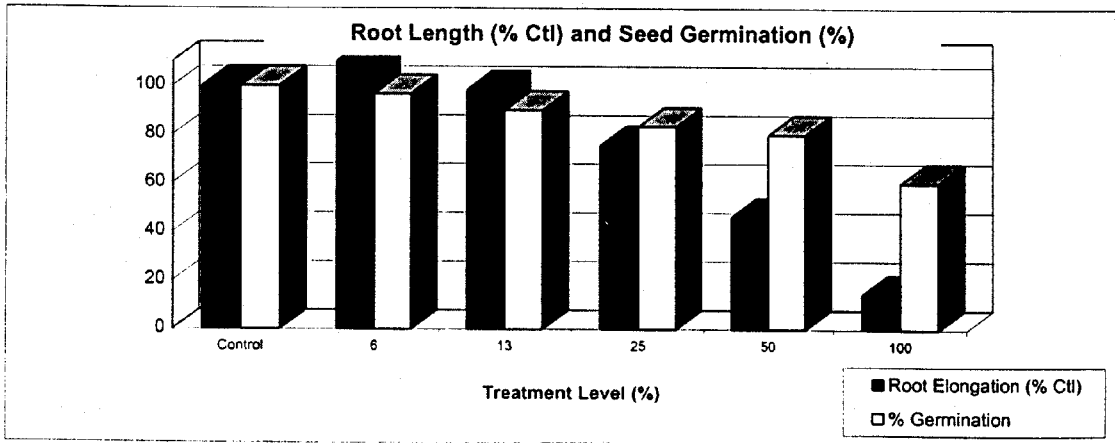
Started On: 98/07/31 At: PM By: JH
 Ended On: 98/08/04 At: PM By: JH
 Reported On: 98/08/06 By: CG

Root Elongation

Test Result:	Value	Confidence Limits		Units	Method Calculated
IC25	22	9	30	%	ICPIN
IC50	43	28	69	%	ICPIN
NOEC	12			%	Estimated
LOEC	25			%	Estimated

Seed Germination

Test Result:	Value	Confidence Limits		Units	Method Calculated
IC25	62	0	>100	%	ICPIN
IC50	>100			%	ICPIN
NOEC	50			%	Estimated
LOEC	100			%	Estimated



Notes: IC25 & IC50, concentrations inhibiting root elongation or germination by 25 and 50%; NOEC & LOEC, no observed and lowest observed effect concentrations



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942
 Client: 98025 Sample: 98759 Test: 981539

ROOT LENGTHS (cm)

Treatment (%)	REPLICATE										AVG	STDEV	Percent (%) Germination
	1	2	3	4	5	6	7	8	9	10			

Date:	97/08/18		Time:	PM		Initials:	MB/JS		Temp (oC):					
Control	3.5	4.0	2.0	5.0	3.5	5.5	3.5	3.0	3.5	0.0	3.4	1.5	100	
	3.5	3.5	3.0	4.0	3.0	2.0	5.0	5.0	1.0	2.5	3.3	1.3	100	
	2.5	4.0	3.0	3.0	1.5	3.0	3.0	2.5	5.0	5.0	3.3	1.1	100	
6	2.5	4.0	2.5	3.0	3.0	4.0	3.0	4.0	4.0		3.3	0.7	90	
	4.3	5.0	3.5	2.0	2.5	3.5	2.5	4.0	2.0	4.0	3.3	1.0	100	
	5.5	4.0	4.0	5.5	6.0	4.0	5.5	4.5	5.0	5.0	4.9	0.7	100	
12	2.0	3.0	2.0	3.0	3.0	5.0	2.0	2.5	2.5	4.5	3.0	1.0	100	
	1.5	2.5	3.5	3.0	5.5	2.0	5.0	3.5	2.0		3.2	1.4	90	
	3.0	3.0	4.0	4.0	5.0	4.5	2.0	3.0			3.6	1.0	80	
25	2.0	3.0	2.5	2.0	3.5	2.0	3.0				2.6	0.6	70	
	2.0	3.5	3.0	2.0	2.0	2.5	1.0	3.0			2.4	0.8	80	
	3.0	2.5	2.0	3.0	3.5	3.0	1.5	2.0	3.0	1.5	2.5	0.7	100	
50	1.5	2.5	2.0	2.0	1.5	2.0	1.0	1.0	1.0	1.5	1.6	0.5	100	
	2.5	3.0	2.0	1.0	2.0	2.0	1.5	1.5			1.9	0.6	80	
	1.5	0.5	1.0	1.0	1.0	1.0					1.0	0.3	60	
100	0.5	0.5	0.5	0.5	0.5	0.5	0.5				0.5	0.0	70	
	0.5	0.5	0.5	0.5							0.5	0.0	40	
	0.5	0.5	0.5	0.5	0.5	0.5	0.0				0.4	0.2	70	

COMMENTS:
 Aqueous extracts prepared using 20 g soil in 80 g DRO. Four mL of the appropriate stock solution was added to a filter paper placed in a plastic petri dish (100 mm), and 10 lettuce seeds were added to each plate. Test incubation was 120 h in the dark at 24 ± 2°C.

All criteria have been met for a valid test and the test data and results are verified correct.

Alampy 98/08/25



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 98759	Test: 981538
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Test Method: 120 h Lettuce Seedling Emergence Screening Test (*Lactuca sativa*)
Reference: Greene et al. 1989. Protocols for Short Term Toxicity Screening of Hazardous Waste Sites. EPA 600/3-88-029.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

Sample Information:

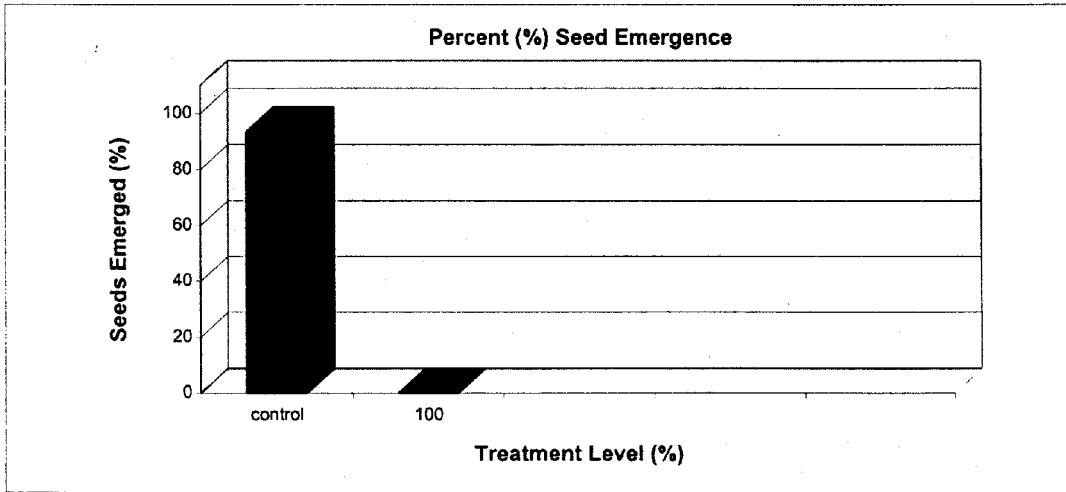
Description: Amine Biopile Composite
 Collected On: 98/07/17 At: not given By: not given

Test Information:

Started On: 98/07/19 At: PM By: JS
 Ended On: 98/07/26 At: PM By: JS
 Reported On: 98/08/17 By: CG

Test Result:

Treatment	Percent (%) Seed Emergence	Comment
control	93	
undiluted sample	0	



Notes: IC25 & IC50, concentrations inhibiting seed emergence by 25 and 50%; NOEC & LOEC, no observed and lowest observed effect concentrations



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

TEST DATA

Client: 98025 Sample: 98759 Test: 981538

# Seeds Emerged at 120 h			
(%)	A	B	C
control	19	18	19
100	0	0	0

% of Seed Emergence				
A	B	C	Avg	StDev
95	90	95	93	3
0	0	0	0	0

COMMENTS
 Dilutions were made using test substance and artificial soil. 30 g of test mixture at appropriate concentration was placed in a 100 x 15 mm plastic petri dish. Twenty lettuce seeds were placed on the soil surface then capped with 30 g sand. Test soils were hydrated with 15 mL of DRO. Test incubation was 120 h at 27 ± 2°C. The photoperiod was 48h dark 18 h light:6 h dark.

All criteria have been met for a valid test and the test data and results are verified correct.

Signature 98108/25



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 98759	Test: 981538
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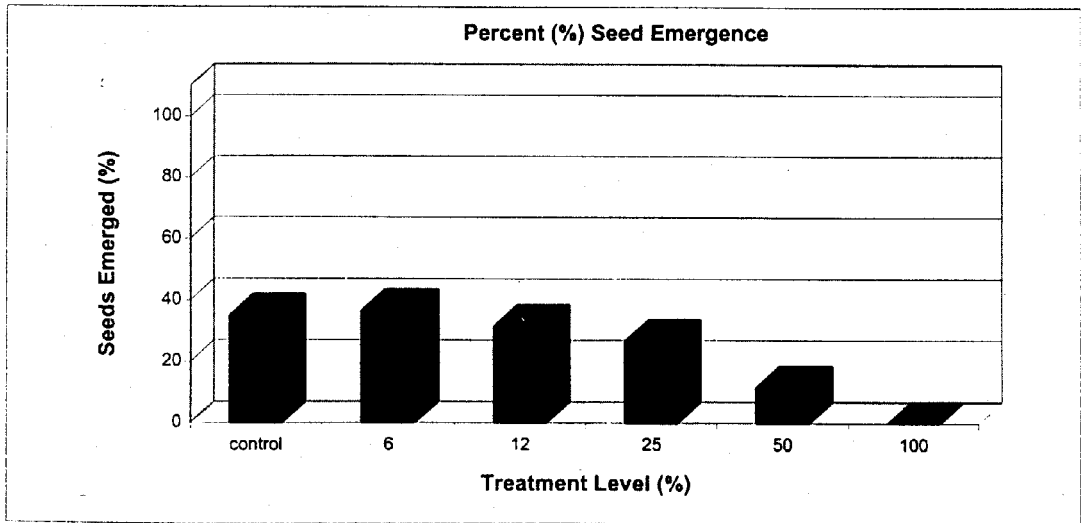
Test Method: 120 h Lettuce Seedling Emergence Test (*Lactuca sativa*)
Reference: Greene et al. 1989. Protocols for Short Term Toxicity Screening of Hazardous Waste Sites. EPA 600/3-88-029.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

Sample Information:
 Description: Amine Biopile Composite
 Collected On: 98/07/17 At: not given By: not given

Test Information:
 Started On: 98/07/31 At: PM By: JS
 Ended On: 98/08/07 At: PM By: JS
 Reported On: 98/08/04 By: CG

Test Result:	Value	Confidence Limits	Units	Method Calculated
IC25	24	16 32	%	ICPIN
IC50	40	33 55	%	ICPIN
NOEC	12		%	William's
LOEC	25		%	William's



Notes: IC25 & IC50, concentrations inhibiting seed emergence by 25 and 50%; NOEC & LOEC, no observed and lowest observed effect concentrations



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

TEST DATA

Client: 98025 Sample: 98759 Test: 981538

# Seeds Emerged at 120 h			
(%)	A	B	C
control	6	7	8
6	7	7	8
12	7	6	6
25	4	5	7
50	2	1	4
100	0	0	0

% of Seed Emergence				
A	B	C	Avg	StDev
30	35	40	35	5
35	35	40	37	3
35	30	30	32	3
20	25	35	27	8
10	5	20	12	8
0	0	0	0	0

COMMENTS
 Dilutions were made using test substance and artificial soil. 30 g of test mixture at appropriate concentration was placed in a 100 x 15 mm plastic petri dish. Twenty lettuce seeds were placed on the soil surface then capped with 30 g sand. Test soils were hydrated with 15 mL of DRO. Test incubation was 120 h at 27 ± 2°C. The photoperiod was 48 h dark 18 h light:6 h dark.
 Seeds responded poorly to being capped with fine grain sand.
 Because the control seed emergence was so low, it is recommended that these results not be used as a definitive indication of toxicity.

All criteria have been met for a valid test and the test data and results are verified correct.

Seemingly 98/08/25



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 98759	Test: 981540
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Test Method: 7 and 14 d Earthworm Survival Test (*Eisina foetida*)

Reference: Greene et al. 1989. Protocols for Short Term Toxicity Screening of Hazardous Waste Sites. EPA 600/3-88-029.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

Sample Information:

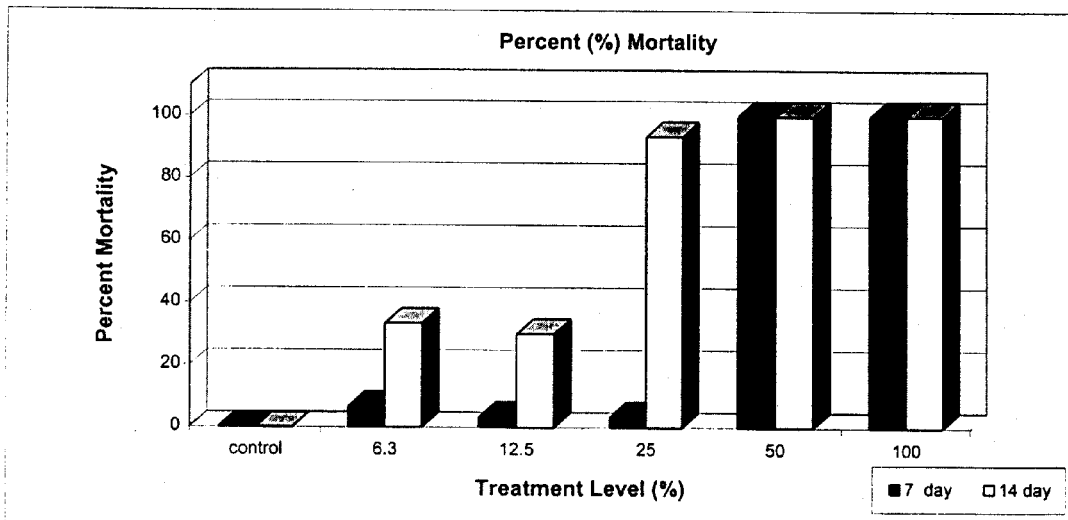
Description: Amine Biopile Composite

Collected On: 98/07/17 At: not given By: not given
 Received On: 98/07/17 At: 1200 By: P. Reimer

Test Information:

Started On: 98/07/31 At: PM By: JH
 Ended On: 98/08/14 At: PM By: JH
 Reported On: 98/08/17 By: CG

14 Day Test Result:	Value	Confidence Limits		Units	Method Calculated:
LC25	4.9	3.5	6.9	%	ICPIN
LC50	16	13	18	%	ICPIN
NOEC	0			%	Dunnett's
LOEC	6.3			%	Dunnett's



Notes: LC25 & LC50, concentrations lethal to 25 and 50% of the test population; NOEC & LOEC, no observed and lowest observed effect concentrations



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

TEST DATA

Client: 98025 Sample: 98759 Test: 981540

% mortality (7 days)			
	A	B	C
control	0	0	0
6.3	10	0	10
12.5	10	0	0
25	0	10	0
50	100	100	100
100	100	100	100

Avg	StDev
0	0
7	6
3	6
3	6
100	0
100	0

% mortality (14 days)			
	A	B	C
control	0	0	0
6.25	40	30	30
12.5	20	40	30
25	100	100	80
50	100	100	100
100	100	100	100

Avg	StDev
0	0
33	6
30	10
93	12
100	0
100	0

COMMENTS
 Soil dilutions were made and soil mixtures were hydrated overnight to 75% of their holding capacities.
 Test vessels were covered, then incubated at 27 ± 2°C for two weeks under low ambient light with a low ambient light with an 18 h light:6 h dark photoperiod.

All criteria have been met for a valid test and the test data and results are verified correct.

Sochiwoply 98108125



Client: 98025	Sample: 981181
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CLIENT INFORMATION

Client: Energy and Environmental Research Centre
 Operation: University of North Dakota, Grand Forks
 Address: 15 North 23rd Street, PO Box 9018
 City: Grand Forks
 Province/State: North Dakota
 Country: USA
 Postal/ZIP Code: 58203
 Billing Information:
 Contact: Jim Sorenson
 Tel: 701-777-5000
 Fax: 701-777-5181

SAMPLE INFORMATION

Sample Type: 020D-502 Plot H, Grab 0-60cm
 Collected On: 98/11/13 At: Not given
 Collected By: Not given
 Shipped On: 98/11/17
 Shipped By: Dropped off
 Received On: 98/11/17 At: 1000
 Received By: J.Hatcher
 Container: 1 x 1 ziplock
 Seals: None
 Initials on Seals: Not applicable

INITIAL CHEMISTRY

pH (units)	Not done	ammonium (mg-N/L)	Not done
Conductance (uS/cm)	Not done	residual chlorine (mg/L)	Not done
Dissolved Oxygen (mg/L)	Not done	Colour:	Loose brown soil
Temperature (oC)	Not done	Odour:	Organic
Alkalinity (mg-CaCO3/L)	Not done		
Hardness (mg-CaCO3/L)	Not done		

COMMENTS

Received limited sample volume; thus some tests were adjusted (EW - see project sheet)

SAMPLE HISTORY

Storage Conditions: 5°C
 Disposed On: n/a by n/a Method: n/a

TEST LOG

Test Type	RE (D)	EW (D)	SE (D)	BL (D)			
Number	983217	983219	983218	983188			
Started	98/11/26	98/11/26	98/11/26	98/11/26			
Ended	98/12/01	98/12/10	98/12/01	98/11/26			
Reported	98/12/21	98/12/21	98/12/21	98/12/21			
Faxed	Not done	Not done	Not done	Not done			



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 981181	Test: 98/12/01
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Test Method: Bacterial luminescence Test (IC50, four or more treatment levels plus a control)
Reference: Biological Test Method: Toxicity Test Using Luminescent Bacteria
 (*Vibrio fischeri*), 1992. Environment Canada, EPS 1/RM/24.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

Sample Information:

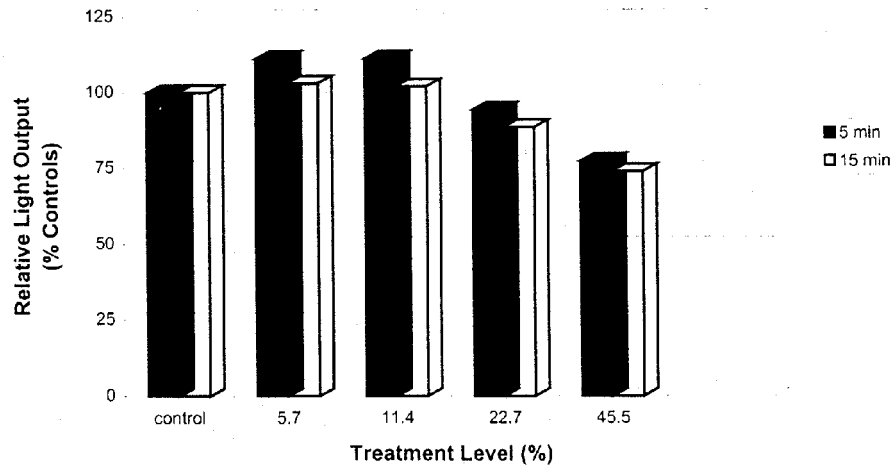
Description: 020D-502 Plot H, Grab 0-60cm
 Collected On: 98/11/13 At: Not given By: Not given
 Received On: 98/11/17 At: 1000 By: J.Hatcher

Test Information:

Started On: 98/11/26 At: PM By: J.Hatcher
 Ended On: 98/11/26 At: PM By: J.Hatcher
 Reported On: 98/12/21 By: M.lafrancesco

Test Result	Value	Confidence Limits	Units	Method Calculated
IC20 @ 15 min	>91		%	Regression analysis
IC50 @ 15 min	>91		%	Regression analysis

Graph of Light Output Relative to Controls at 5 and 15 Minutes



Notes: IC20 & IC50, concentrations that inhibit light output relative to controls by 20 and 50%



HydroQual
Laboratories Ltd.

#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

TEST DATA

Client: 98025 Sample: 981181 Test: 98/12/01

SAMPLE PRETREATMENT	
pH adjustment	Not required
preaeration	Not required
turbidity	Not centrifuged/filtered
other	None

LIGHT READINGS		
Treatment Level (%)	Time (min)	
	5	15
control	89	96
5.7	99	99
11.4	99	98
22.7	84	85
45.5	69	71

INHIBITION (%CTLS)	
5 min	15 min
100	100
111	103
111	102
94	89
78	74

COMMENTS

All criteria have been met for a valid test and the test data and results are verified correct.

Verona Kanysee-Tuck



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

QUALITY ASSURANCE INFORMATION

Test Method: Bacterial luminescence Test (IC₅₀, four or more treatments plus a control)
Reference: Biological Test Method: Toxicity Test Using Luminescent Bacteria (*Vibrio fischeri*), 1992. Environment Canada, EPS 1/RM/24.

TEST ORGANISM		TEST DESIGN	
test species	<i>Vibrio fischeri</i>	storage temp.	-20 to -25°C
batch number	ACV012-3	analyzer	Model 500
date obtained	98/07/07	test temperature	15°C
expires	99/07/01		

NOTE: NEW REAGENT LOT 98/07/09

Quality Assurance Information:

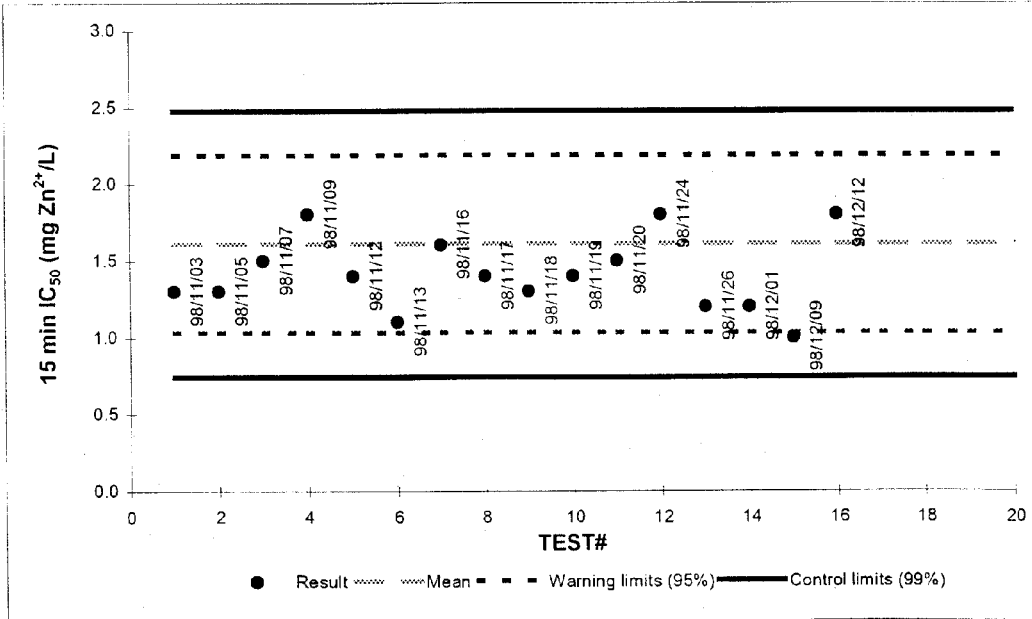
All criteria have been met for a valid test and the test data and result are verified correct.

Verna Barriere-Turk 98/12/23

WARNING CHART

TOXICANT: zinc (ZnSO₄*7H₂O)
CURRENT TEST: started: 98/12/12 ended: 98/12/12
RESULT: 1.8 (1.8-1.9) mg Zn²⁺/L

HISTORICAL MEAN: 1.6 **std.dev:** 0.3 **CV(%):** 17.9
CHART LIMITS: **warning:** 1.0 2.2 **control:** 0.7 2.5
 95% , two standard deviations 99% , three standard deviations





#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 981181	Test: 983217
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Test Method: 120h Lettuce (*Lactuca sativa*) Root Elongation Test

Reference: Greene et al., 1989. Protocols for Short Term Toxicity Screening of Hazardous Waste Sites. EPA 600/3-88-029.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

Sample Information:

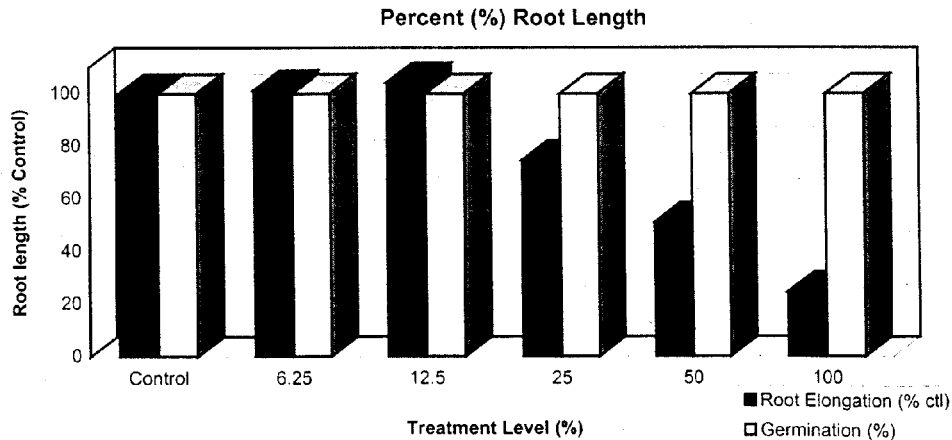
Sample: 020D-502 Plot H, Grab 0-60cm

Collected On: 98/11/13 At: Not given By: Not given
 Received On: 98/11/17 At: 1000 By: J.Hatcher

Test Information:

Started On: 98/11/26 At: AM By: J.Hatcher
 Ended On: 98/12/01 At: AM By: C.Christie
 Reported On: 98/12/21 By: M.lafrancesco

Test Result:	Value	Confidence Limits	Units	Method Calculated
ROOT ELONGATION				
IC25	24	17 37	%	Linear Interpolation
IC50	50	32 78	%	Linear Interpolation
NOEC	12.5		%	Dunnetts
LOEC	25		%	Dunnetts
PERCENT GERMINATION				
IC25	>100		%	Estimated
IC50	>100		%	Estimated
NOEC	100		%	Estimated
LOEC	>100		%	Estimated



Notes: IC25 & IC50, concentrations inhibiting root elongation by 25 and 50%; NOEC & LOEC, no observed and lowest observed effect concentrations



HydroQual
Laboratories Ltd.

#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

TEST DATA

Client: 98025 Sample: 981181 Test: 983217

ROOT LENGTHS (cm)

Treatment (%)	REPLICATE										AVG	STDEV	Percent (%) Germination
	1	2	3	4	5	6	7	8	9	10			

Date:	Time:		Initials:			Temp (oC):							
Control	4.0	5.0	4.0	4.0	4.0	4.0	3.0	4.5	5.0	4.0			100
	6.0	2.5	4.0	3.5	4.0	1.0	4.5	3.5	4.0	0.0	3.8	1.3	100
	2.5	4.0	3.5	4.5	1.5	3.0	5.0	4.5	4.0	5.5			100
6.25	5.0	5.0	3.5	3.0	3.5	4.0	3.5	4.0	1.0	0.0			100
	4.5	4.0	4.0	3.5	4.0	4.0	3.5	4.0	4.5	4.0	3.6	1.2	100
	4.5	3.0	4.0	4.0	3.5	4.5	4.5	3.5	3.0	0.0			100
12.5	4.0	3.5	4.5	2.5	5.0	4.0	3.5	4.5	4.0	3.5			100
	5.0	4.5	4.0	4.0	3.0	4.5	4.5	3.5	4.5	0.0	3.7	1.1	100
	4.0	4.0	3.0	4.5	3.5	3.5	3.5	2.0	4.0	1.5			100
25	2.5	3.0	3.0	2.5	2.5	3.5	2.0	2.5	1.0	0.0			100
	1.0	3.0	4.0	4.0	1.5	2.5	3.0	3.5	3.0	2.0	2.6	0.9	100
	1.5	2.5	2.5	3.5	3.0	4.0	3.5	3.0	2.5	3.0			100
50	2.5	2.5	2.0	2.0	3.0	2.0	2.0	2.0	2.0	0.0			100
	2.0	2.0	2.5	2.5	1.5	2.0	2.5	2.0	1.5	2.5	1.8	0.7	100
	2.0	1.5	1.0	1.0	2.0	1.5	1.5	1.5	1.0	0.0			100
100	0.5	1.0	0.5	1.0	1.0	1.0	1.0	1.5	1.0	0.5			100
	1.0	0.5	0.5	1.0	0.0	0.5	0.5	1.5	0.0	0.5	0.9	0.5	100
	1.5	2.0	1.0	0.5	1.0	1.5	1.0	1.0	1.0	0.5			100

COMMENTS:
Seed lot: LE981014
Test vessel: 100 x 15 mm plastic petri dish with filter
Hydration volume: 4mL
Temperature: 23±2°C
Lighting: none

All criteria have been met for a valid test and the test data and results are verified correct.

Verna Rose-Tuck



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 981181	Test: 983218
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Test Method: 120 h Lettuce (*Lactuca sativa*) Seedling Emergence Test
Reference: Greene et al. 1989. Protocols for Short Term Toxicity Screening of Hazardous Waste Sites. EPA 600/3-88-029.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

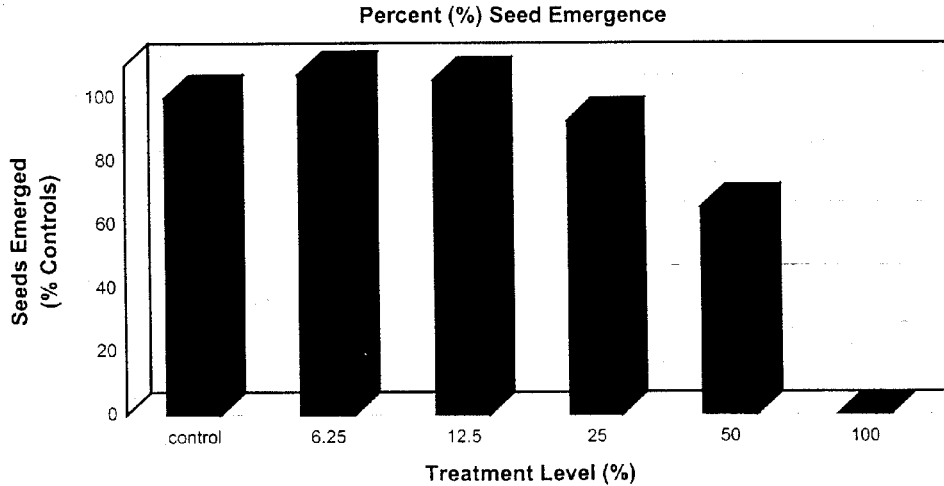
Sample Information:

Description: 020D-502 Plot H, Grab 0-60cm
 Collected On: 98/11/13 At: Not given By: Not given
 Received On: 98/11/17 At: 1000 By: J.Hatcher

Test Information:

Started On: 98/11/26 At: AM By: J.Hatcher
 Ended On: 98/12/01 At: AM By: C.Christie
 Reported On: 98/12/21 By: M.lafrancesco

Test Result:	Value	Confidence Limits	Units	Method Calculated
IC25	39	24 52	%	Linear Interpolation
IC50	60	43 72	%	Linear Interpolation
NOEC	25		%	Williams
LOEC	50		%	Williams



Notes: IC25 & IC50, concentrations inhibiting seed emergence by 25 and 50%; NOEC & LOEC, no observed and lowest observed effect concentrations



TEST DATA

Client: 98025 Sample: 981181 Test: 983218

# Seeds Emerged at 120 h			
	A	B	C
control	17	19	19
6.25	20	20	19
12.5	20	19	19
25	17	19	15
50	10	12	14
100	0	0	0

% of Seed Emergence				
A	B	C	Avg	StDev
85	95	95	92	6
100	100	95	98	3
100	95	95	97	3
85	95	75	85	10
50	60	70	60	10
0	0	0	0	0

COMMENTS :
 Lettuce seed lot: LE981014 Lighting: 48 hr dark; 72 hr of 16:18 light:dark
 Artificial soil batch: #20 Temperature: 23 ± 2°C
 30 g of artificial soil (control) or sample was placed in 100 x 15 mm plastic petri dishes. Twenty lettuce seeds were added to the soil and covered by a 30 g sand cap. Soil was hydrated with 15 mL of deionized water.

All criteria have been met for a valid test and the test data and results are verified correct.

Verna Fournier Tuck



#3, 6125 - 12 Street S.E. Calgary, Alberta Canada T2H 2K1
 TEL: (403) 253-7121 FAX: (403) 252-9363 1-800-808-6942

Client: 98025	Sample: 981181	Test: 983219
---------------	----------------	--------------

Test Method: 7 and 14 d Earthworm (*Eisenia fetida*) Survival Test (screening test, one treatment plus a control)

Reference: Greene et al. 1989. Protocols for Short Term Toxicity Screening of Hazardous Waste Sites. EPA 600/3-88-029.

Client Information: Energy and Environmental Research Centre
 University of North Dakota, Grand Forks

Sample Information:

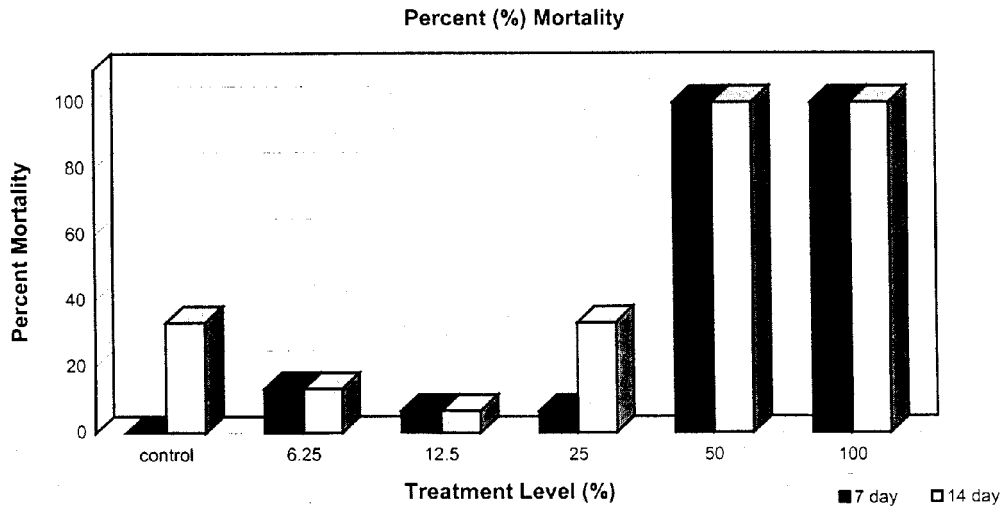
Description: 020D-502 Plot H, Grab 0-60cm

Collected On: 98/11/13 At: Not given By: Not given
 Received On: 98/11/17 At: 1000 By: J.Hatcher

Test Information:

Started On: 98/11/26 At: AM By: J.Hatcher
 Ended On: 98/12/10 At: AM By: C.Christie
 Reported On: 98/12/21 By: M.lafrancesco

Test Result:	Value	Confidence Limits	Units	Method Calculated
LC25	27	3 36	%	Linear Interpolation
LC50	34	1 41	%	Linear Interpolation
NOEC	25		%	Williams
LOEC	50		%	Williams



Notes: IC25 & IC50, concentrations inhibiting seed emergence by 25 and 50%; NOEC & LOEC, no observed and lowest observed effect concentrations



TEST DATA

Client: 98025 Sample: 981181 Test: 983219

% mortality (7 days)			
	A	B	C
control	0	0	0
6.25	20	0	20
12.5	20	0	0
25	20	0	0
50	100	100	100
100	100	100	100

Avg	StDev
0	0
13	12
7	12
7	12
100	0
100	0

% mortality (14 days)			
	A	B	C
control	0	0	100
6.25	20	0	20
12.5	20	0	0
25	100	0	0
50	100	100	100
100	100	100	100

Avg	StDev
33	58
13	12
7	12
33	58
100	0
100	0

COMMENTS :
Earthworm lot: EW981104
Artificial soil batch: #20
Test soil volume: 200 g
Test vessel: 250 mL plastic cup
Soil hydration volume: 40 mL deionized water
Lighting: continuous
Temperature: 23±2°C
Note: There was a limited sample volume, therefore the test volume was adjusted accordingly to 100g per vessel + 5 worms with 20mL DRO to hydrate. The dilution series were made with artificial soil.

All criteria have been met for a valid test and the test data and results are verified correct.

Verna Thompson

MATRIX SAMPLING DATA

APPENDIX E

Date	Sampler	Grids Sampled	Leachate	Temperature (C)			Wind Speed (m/s)				Notes
				T1	T2	T3	W1	W2	W3	W4	
17 Jul 98*	-	-		-	-	-	-	-	-	-	
14 Aug 98	Arnell	B, D, H	yes	45.1	39.7	44.5	N/A	N/A	N/A	N/A	Wind speed unavailable due to unremovable covers
28 Aug 98	Arnell	A, E, F	yes	28.3	32.4	28.7	1.2	1.5	2.1	2.1	Total leachate present ~ 800L
11 Sep 98	Arnell	B, C, H	yes	27.4	32.3	23.8	0	0	0	0	Total leachate present ~ 1000L
25 Sep 98	Arnell	D, F, G	yes	N/A	N/A	21.6	2	1.9	1.6	1.4	Total leachate present ~ 1000L
09 Oct 98	Arnell	A, C, F	yes	18.8	28.7	15.6	1.8	1.8	1.3	1.5	Leachate tank drained, sample collected from sump
27 Oct 98	Arnell	B, D, H		12.1	24.7	15.3	1.9	1.4	1.6	1.2	Leachate tank empty
09 Nov 98	Arnell	H		11	N/A	N/A	1.4	1.4	1.2	1.6	Leachate not obtainable, snow and ice on tarp
26 Nov 98*	-	-		-	-	-	-	-	-	-	
23 Jul 99	Larson	B, D, G	yes	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
17 Aug 99	Arnell	A, E, F	yes	20	22	20	3	2.7	3.2	2.8	
08 Sep 99	Larson	D, E, G	yes	18	N/A	16	1.7	1.8	1.5	1.2	
27 Sep 99	Arnell	A, C, F	yes	18	17	15	N/A	N/A	N/A	N/A	Blower off
12 Oct 99	Arnell	B, D, H		N/A	N/A	N/A	N/A	N/A	N/A	N/A	
25 Oct 99	Arnell	A, E, G		10	N/A	10	N/A	N/A	N/A	N/A	Blower off

*Field notes unavailable for this date.

KOMEX WELL 95-44A DATA

APPENDIX F

Data on Well 95-44A, Adjacent to East Side of Biopile

Ground Elevation = 1067.11 masl

Stick-up PVC Pipe = 0.63 m

Datum Elevation to Top of PVC = 1067.74 masl

Depth of Piezometer Below Ground = 7.30 m

Depth Interval of Sand = 4.00 – 7.30 m

Date	Depth to Groundwater (m)	Groundwater Surface Elevation (masl)	Hydraulic Conductivity (m/s)	Lithology
30 Nov 95	3.73	1064.0	1.80E-07	sand and gravel
24 Oct 96	4.57	1063.2		
16 Jun 97	3.22	1064.5		
19 May 98	5.15	1062.6		
16 Oct 98	3.59	1064.2		

Water Quality Data from Well 95-44A, Adjacent to the East Side of Biopile

Date	Temperature (C)	EC (µS/cm)	pH (units)	Turbidity	Dissolved Oxygen (mg/L)
15 Aug 95	8.0	1863	7.1	–	–
30 Nov 95	6.0	1584	7.1	–	1.80
05 Jun 96	7.1	1653	7.1	211.8	–
24 Oct 96	7.8	1746	7.2	–	0.04
16 Jun 97	11.0	1771	7.1	–	–
20 May 98	10.0	1865	7.2	–	–
16 Oct 98	7.6	1491	7.1	–	–
01 Jun 99	7.1	1249	7.2	–	–

Date	Chloride (mg/L)	DOC (mg/L)	DKN (mg/L)	TDS (mg/L)
14 Aug 95	1.6	149.0	67.5	1190
30 Nov 95	1.0	5.6	1.7	939
06 Jun 96	1.3	90.0	55.5	1120
06 Jun 96*	1.0	53.6	31.0	1100
24 Oct 96	0.7	79.5	42.5	1350
17 Jun 97	1.3	5.4	1.3	1250
20 May 98	1.3	111.0	91.0	1550
16 Oct 98	0.9	5.7	0.8	1190
01 Jun 99	1.6	6.7	1.1	1570

* duplicate

Data from Komex Reports

1. 1997 Groundwater Quality Monitoring Report, Okotoks Gas Plant, Komex International Ltd., January 1998.
2. 1998 Groundwater Quality Monitoring Program, Okotoks Gas Plant, Komex International Ltd., April 1999
3. Summary of Spring Groundwater Monitoring Results – Okotoks Gas Plant, Komex International, Ltd., July 16, 1999

BIOPILE PHOTOGRAPHS

APPENDIX G



Photo 1. Amine contaminated soils (dark greenish grey) being excavated to utilize as treatment soils for laboratory and field based activities.



Photo 2. Biopile cell holding 40 mm drainage rock overlying the RPE 25 mil poly liner.



Photo 3. Biopile cell's filter fabric overlying the drainage rock.



Photo 4. Leachate collection pump in the Southwest corner of the biopile cell.



Photo 5. Contaminated soil being mixed with straw, storage tanks for additives and leachate collection on the southwest side of biopile cell.



Photo 6. Aeration piping above the first 250 m³ of contaminated soil and straw.



Photo 7. Aeration system header.



Photo 8. Close view of electrical supply and leachate collection tank on the southwest side of the biopile cell.



Photo 9. Biopile during sampling event. Note air vent running along the center of the pile and the cover trap pulled off to the north.