

Test Plan - Phase I Treatability Study of Bioslurry Treatment Technology

Moss-American Site Milwaukee, Wisconsin

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TEST PLAN PHASE I TREATABILITY STUDY OF BIOSLURRY TREATMENT TECHNOLOGY

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List of Acronyms_____

ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
APC	air pollution control
BAC	Biotechnology Applications Center
BSRT	biological solids retention time
BTX	benzene, toluene, xylene
CCA	chromated copper arsenate
CD	Consent decree
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Register
CHP	Chemical Hygiene Plan
C:N:P	Carbon to nitrogen to phosphorus ratio
CPAH	carcinogenic polycyclic aromatic hydrocarbons
DOT	Department of Transportation
GC/MS	gas chromatography/mass spectrometer
g	gram
HEPA	high-efficiency particulate air
HPLC	high performance liquid chromatography
HRT	hydraulic retention time
IT	IT Corporation
ITAS	IT Analytical Services
kg	kilogram
KMCC	Kerr-McGee Chemical Corporation, Inc.
L	liter(s)
L/day	liter per day
mg/L	milligrams per liter
mg/kg	milligrams per kilogram
min	minute
mL	milliliter

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List of Acronyms (continued)_____

mL/min	milliliter per minute
mm	millimeter
MGP	Manufactured Gas Plant
MSDS	Material Safety Data Sheets
N	Normal
NIOSH	National Institute of Occupational Safety and Health
nm	nanometer
NPL	National Priorities List
OSHA	Occupational Safety and Health Administration
PAH	polynuclear aromatic hydrocarbons
PEL	permissible exposure limit
PFD	process flow diagram
PPE	personal protective equipment
q	specific substrate utilization rate
QAO	Quality Assurance Officer
QA/QC	quality assurance/quality control
RAS	return activated sludge
RI/FS	remedial investigation/feasibility study
ROD	Record of Decision
rpm	revolutions per minute
SOP	Standard Operating Procedure
SOW	Statement of Work
T&E	Test and Evaluation Facility
TOC	total organic carbon
TS	total solids
U.S. EPA	United States Environmental Protection Agency
UV	ultra violet
VOC	volatile organic compound
VS	volatile solids
WAS	waste activated sludge

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1.0 Project Description

1.1 Background

The United States Environmental Protection Agency (U.S. EPA), pursuant to Section 105 of 1980 Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), placed the Moss-American site in Milwaukee, Wisconsin (the Facility) on the National Priorities List (NPL). The U.S. EPA conducted a remedial investigation/feasibility study (RI/FS) for the Facility and issued the corresponding RI and FS reports on January 9, and May 24, 1990, respectively.

On May 29, 1990, U.S. EPA published a notice of completion of the RI/FS and issued the proposed remedial action plan for the Facility. A public comment period began with issuance of the proposed plan and extended until August 6, 1990. On September 27, 1990, the U.S. EPA Regional Administrator signed the Record of Decision (ROD), which describes the remedial action plan for the Facility. Public comments that were received, and the U.S. EPA response to the comments were included in the ROD with which the state of Wisconsin has expressed concurrence.

A Consent Decree (CD) incorporating the Statement of Work (SOW) was signed by Kerr-McGee Chemical Corporation, Inc. (KMCC) on July 17, 1991. The CD was lodged by the U.S. Department of Justice on December 28, 1991. Under this CD, the Settling Defendant, KMCC, will lead in developing and implementing the remedial design and remedial action plan for the Facility.

1.2 Facility Location

The Facility is located in the northwestern section of the city of Milwaukee, county of Milwaukee, state of Wisconsin, at the southeast corner of the intersection of Brown Deer and Granville Roads, at 8716 Granville Road. The Facility, as defined by the CD, includes the former Moss-American wood preserving plant property and approximately 5

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miles of the Little Menomonee River. The Little Menomonee River, portions of which are defined as part of the Facility, flows through the eastern portion of the former wood preserving plant, continuing on through the Milwaukee County Parkway, to its confluence with the Menomonee River about 5 miles south. Portions of the Little Menomonee River's floodplain are included in the Facility boundary. Fifty-one acres of the former wood preserving plant are undeveloped Milwaukee County park land. Twenty-three acres are owned by the Chicago and North Western Transportation Company and used as a loading and storage area for automobile transport. The Facility is located in a moderately-populated suburban area of mixed industrial, commercial, residential, and recreational use. Population in the nearby area is estimated at 2,036 persons per square mile.

1.3 Purpose and Content of Test Plan

Roy F. Weston (Weston) is the prime contractor to the Settling Defendant, KMCC, responsible for the CD implementation. Weston has contracted IT Corporation (IT) to conduct Phase I laboratory-scale treatability studies to evaluate the effectiveness of bioslurry technology in treating creosote-impacted soils at the Moss-American site. The treatability studies will be conducted as part of Predesign Task 16 of the Statement of Work (SOW).

IT will provide all services necessary to plan, implement, analyze and report the results of treatability testing of the biological slurry treatment process. The intent of testing is to determine the ability of such processes to treat creosote-contaminated soils from the Moss-American site. The polycyclic aromatic hydrocarbon (PAH) components of creosote, benzene-toluene-xylene (BTX), and carcinogenic polynuclear aromatic hydrocarbons (CPAH) are the site contaminants of concern. According to the RI, the maximum PAH concentration is 32,000 milligrams per kilogram (mg/kg); BTX concentrations range up to 17 mg/kg. The CPAH concentrations are 300 to 400 mg/kg. Maximum CPAH concentrations are approximately 1,900 mg/kg. The SOW requires treatment of contaminated site soils and sediments to at least 6.1 mg/kg of total CPAHs.

The SOW for treatability testing organized the testing scope into the following categories:

- Test Plan/Sampling and Analysis Plan Preparation
- Procurement of Test Materials
- Test Material Characterization
- Treatability Testing
- · Quality Assurance/Quality Control (QA/QC) Requirements
- · Technical Report
- Schedule

The test plan contains information addressing the procurement and characterization of test materials. In addition, the technical scope, materials and methods, analytical protocols, schedule, and deliverables of the treatability testing are also described.

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2.0 Remedial Technology Description

The primary remedial treatment alternative chosen by U.S. EPA for implementation at the Moss-American site is biological slurry treatment. The selection of a remedial treatment alternative was driven by compliance with the mandated cleanup criterion. The treatment standard for the soil and sediment is 6.1 milligrams per kilogram (mg/kg) of the carcinogenic polycyclic aromatic hydrocarbons (CPAH) fraction of the creosote contamination, specifically, chrysene, benz(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g-h-i)perylene, dibenz(a,h)anthracene, and indeno(1,2,3-c,d)pyrene. These compounds are characterized by high organic partition coefficients, low aqueous solubility, and low vapor pressures (Table 1).

Bioremediation of the CPAH in soils and sediments will employ bioslurry reactors. The main objectives of employing this technology are to oxidize the contaminants of interest and to reduce the volume of impacted material. Bioslurry reactors can provide rapid biodegradation of contaminants due to enhanced mass transfer rates and increased contaminant to microorganism contact [United States Environmental Protection Agency (U.S. EPA), 1990].

Due to the increased concentration of active biomass, improved oxygen delivery, and optimized environment in bioslurry reactors, the units are capable of treating high concentrations of organic contaminants in soils and sludges. Biodegradation of soils and sludges with selected contaminant concentrations ranging from 2,500 to 250,000 mg/kg in bioslurry reactors has been demonstrated (U.S. EPA, 1990).

Bioslurry reactors can be employed in on-site treatment with minimum space requirements and little risk of off-site contaminant migration. Bioslurry treatment is similar to conventional activated sludge treatment in that it utilizes suspended biomass.

Carcinogenic Polynuclear Aromatic Hydrocarbons	K _{ow} (log)	Aqueous Solubility (µg/L)	V.P. at 20°C (torr)
benz(a)anthracene	5.61	14	5.0x10 ⁻⁹
benzo(a)pyrene	6.04	3.8	5.0x10 ⁻⁷
benzo(b)fluoranthene	6.57	1.2	5.0x10 ⁻⁷
benzo(k)fluoranthene	6.84	0.55	5.0x10 ⁻⁷
chrysene	5.61	· 2	6.3x10 ⁻⁷
dibenz(a,h)anthracene	5.97	.50	1.0x10 ⁻¹⁰
benzo(g,h,i)perylene	7.23	0.26	1.0x10 ⁻¹⁰
indeno(1,2,3-c,d)pyrene	7.66	62	1.0x10-10

TABLE 1 Physical/Chemical Properties of CPAH Constituents

Sims, R. C., and Overcash, M. R., "Fate of Polynuclear Aromatic Compounds (PNAs) in Soil - Plant Systems", Residue Reviews, 1983.

Bioslurry reactor systems aerobically biodegrade aqueous slurries created through the mixing of soils or sludges with water. The most common mode of bioslurry treatment is batch; however, continuous-flow operation can be achieved. Aeration is provided through floating or submerged aerators or compressors and spargers. Mixing may be achieved through aeration alone or in conjunction with mechanical mixers. Nutrient addition and pH adjustment are accomplished through metered chemical addition to the reactor vessel. Following aeration, the treated slurry is dewatered via standard dewatering equipment, such as, clarifiers or filtration.

Maximum contaminant reduction is accomplished in bioslurry reactors primarily through proper feed preparation. Preparation of the influent waste stream should produce the general characteristics presented in Table 2.

Full-scale commercial bioslurry units require approximately 0.5 to 1 acre per million gallons of reactor volume (U.S. EPA, 1990). Reactor size is determined based on the hydraulic and biological solids retention times required for treatment. Retention times are established based on the biodegradability of the waste, level of treatment required, influent contaminant concentration, and physical/chemical nature of the waste.

The residual streams created during bioslurry remediation include treated solids, process water, and possible air emissions. The process water collected during the solids/liquid separation phase is usually recycled for influent waste stream slurrying or discharged under permit. Air emissions may be controlled through air pollution control (APC) devices.

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TABLE 2

General Influent Feed Characteristics for Bioslurry Treatment

Parameter	Target
Organics	0.025 - 25 percent by weight
Solids	10 - 40 percent by weight
Water	60 - 90 percent by weight
Solids Particle Size	Less than 1/4 inch
Temperature	15 - 35°C
pH _	4.5 - 8.8

U.S. EPA, 1990, "Slurry Biodegradation," EPA/540/290/016.

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3.0 Test Objectives

The Phase I treatability studies for biological slurry treatment includes a batch slurry study and a continuous-flow bioslurry reactor study. The batch slurry study will produce supporting data for the enhanced operation of the laboratory-scale bioslurry reactor. Batch slurry testing will be conducted in sealed, 1-liter (L) vessels at solids loadings of 20 and 30 percent. The duration of the batch study is 6 weeks with sample analysis during initiation, Week 3, and Week 6. The objectives of this study include:

- · Providing support data for enhanced operation of bioslurry reactor
- · Determination of the impact of solids loading on operation
- · Calculation of preliminary substrate utilization rates.

Following the collection of the batch slurry study Week 3-data, the bioslurry reactor study will be initiated. During the 3-month bioslurry study, a 60-L, stainless-steel, Eimco BioliftTM slurry reactor will be operated in continuous flow mode. Operation of this unit under the optimum solids loading determined during batch testing will provide performance data generated to determine the efficacy of meeting the specified treatment standard. The objectives of the bioslurry investigation include:

- Estimation of hydraulic retention time (HRT) and biological solids retention time (BSRT) set points for operation
- · Determination of the efficacy of meeting the specified treatment standard
- · Identification of requirements for additional physical/chemical pretreatment
- Generation of performance data upon which pilot-scale design can be established.

The dewatering of treated solids will not be evaluated during the course of the investigation.

4.0 Experimental Design and Procedures

The test material employed in the batch and bioslurry reactor studies will be collected from the Moss-American site. Two samples will be collected, with one composite soil sample containing carcinogenic polycyclic aromatic hydrocarbons (CPAH) in the range of 300 to 600 milligrams per kilogram (mg/kg) and one sample containing CPAH in the range of 1,000 to 1,500 mg/kg. Initial characterization of the samples will be conducted immediately following sample collection. Test parameters will include bulk density, particle size distribution, porosity, moisture, liquid/plastic limits, pH, total organic carbon (TOC), and total and specific polycyclic aromatic hydrocarbons (PAH)-degrading microbial populations.

4.1 Sample Procurement

The two composite soil samples will be shipped to IT Corporation's (IT) Biotechnology Applications Center (BAC) via a certified, commercial carrier. Approximately 75 pounds (lb) of each soil sample will be collected for the execution of the treatability testing; total soil mass required is approximately 150 lb. Appropriate shipping documentation presented in Appendix A will accompany all samples sent to the BAC. All samples will be shipped to the following address:

> IT Corporation Biotechnology Applications Center 9041 Executive Park Drive, Suite 309 Knoxville, Tennessee 37923 Attention: Kandi Brown

Following the receipt of the soil, the containers will be visually inspected and sample volumes recorded. All samples received at the BAC are automatically logged into a sample tracking system and given independent sample identification numbers. All

treatability study samples will be refrigerated at 4°C. The operating temperature of the refrigeration unit is maintained at 4°C, with this temperature being verified biweekly.

Once receipt of the samples has been properly documented, a representative composite of the two samples will be prepared. Equal volumes (by weight) of both soil samples will be composited in ventilated hoods and thoroughly mixed. Mixing will be accomplished manually. Following composition, 500 grams (g) of the composite will be submitted to the analytical laboratory for analysis. Analysis will include bulk density, particle size distribution, porosity, moisture, liquid/plastic limits, pH, TOC, and total and specific PAH-degrading microbial populations. All volumes of soil removed from the composited fraction will be logged on Sample Collection Logs (Appendix B). The remaining volume of composited soil will stored at 4° C.

4.2 Batch Slurry Testing

The batch slurry study will be performed for 6 weeks. The six treatments described in Table 3 will be evaluated during the batch study. As stated in Chapter 3.0, the objective of this study is to determine the impact of solids loading on operation, provide supporting data for bioslurry reactor operation, and establish preliminary substrate utilization rates.

Treatments 1 and 2 are nutrient- and oxygen-amended treatments containing 20 percent contaminated solids. Treatments 3 and 4 are also nutrient- and oxygen-amended treatments evaluating a 30 percent solids loading. Treatments 5 and 6 will serve as the biologically inhibited controls for the study; analysis of these treatments will be used to determine abiotic losses of target compounds from the treatments. Biologically inhibited controls will be established through the addition of 250 to 500 milligrams per liter (mg/L) of mercuric chloride. Treatments 1 and 2, 3 and 4, and 5 and 6 are duplicates.

The batch study will be conducted in sterile, glass, sealed, 1-liter (L) bottles. The sample collection port on the containers consists of TeflonTM tubing inserted through a TeflonTM stopper positioned in the neck of the bottle. Sample collection port connections will be

TABLE 3 Batch Slurry Study Treatments

Treatment No.	Description			
1	20 percent solids, nutrients, oxygen			
2	20 percent solids, nutrients, oxygen			
3	30 percent solids, nutrients, oxygen			
4	30 percent solids, nutrients, oxygen			
5	20 percent solids, nutrients, mercuric chloride, oxygen			
6	30 percent solids, nutrients, mercuric chloride, oxygen			

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sealed using Viton O-rings and leak tested before the initiation of the study. Samples will be withdrawn through the TeflonTM tubing using a gas-tight syringe. Hydrocarbon-free air will be introduced into the treatments following sample collection in order to prevent the creation of a vacuum.

Composited soils, screened to a representative particle size of less than 1 millimeter (mm) in diameter, will be placed in the bottles at solids densities of 20 and 30 weight percent. To achieve accurate solids loading, three 280-g and three 420-g aliquots of soil (dry weight) will be weighed and placed into six, 2-L glass containers. Sterile distilled/deionized water will then be used to fluidize the samples and bring the final volume to 1.4 L. Approximately 400 mL of this volume will be submitted for initial analysis. The remaining portion will be placed in 1-L vessels and initially maintained with zero headspace. Each vessel will contain a sterile stir bar to aid in mixing during sample collection.

The treatment containers will be placed on a magnetic stir plate and vigorously mixed during the collection of Week-3 and Week-6 samples. Initial determination of the slurry pH and macronutrient, i.e., ammoniacal nitrogen and ortho-phosphate, concentrations will be completed. The slurry pH will be adjusted to 7.0 using either 1 Normal (N) hydrochloric acid or 1 N sodium hydroxide.

The treatment macronutrient and dissolved oxygen concentrations will be maintained at the operating conditions presented in Table 4. Macronutrient concentrations will be controlled through the addition of ammonium chloride and potassium phosphate to each treatment during the charging of the treatment vessels. These compounds will be added based on the initial characterization of the treatability test composite. The target carbon:nitrogen:phosphorous (C:N:P) ratio is 100:10:1. At these levels, nutrient concentrations are not expected to limit biological activity during the 6-week investigation. If nutrient addition is required, macronutrients will be added in dry form directly to the treatments through the sample collection port.

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TABLE 4

Batch Slurry Study Nutrient Target Concentrations

Constituent	Sample Point	Target Concentration at 100 mg/L TOC
Ammoniacal nitrogen	Aqueous phase	10 mg/L
Ortho-phosphate	Aqueous phase	1 mg/L
Dissolved oxygen	Slurry phase	3 mg/L

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Dissolved oxygen concentrations will be maintained at the target concentration of 3 mg/L. This target was set to approximate values that could be practically achieved in a full-scale bioslurry reactor system. Dissolved oxygen measurements will be made weekly during the course of the investigation using a modified, galvanic-cell, oxygen probe (Graves, et al., 1992). Approximately 1 milliliter (mL) of sample will be collected from each treatment and submitted for this analysis. Dissolved oxygen concentrations will be maintained in the treatments through the addition of hydrogen peroxide and continual mixing on a modified-tube rotator. Hydrogen peroxide is used during this study as a consequence of the equipment configuration and will not be used in the bioslurry reactor study or for full-scale treatment.

A tube rotator modified to accommodate 1-L bottles was chosen over conventional sparging and agitation systems due to improved suspension of the treatment solids, conservation of volatile fractions, and more consistent suspended solids concentrations due to reduced solids adsorption at the liquid/air interface. Following preparation, the treatments will be placed on the modified-tube rotator, rotated at 200 revolutions per minute (rpm), and maintained at room temperature throughout the course of the study.

The sampling schedule for the batch slurry study is presented in Table 5. Approximately 400 mL of slurry will be collected during each of the three sampling periods. Approximately 30 mL of the well-mixed slurry will be submitted for analysis. The remaining 370 mL of the sample volume will be separated under quiescent conditions and analyzed for contaminants in the aqueous and solid phases. Aqueous and solids phases will be gravimetrically separated with the aqueous phase separated by decanting.

The analytical parameters monitored at study initiation, Week 3, and Week 6 in the aqueous phase of each treatment are PAH, benzene, toluene, xylenes (BTX), macronutrients, pH, oxygen, and TOC. The slurry phase will be monitored for total solids (TS) and VS concentrations and microbial density of heterotrophic bacteria and anthracene degraders. The soil fraction of each treatment will be monitored for PAH and BTX concentrations.

 TABLE 5

 Batch Slurry Study Sampling and Analysis Schedule

Treatment No. 1-6	Analysis								Volume Remove d	Frequency	
	PAH	втх	тос	N&P	DO	pН	TS	VS	Micro	•	
Aqueous	100 mL	50 mL	10 mL	20 mL	1 mL					370 mL	3/Treatmen t
Slurry							20 mL		10 mL	30 mL	3/Treatmen t
Solids	20 g	10 g									3/Treatmen t
Total										400 mL	

PAH - Polycyclic aromatic hydrocarbons

BTX - Benzene-toluene-xylene

TOC - Total organic carbon

N&P - Ammoniacal nitrogen and ortho-phosphate

DO - Dissolved oxygen

TS - Total solids

mL - milliliter

g - grams

VS - Volatile solids

Micro - Microbial enumerations

The preliminary substrate utilization rates determined during the batch slurry investigation will be used to confirm the selection of retention time set points proposed for the bioslurry reactor study. In addition, performance data generated during this investigation will be evaluated to determine the appropriate solids loading for the bioslurry reactor.

4.3 Bioslurry Reactor Study

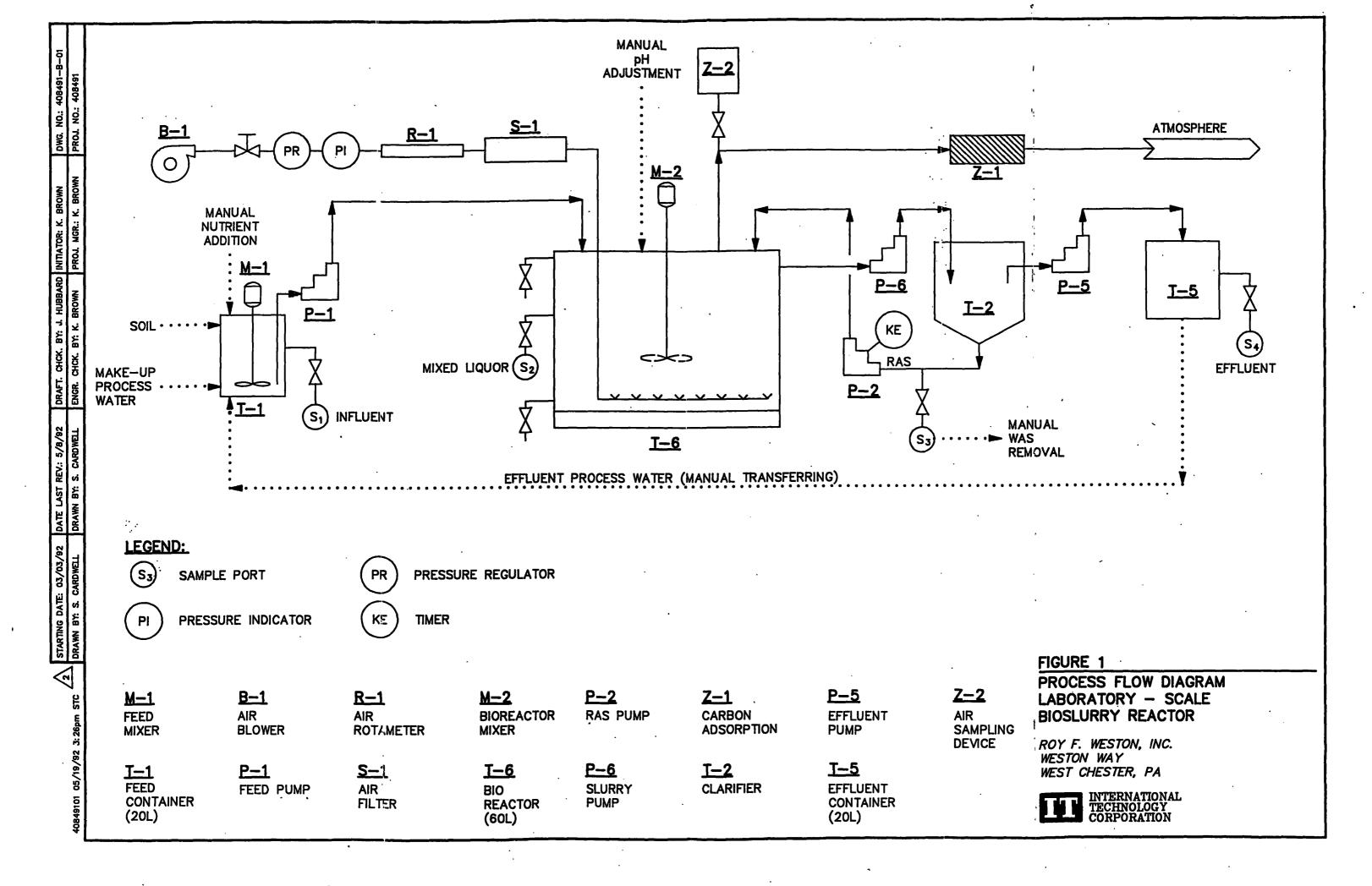
Due to the similarity of activated sludge and bioslurry reactor systems, conventional acronyms such as waste activated sludge (WAS) and return activated sludge (RAS) will be used to describe the slurry recycle system in the laboratory-scale bioslurry reactor.

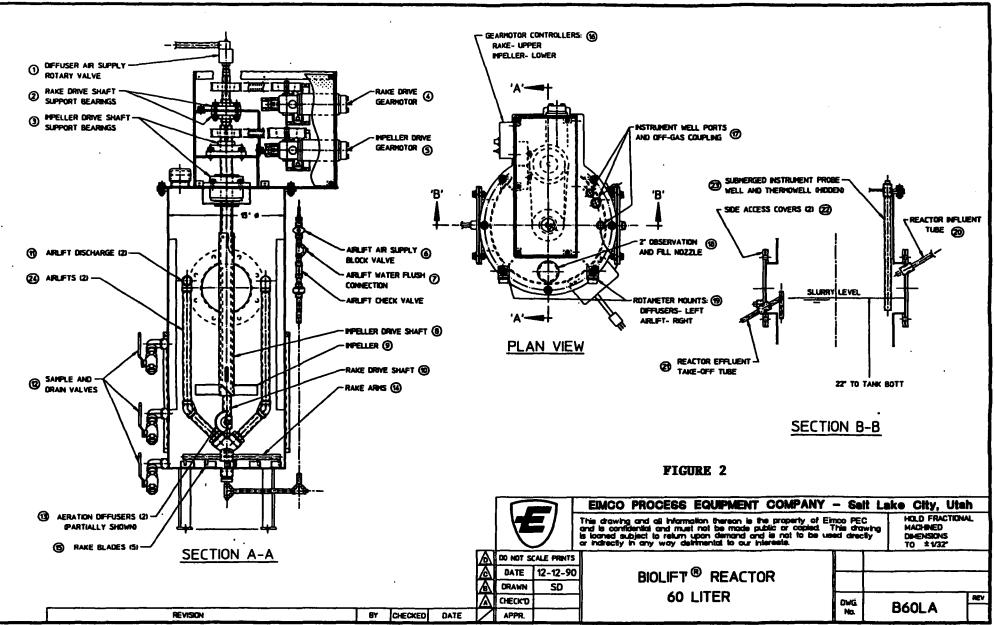
4.3.1 Process Description

A 60-L, stainless steel, Eimco BioliftTM slurry reactor will be employed during this 3month study and will operate in continuous flow mode. The process flow diagram (PFD) for the reactor system is presented in Figure 1. Figure 2 illustrates the internal design of the reactor. The influent waste stream will be continuously stirred and fluidized in a closed container before introduction in the reaction vessel. Feed will be introduced to the reaction vessel (Bioreactor T-6) at an average daily flow rate of 2 liters per day (L/day).

At this daily flow rate, the bioslurry reactor hydraulic retention time (HRT) will be maintained at 30 days. Following aeration, the treated slurry will be pumped to the system clarifier. The clarifier will be covered to reduce the emission of volatile compounds. The treated slurry will be maintained under quiescent conditions during clarification to allow for gravitational settling. Effluent will be removed from the clarifier to the effluent container.

During the steady-state operation of a completely mixed, continuous-flow reactor, the contaminant concentration in the effluent is equal to the concentration in the reactor (Benefield and Randall, 1980). Therefore, solids collected from the bioslurry reactor are considered to represent the final remediated product. Clarified water in the effluent





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container will be manually recycled to mix with the influent feed. Recycling of the process effluent will be conducted to more accurately simulate full-scale operations. A portion of the settled solids in the clarifier will then be returned to the aeration vessel to manipulate and control the biological solids retention time (BSRT), with the remaining portion removed from the system as WAS.

Before charging the reactor, the creosote-impacted soil composite (nonfluidized) will be screened to a representative particle size of less than 1.0 mm in diameter. The purpose of soil screening is to minimize mechanical problems in the laboratory reactor. The influent slurry will be prepared in 20-L portions and placed in a closed container. The slurry will be continuously stirred to reduce the separation of solids. The percent solids concentration of the feed will be determined based on Week-3 data from the batch slurry study and the maximum carbon loading attainable in a bioslurry system.

Pumping of system materials will be accomplished through the use of RandolphTM pumps that efficiently handle solid and semi-solid materials. Pump flow rates will be checked and calibrated daily. For streams that require low flow rates, i.e., RAS stream, pumping operations will be controlled via a timer allowing for higher flow rates over reduced periods of time.

Reactor operating conditions will be maintained at room temperature, 3 mg/L dissolved oxygen, and pH 7. Dissolved oxygen will be supplied to the unit via the sparging of breathing quality air, and system pH will be maintained through manual additions of 1 N hydrochloric acid or 1 N sodium hydroxide to the reaction vessel.

The system BSRT set point is 40 days. Operation at this set point will determine compliance with effluent standards during optimum operation. The operating set point will be maintained for a period equal to 3 times the BSRT value to ensure collection of data generated during steady-state operation. If the results generated during operation at the 40-day BSRT set point are favorable, continued operation at a 5-day set point to bracket the bioslurry reactor performance range may be conducted.

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The BSRT set point will be maintained through a mass balance of the system solids. Following the determination of TS and VS concentrations in the RAS, mixed liquor in the reactor, and effluent, the volume of WAS will be calculated based on Equation 2 of Section 4.4.2. It is assumed that the solids concentration in the WAS is equal to the concentration of the RAS stream. If TS and VS measurements are not available for each sample point, the previous measurement will be employed in the calculation of waste solids. For the purpose of mass balance, the sample volume removed from the reactor is also considered as WAS. The system biomass recycle rate will be initially set at approximately 10 percent of the influent feed rate, e.g., 0.2 L/day. This ratio has been demonstrated effective for bioslurry reactor applications (Marks, et al., 1991).

All operational set points are listed below:

•	Feed flow	2 L/day
•	HRT	30 days
•	Temperature	Room temperature
•	Dissolved oxygen	3 mg/L
•	pH	7 - 8
•	Agitation	500 rpm
•	BŠRT	≥30 days
•	Reactor volume	60 L
•	Return activated sludge	0.2 L/day.

Volatilization of influent constituents will be quantified in the reactor system. The influent waste container and clarifier are covered to minimize the loss of contaminants to the atmosphere and reduce employee exposure to the waste. Volatilization that will occur during waste aeration and mixing will be controlled through carbon adsorption (see Figure 1). Bioslurry reactor headspace sampling will be conducted and quantified to assist in the calculation of the system's materials balance.

Following the completion of the bioslurry reactor study, the influent feed container, bioslurry reactor, clarifier, system tubing (Viton), and the effluent container will be

solvent extracted and analyzed for CPAH. Extraction will be completed to determine the quantity of contaminants that were adsorbed in the system, rather than biodegraded.

4.3.2 Analytical Schedule

The operating conditions for temperature, dissolved oxygen, and pH will be monitored daily. All reactor sampling points illustrated on the PFD will be identified and color-coded. For clarification of the following text, sample identifiers are defined in Table 6 and labeled in Figure 1. All mixed liquor reactor samples will be collected from the second sampling port located on the side of the bioslurry reactor (S2).

The sampling schedule for the bioslurry reactor study is presented in Table 7. The influent waste stream (S1) will be characterized for PAH concentrations in the aqueous and solids phase twice per week. The slurry will be analyzed for TS and VS concentrations twice per week.

The reactor slurry will be collected from Sample Port S2. The reactor slurry particle size will be monitored once a week to determine its impact on the release of soil-bound contaminants. The monitoring of particle size is important due to the effect of stirred-tank reactors internal hydrodynamics that influence both the particle size and the liquid-phase mass transfer coefficient. In general, large particles result in diffusional limitations on the ingress of substrate, thereby, effecting the overall specific rate of reaction (Atkinson and Mavituna, 1983).

The reactor slurry phase will also be monitored twice weekly for TS and VS concentrations. Microbial enumerations of total heterotrophs and anthracene degraders will be conducted once per week. The analysis will be conducted on slurry grab samples collected from Sample Port S2. Analytical methods describing the techniques used to determine microbial populations are provided in Chapter 6.0.

The reactor aqueous-phase macronutrient concentrations will be monitored once per week. The macronutrient concentrations will be controlled based on maintaining a

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TABLE 6

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Bioslurry Reactor Study Sample Point Descriptions

Sample ID	Sample Point Description	Sample Type		
S1 -	Influent waste stream	Grab		
S2	Reactor slurry	Grab		
S3	RAS	Grab		
S4	Clarified effluent	24-hour composite		
Z-2	Reactor headspace	24-hour composite		

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 TABLE 7

 Bioslurry Reactor Study Sampling and Analysis Schedule

Sample Point	Analysis									
	PAH	ВТХ	тос	N&P	·· DO	pН	TS	VS	Micro	Particle Size
influent Waste Stream (S ₁)										
Aqueous (S ₁)										
Volume	100 mL	-	-	-		-	-	8	1	
Frequency	2/wk	-	1	-	_	1	-	-	-	
Slurry (S ₁)										
Volume	*	-	1	-	-	1	20 mL	-	-	-
Frequency	-	-	-	-	-	1	2/wk	2/wk	-	
Solids (S ₁)										
Volume	20 g	-	8	-	-		-	1		
Frequency	2/wk	-	1	-	-			-		-
Reactor Mixed Liquor (S₂)										
Aqueous (S ₂)										
Volume	100 mL	50 mL	10 mL	20 mL			-	-		
Frequency	2/wk	1/wk	2/wk	1/wk	-		-	-	-	-
Slurry (S ₂)										
Volume	-		-		1 mL		20 mL		10 mL.	500 mL
Frequency	-		-	-	2/wk	2/wk	2/wk	2/wk	1/wk	1/wk

TABLE 7 (Continued) Bioslurry Reactor Study Sampling and Analysis Schedule

Moss-American Site Test Plan - Bioslurry Treatability Date: 29 May 1992 Revision: 0

					<u> </u>					
Sample Point		Analysis								
	PAH	BTX	тос	N&P	DO	рН	TS	vs	Micro	Particle Size
Solids (S ₂)										
Volume	20 g	10 g		-				-		·
Frequency	2/wk	1/wk	2/wk	-						
RAS (S ₁)										
Aqueous (S ₃)										
Volume	100 mL	-	-	-	-	-	-	-	••	
Frequency	1/wk	-		1	8	-	-	8	. 1	
Slurry (S ₂)										
Volume		-		8	-	-	20 mL	-		
Frequency		-	-	-	-		2/wk	2/wk	-	-
Solids (S3)										
Volume	20 g	-	-	-	-			-	-	
Frequency	1/wk	-	-	-	-		_	-	-	-
Effluent Waste Stream (S4)										
Slurry (S ₄)										
Volume	-	-	-	-	-	-	20 mL	-	-	
Frequency	-	-		-	-		2/wk	2/wk	-	-
Air (Z ₂)	1/wk	1/wk		-	-	-	-	-	-	-

PAH - Polycyclic aromatic hydrocarbons

TS - Total solids

BTX - Benzene-totuene-xylene

mL - milliliter

RAS - Return Activated Sludge wk - Week

S₃ - RAS sample port

S₄ - Effluent sample port

- TOC Total organic carbon
- g grams

to - Initial characterization $\bar{S_1}$ - Influent sample port

DO - Dissolved oxygen

- N&P Ammoniacal nitrogen and ortho-phosphate VS Volatile solids Micro - Microbial enumerations
- S₂ Mixed liquor sample port

C:N:P ratio of 100:10:1. Macronutrients will be directly added in batch to the influent feed.

Aqueous- and solid-phase PAH and TOC concentrations in the reaction vessel will be monitored twice weekly (S2). The BTX concentration of the aqueous and solids phase will be determined once per week. The PAH content of the aqueous and solids phase will be measured in the RAS stream once per week.

In order to accurately determine the system BSRT, TS and VS concentrations of the RAS will be determined twice weekly in the slurry phase collected through Sample Port S3. In addition, the TS and VS concentrations in the clarified effluent will also be monitored twice weekly (S4).

Volatilization of contaminants must be monitored to complete the mass balance of carbon in the system. Air monitoring for volatiles and semivolatiles will be conducted weekly. Headspace constituents will be determined through the analysis of air sampled through Z-2. Analysis of headspace samples is thoroughly discussed in Chapter 6.0. Analysis of carbon adsorption materials will not be performed.

Complete mixing of the reactor solids will be verified periodically during the 40-day BSRT set point. Verification will be accomplished through the analysis of sample TS concentrations. Samples will be extracted from the three sample ports located on the side of the bioslurry reactor. The three ports represent three potentially distinct zones of the slurry. The bottom sample port will provide sample material from within the rakemixing zone. The middle port will provide sample material from within the most wellmixed zone. Finally, the top sample port will provide sample material of any oil phase that may be present. Analysis of the samples for TS concentration will then determine the mixing efficiency of the reactor system. If mixing is found to be extremely nonuniform, the agitation speed or airlift system will be altered and complete mixing will be rechecked.

Sample logs will be maintained in a bound laboratory notebook, that is solely dedicated to this project. The logs will document volumes removed or added so the system HRT can be accurately calculated.

Performance data, i.e., effluent solids quality, generated during the completion of the bioslurry investigation will be used to determine compliance with the mandated CPAH target concentrations. The performance data will also be used to establish HRT and BSRT set points for operation and, thereby, establish the pilot-scale design. In addition, the need for physical/chemical pretreatment will be evaluated based on adherence to cleanup criteria.

4.4 Biokinetic Calculations

Operation of laboratory scale reactors in this treatability study will rely upon the total solids levels in the test reactors as the control parameter.

4.4.1 Batch Slurry Study

Equations 1 through 3 were derived from equations presented by Benefield and Randall, 1980. The preliminary specific substrate utilization rates (q) based on TOC and CPAH utilization per unit biomass will be determined in the batch study using the following equation:

$$q = \frac{(S_i - S_f)/\Delta t}{X}$$

(Equation 1)

Where:

q = specific substrate utilization rate (hr⁻¹)

 S_i = Initial substrate concentration (mg/L)

 S_f = Final substrate concentration (mg/L)

 $\Delta t = Time elapsed (hours)$

X = TS concentration in slurry (mg/L).

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4.4.2 Bioslurry Reactor Study

The BSRT (day) of the laboratory-scale system will be approximated through mass balance of the system solids:

$$BSRT = \frac{XV}{Q_w X_r + (Q-Q_w) X_e}$$
(Equ

(Equation 2)

Where:

X = TS concentration in the aeration vessel (mg/L) V = Volume of the aeration vessel (L)

 $Q_w = WAS$ flow rate (L/day)

 $X_{r} = TS$ concentration in RAS (mg/L)

Q = Influent flow rate (L/day)

 $X_e = TS$ concentration in system effluent (mg/L).

The HRT of a bioreactor is mathematically determined by dividing the volume of the reactor by the influent flow rate. Under recycle conditions, the BSRT exceeds HRT, consequently control of HRT at 30 days with recycle will result in BSRT values greater than or equal to 30 days.

The RAS recycle ratio will be determined in the laboratory-scale investigation using the following equation:

$$Q_{R} = \frac{OX - Q_{W}X_{r}}{X_{r} - X}$$
 (Equation 3)

Where:

 Q_R = Recycle ratio Q = Influent feed flow rate (L/day) X = TS concentration in the aeration basin (mg/L) Q_W = WAS flow rate (L/day) X_r = RAS TS concentration (mg/L).

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5.0 Equipment and Materials

The batch study will be conducted in sterile, glass, sealed, 1-liter (L) bottles. The sample collection port on the containers consists of TeflonTM tubing inserted through a TeflonTM stopper positioned in the neck of the bottle. Sample collection port connections will be sealed using Viton O-rings and leak tested before the initiation of the study. Samples will be withdrawn through the TeflonTM tubing using a gas-tight syringe.

The laboratory-scale bioslurry investigation will be conducted in a continuous-flow, completely mixed, 60-L, stainless-steel, Eimco BioliftTM slurry reactor (Eimco Process Equipment, Company, Salt Lake City, Utah). The system process flow diagram (PFD) is shown in Figure 1. Materials of construction are primarily stainless steel and Viton tubing. All portions of the reactor system that will contact the slurry mixture are stainless steel and Viton tubing. During air sampling, the reactor headspace will be in contact with Teflon probes and nickel tubing.

The influent waste stream will be continuously stirred (M-1) in a sealed container (T-1) before introduction to the reactor aeration vessel (Bioreactor T-6) via Pump P-1. An influent feed sample collection port is provided at S1.

The bioreactor (T-6) is equipped with controllers to maintain agitation and air flow rate. Agitation will be maintained using an impeller mixing system (M-2), sparged air, and an airlift system to improve the system oxygen transfer efficiency. Hydrocarbon-free, ambient air from a blower will be used to supply oxygen to the bioreactor through an air sparger installed at the bottom of the reactor. Air is filtered (0.45-micron Whatman filter) before introduction into the reactor.

The mixed liquor pH will be maintained at neutral by the manual addition of acid or base. Manual wasting of the reactor mixed liquor will be performed daily from the return activated sludge (RAS) recycle line. Volumes removed for sample analysis will be considered when calculating the waste activated sludge (WAS) volume.

Reactor mixed liquor will be pumped to Clarifier T-2 by Pump P-6. The system clarifier is covered to minimize the mass of volatiles lost from the system. Pump P-5 will transfer the clarified effluent to Effluent Container T-5. The effluent container may be sampled through Sample Port S4. A portion of solids will be returned to the bioreactor from the clarifier through RAS Pump P-2. The system RAS will be sampled through Sample Port S3.

Reactor mixed liquor grab samples will be obtained from the sampling port (S2) on the reactor. Influent feed samples will be collected from S1. RAS and effluent samples will be collected from Sample Ports S3 and S4, respectively. Volatilization of the aerated slurry will be controlled using a carbon adsorption system (Z-1) before discharge to the atmosphere. Headspace semivolatile constituents will be measured through air sampling at Z-2. The air sampling train will consist of a TeflonTM probe, a 47-millimeters (mm) TeflonTM membrane filter, and an XAD-2 sorbent sampling tube. The air sampling system used to collect volatile off-gas samples will consist of a stainless-steel SummaTM polished canister, Milaflow pneumatic flow controller 10 cubic centimeters per minute (cm³/min), and nickel tubing.

Other major pieces of equipment that will be utilized during this project are listed below:

- Tekmar Purge and Trap
- · Gas chromatograph, Hewlett Packard 5890
- · High pressure liquid chromatograph (HPLC), Perkin Elmer Series 4
- Total Organic Compounds (TOC) Analyzer, Dohrmann DC-80
- · Oxygen detection device, IT Corporation patent pending
- Eimco BioliftTM Slurry Reactor
- · Finnigan Model OWA 1050 gas chromatograph/mass spectrometer (GC/MS)

- · Various incubators, shakers, pH electrodes, ion-selective electrodes, spectrophotometers, and sterile and fume hoods
 RandolphTM pumps
 Personal computers.

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6.0 Sampling and Analysis

All treatability testing will be completed in the Biotechnology Applications Center (BAC) laboratory located in Knoxville, Tennessee. This facility holds a special exemption from the State of Tennessee that permits execution of treatability studies. The BAC laboratory operates in accordance with an approved Chemical Hygiene Plan (CHP). All activities at the BAC conform to the standards set forth in the CHP.

All analyses conducted for the target compounds will use United States Environmental Protection Agency (U.S. EPA)-approved methods, or modifications thereof. Polynuclear aromatic hydrocarbons (PAH) concentrations will be determined using modified U.S. EPA Method 8310. The benzene, toluene, xylene (BTX) concentration will be determined using U.S. EPA Method 8020. Modifications of EPA methods will be made to accommodate the collection of smaller sample volumes during the batch slurry investigation. The methods will also be employed during the bioslurry reactor study for uniformity. Aqueous and solids phases will be separated gravimetrically and submitted for analysis.

U.S. EPA Method 8310 will be modified for sample extraction of the solids phase with methylene chloride. Following extraction, the sample will be analyzed by high performance liquid chromatography (HPLC) equipped with a florescence detector. The aqueous-phase will be directly injected into the HPLC and analyzed by a ultraviolet (UV) detector at 255 nanometers (nm).

Table 8 illustrates detection limits achieved during the analysis of standards, soils collected from a coal-coking waste lagoon, and a manufactured gas plant site using this methodology and the equipment listed in Chapter 5.0. Analysis of these compounds was conducted by IT Corporation (IT) at the University of Tennessee Center for

TABLE 8

Summary of PAH Concentrations Detectable by Fluorescence Detection and Photo-Diode Array Detection

РАН	Pure Standards ¹⁺	Pure Standards ²⁺	Coke Waste ²⁺	Untreated ⁴ MGP Soil ^{2*}	Treated ⁴ MGP Soil ^{2*}
Naphthalene	0.1	0.2	50	3.5	1
Acenaphthylene	NA	0.2	100	3	1
Acenaphthene	0.1	0.2	50	2	1
Fluorene	0.02	0.4	10	1.5	1
Phenanthrene	0.01	0.2	5	2.5	1
Anthracene	0.01	0.2	5	1	1
Fluoranthene	NA	0.4	10	3.5	1.5
Pyrene	· 0.01	0.2	5	1	1
Benz(a)anthracene ³	0.01	0.2	5	1	1
Chrysene ³	0.01	0.2	5	1	1
Benzo(b)fluoranthene ³	0.02	0.4	10	1	· 1
Benzo(k)fluoranthene ³	0.01	0.2	5	1	1
Benzo(a)pyrene ³	0.01	0.2	5	1	1.5
Dibenz(a,h)- anthracene ³	0.02	0.4	10	1.5	1.5
Benzo(g,h,i)perylene ³	0.02	0.4	10	1	1
Indeno(1,2,3- c,d)pyrene ³	0.01	0.2	5	1	1

¹ Fluorescence detector

⁴ MGP - Manufactured Gas Plant

² Photo-diode array detector

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*mg PAH/kg soil

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³ CPAHs

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Environmental Biotechnology using an HPLC equipped with florescence and photo-diode detectors. Matrix spikes and blanks will be analyzed in at least 10 percent of the samples collected for PAH and BTX analysis. These samples will be analyzed to determine the method recovery efficiency.

Total solids (TS) and volatile solids (VS) measurements will be made in accordance with Standard Method 2540G (Clesceri, et.al., 1989). This method is applicable to determining TS and VS fractions in solid and semisolid samples.

Aqueous-phase ammoniacal nitrogen will be determined using an ion-selective electrode method (Standard Method 4500-NH₃ F). Analysis of ortho-phosphate will be completed using the ascorbic acid Standard Method 4500-P E. A Standard Operating Procedure (SOP) for ortho-phosphate analysis is provided in Appendix C.

Total organic carbon (TOC) measurements in the aqueous phase will be made using a Dohrmann TOC Analyzer. The persulfate-ultraviolet oxidation Standard Method 5310 C is used for this determination. A BAC SOP for this measurement is shown in Appendix C.

The total heterotrophic microbial enumeration analyses will be performed by the IT BAC in Knoxville, Tennessee and will follow the SOP for plate count techniques. To assess the activity of the PAH-degrading bacteria, activity against anthracene will be determined by spraying selected plates with a 0.5 percent anthracene solution (acetone as the carrier). The carrier evaporates leaving a white anthracene film on the surface of the plate. As bacterial colonies metabolize the anthracene, clear zones are observed around the colonies. Anthracene was chosen because it is a general indicator of activity against PAH. The spray plate method is not useful for higher molecular weight PAH.

The slurry-phase pH will be determined using Standard Method 4500-H⁺ B. The oxygen concentration in the slurry will be determined using a modified, galvanic-cell, oxygen probe (Graves, et al., 1992).

Table 9 presents the precision/accuracy and detection limits expected for each analytical method. The stated detection limits may be altered if analytical interferences are present. Table 10 summarizes all methods used during the treatability studies.

6.1 Air Monitoring

Air sampling will be conducted following guidelines of U.S. EPA Method 18 and National Institute of Occupational Safety and Health (NIOSH) Method 5506 for measurement of PAH. The air sampling train will consist of a TeflonTM probe, a 47millimeter (mm) TeflonTM membrane filter, and an XAD-2 sorbent sampling tube. The filter and the XAD-2 tube will be connected with a minimal length of TeflonTM tubing (1 to 2 inches). The XAD-2 tube will be a prepared glass tube with XAD-2 resin packed in two portions. The front and back portions will contain 150 and 75 milligrams (mg) of XAD-2, respectively. The glass tube ends will be flame sealed.

Sample volume will be measured using a 2 liters per minute (L/min) Singer dry gas meter. The sample will be collected at a constant rate of 250 cubic centimeters per minute (cm³/min) for 24 hours. The detection limits for the semivolatile compounds will be determined during the course of the investigation. Analyses will be conducted by gas chromatography/mass spectrometer (GC/MS) in accordance with the procedures of Method 8270 of <u>Test Methods for Evaluating Solids Wastes</u>, <u>Physical/ Chemical</u> <u>Methods</u>, U.S. EPA SW 846.

A U.S. EPA Method TO-14 sampling system will be used to measure the volatile organics. The air sampling system used to collect volatile off-gas samples will consist of a stainless steel SummaTM polished canister, Milaflow pneumatic flow controller (10 cm³/min), and nickel tubing. SummaTM polished canisters have been used in several ambient studies to collect samples for volatile organic carbon (VOC) analyses. These studies have shown that the canisters are well suited for collecting air samples for VOC analysis and that VOC levels do not deteriorate in the canisters during reasonable holding times.

TABLE 9

Analytical Method Precision/Accuracy and Detection Limits

Method	Precision Solids	Accuracy (%) Aqueous	Detection Limit mg/L
PAH	+20	<u>+</u> 10	TBD
втх	<u>+</u> 20	<u>+</u> 10	TBD
Nutrients	<u>+</u> 10	<u>+</u> 10	0.5
pH	0.5 SU	0.5 SU	NA
Oxygen	<u>+</u> 10	<u>+</u> 10	0.1
TOC	<u>+</u> 10	<u>+</u> 10	0.1
TS/VS	<u>+</u> 30	<u>+</u> 10	NA
Microbial counts	<u>+</u> 20	<u>+</u> 20	10 ³ CFU/mL

Note:

NA - Detection limit not applicable to the analysis.

TBD - To be determined for the subject waste stream.

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SU - Standard units.

TABLE 10Analytical Methods

Analyses	Method	
TOC	BAC SOP	
РАН	Modified EPA Method 8310	
ВТХ	Modified EPA Method 8020	
Ammoniacal Nitrogen	Standard Method 4500-NH ₃ F	
Ortho-Phosphate	Standard Method 4500-P E	
TS/VS	Standard Method 2540 G	
Plate Counts	BAC SOP	
Air Sampling	EPA Method 18/NIOSH Method 5506	
Ha	Standard Method 4500-H ⁺ E	
Oxygen	Modified, galvanic cell	

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Canister samples will be analyzed in a Finnigan Model OWA 1050 GC/MS system with a quadruple MS. This system will be equipped with a Tekmar Model 5000 cryogenic concentrator and sample introduction system.

7.0 Data Management/Quality Assurance

The generation of valid data required for full-scale design must be accomplished through an established quality assurance/quality control (QA/QC) program. IT Corporation (IT) has developed and implemented a formal QA Program to provide direction for corporate operations so they will be performed in a controlled manner. This program, established in 1973, operates in compliance with the Code of Federal Regulations (CFR), 10 CFR 50, Appendix B; American National Standards Institute/American Society of Mechanical Engineers (ANSI/ASME) NQA-1 "Quality Assurance Program Requirements for Nuclear Facilities;" and current United States Environmental Protection Agency (U.S. EPA) guidelines and recommendations (e.g., QAMS-005/80, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans"). The purpose of the program is to establish policies that facilitate the implementation of regulatory requirements and to provide internal means for control and review, thus ensuring that the work performed by IT complies with all requirements.

Site-specific QA/QC procedures will be in accordance with the following documents:

- · IT Engineering Operations QA Manual, Revision 1, July 1, 1987.
- U.S. EPA, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," QAMS-005/80.
- · IT Analytical Services QA Manual, Revision 1, February 1, 1988.

QA/QC procedures prescribed in the U.S. EPA methods will be used in all analyses. Records of all analyses will be recorded in a bound laboratory notebook dedicated solely to this investigation.

The quality level for these investigations is Level III, as described in "Guide for Conducting Treatability Studies Under Comprehensive Environmental Response, Comprehensive and Liability Act (CERCLA)" EPA/540/2-89/058. This level was selected to provide the highest quality analytical results without using Certified Laboratory Procedures (CLP).

7.1 Data Acquisition

Measuring and test equipment used in the field or laboratory will be subject to a formal calibration program as described in Section 5.5 of IT's <u>Environmental Projects Group</u> <u>Engineering Operations OA Manual</u>. Calibration of field and laboratory equipment will be documented on the appropriate equipment calibration records. Equipment that fails calibration will be taken out of service, and a Notice of Equipment Calibration Failure record will be completed. Records of equipment calibration will be maintained in the project file located in Knoxville, Tennessee.

Sampling of all experiments and tests will be conducted by IT personnel. Sampling will be performed in accordance with Chapter 5.0 of IT's <u>Environmental Projects Group</u> <u>Engineering Operations OA Manual</u>. Sampling activities conducted by IT will be documented in the Sample Collection Log and on the Chain-of-Custody/Request-for-Analysis form. Variances will be granted by the project manager and documented on a Variance Log. Examples of these documents are presented in Appendix B.

7.2 Data Collection Sheets

Data collected during execution of the study will be recorded in a bound, controlled laboratory notebook. Data generated from integrators and computerized instruments will be printed with the resulting data sheets kept with the project file. All data will be verified and checked by a Biotechnology Applications Center (BAC) scientist. Proof of verification is the dated signature of the checker at the bottom of each notebook page.

7.3 Data Reduction, Validation, and Reporting

Data reduction, validation, and reporting will be in accordance with the requirements contained in the following:

- IT Engineering Operations QA Manual, Revision 1, July 1, 1987, "Modeling and Design."
- IT Analytical Services QA Manual, Revision 1, February 1, 1988, Chapter 10.0.

Numerical analysis will be performed in accordance with the implementation methods described in these documents, as amplified or modified by the following discussion of numerical/analysis activities of particular significance.

To provide evidence of satisfactory work performance and the basis for information presented in the Technical Memorandum, numerical analyses and results will be completely documented. Documentation may include calculations, computer programs, and associated input/output logs, drawings, and tables. Analysis activities will be performed in a planned and controlled manner. Documented and approved work instructions will be employed when appropriate.

Calculations will be legible and in a form suitable for reproduction, filing, and retrieval. Documentation will be sufficient to permit a technically qualified individual to review and understand the calculations and to verify the results. Calculations will be performed on standard calculation paper or laboratory notebooks whenever possible. Data validation will be performed in accordance with the IT QA Manual, Section 6.2.1.

Computer programs will be documented in sufficient detail to satisfy requirements, needs, and intended use of the program. Computer program validation requirements are contained in the IT QA Manual, Section 6.2.2.

7.4 Internal QC Checks

QA audits and surveillances will be performed in accordance with the requirements contained in the following:

- · IT Engineering Operations QA Manual, Revision 1, July 6, 1990, Chapter 11.0.
- IT Analytical Services QA Manual, Revision 1, February 1, 1988, Chapter 14.0.

QA audits and surveillances will be performed in accordance with the implementation methods described in these documents, as amplified or modified by the following discussion of QA audits and surveillance activities of particular significance. Monthly surveillances of the IT Knoxville central files will be performed by an IT QA Officer (QAO).

7.5 Corrective Action

Nonconformance identification, reporting, disposition, corrective action performance, verification, and acceptance will be in accordance with the requirements contained in the following:

- · IT Engineering Operations QA Manual, Revision 1, July 1, 1987, Chapter 8.0.
- · IT Analytical Services QA Manual, Revision 1, February 1, 1988, Chapter 13.0.

Nonconformance identification, reporting, disposition, corrective action performance, verification, and acceptance will be performed in accordance with the aforementioned documents, as amplified or modified by the following discussion of nonconformances and corrective action activities of particular significance.

Items, services, or activities that do not meet Test Plan requirements or IT accepted standard practices are to be reported as nonconformances if they fit any of the following criteria:

- · Are detrimental to quality if not corrected immediately
- Are detrimental to quality if not ultimately corrected, are not corrected immediately, and are not tracked by a system that will ensure their correction in a timely manner
- Are of a nature such that others can benefit from learning of the circumstances and a Nonconformance Report is judged the most appropriate way to communicate this information
- Pertain to items that should be segregated or visually identified to preclude inadvertent use
- Are significant conditions adverse to quality in which case they require reporting so that the cause of the condition can be determined and appropriate corrective action taken to preclude recurrence
- Are equipment that fails calibration or becomes impaired during use and is not immediately repaired.

The purpose of nonconformance reports is to prevent the installation of items, the use of services, or the performance of activities that are detrimental to quality and also to provide their correction. Nonconformance reports also disseminate "lessons learned" information so that appropriate preventive measures can be taken by others to avoid repeating mistakes. Finally, nonconformance reports can prove invaluable by identifying the need for and triggering the initiation of corrective actions to preclude recurrence of significant adverse conditions to quality.

Each nonconformance affecting quality will be documented, normally by the personnel that identifies or creates it. For this purpose, a Nonconformance Report form (Appendix B), IT Analytical Services (ITAS) laboratory nonconformance memorandum forms, or audit report will be used as appropriate. The Project Manager will determine the appropriate corrective measures to be taken and obtain the concurrence of the QAO.

Nonconformances identified within IT analytical laboratories will be reported in accordance with Chapter 13.0 of the ITAS QA Manual. Laboratory nonconformances identified during routinely-performed audits will be reported in accordance with Chapter 14.0 of the aforementioned document. The QAO will be provided with copies of nonconformance reports and audit reports that identify nonconformances adversely affecting the quality of the data.

Nonconformances identified during audits conducted by, or under the direction of, the QAO will be reported in accordance with Chapter 11.0 of the IT Engineering Operations QA Manual. All other nonconformances are to be reported on a Nonconformance Report (Appendix B). The person(s) identifying or creating a nonconformance is responsible for reporting it, or he/she must ensure that it will be reported by someone else. The issuance of the report is also verified by the person identifying or creating the nonconformance. The nonconformance will be reported directly to the IT Project Manager. Upon evaluation of the severity of the nonconformance and its impact on the project, the IT Project Manager will relay the information to the client. Nonconformances will be identified in the Technical Memorandum as deviations from the Test Plan.

Deviations from the analytical accuracy and precision reported in Table 9 will be handled as nonconformances and reported directly to the Project Manager. The Project Manager will take action to correct the situation, such as repeating the analysis or reviewing analytical procedures. Analytical nonconformances will be reported to the client and identified in the Technical Memorandum.

8.0 Data Analysis and Interpretation

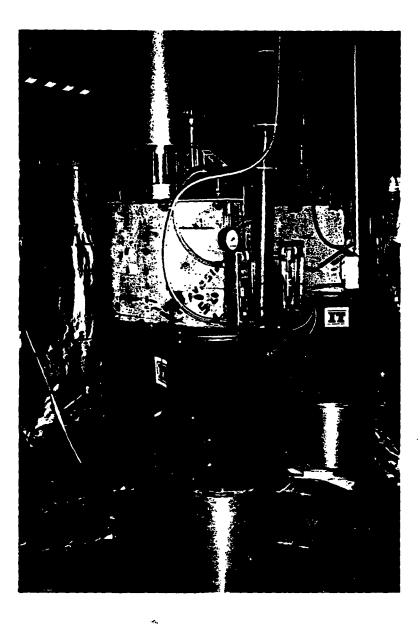
Previous studies conducted by IT Corporation's (IT) Biotechnology Applications Center (BAC) using bioslurry reactors have generated performance data required for the evaluation of treatment alternatives. IT conducted an internally-funded, pilot-scale investigation to determine the biodegradability of petroleum tank bottom sludges. The 2-month investigation was conducted at the United States Environmental Protection Agency (U.S. EPA) Test and Evaluation Facility (T & E) in Cincinnati, Ohio utilizing two 64-liter (L), stainless-steel, Eimco Biolift[™] slurry reactors similar to the 60-liter reactor proposed for this investigation (Figure 3). Table 11 illustrates the organic constituent removal efficiency demonstrated during the investigation.

In addition to the internally-funded investigation, IT was involved in the operation and maintenance of the U.S. EPA T&E reactors during an investigation evaluating the bioremediation of creosote-impacted soils regulated under Superfund (Table 12). The pilot-scale reactors were operated in batch for a period of 12 weeks. During the course of the investigation, bioaugmentation and surfactant addition (Tween 80) were evaluated.

Currently, the BAC is operating 1-L bench-scale, bioslurry reactors to evaluate varying treatment regime's impact on the bioremediation of contaminated soils collected from a Manufactured Gas Plant (MGP) site. During the course of the study nine treatment scenarios will be evaluated. Results from the first three treatments are presented in Table 13. Analytical techniques described in Chapter 6.0 were used to generate this data.

In addition to bioslurry investigations, the BAC has completed numerous treatability studies to determine the biodegradation of polycyclic aromatic hydrocarbons (PAH). These projects have included tasks requiring the evaluation of in situ aquifer remediation of coal-coking waste lagoons, determination of the effect of surfactant addition on PAH

FIGURE 3



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Organic Constituent	Reactor 1 Removal Efficiency	Reactor 2 Removal Efficiency
<c8< td=""><td>100</td><td>100</td></c8<>	100	100
C8 - C12	98	97
C12 - C14	60	14
C14 - C18	53	0
>C18	30	0
трн	61	10

TABLE 11 Organic Constituent Removal During Crude Oil Remediation

Note:

C8 - Compounds containing 8 carbon atoms.

C12 - Compounds containing 12 carbon atoms.

C14 - Compounds containing 14 carbon atoms.

C18 - Compounds containing 18 carbon atoms.

TPH - Total petroleum hydrocarbons.

Note:

Reactor 1 was the control.

Reactor 2 was bioaugmented.

TABLE 12

Bioremediation of Creosote-Impacted Soil

Weeks of Operation	Total PAH Percent Removal	2 to 3 Ring PAH Percent Removal	4 to 6 Ring PAH Percent Removal
2	89.3 <u>+</u> 3.9	95.9 <u>+</u> 1.8	81.58 <u>+</u> 6.7
12	93.4 <u>+</u> 3.2	97.1 <u>+</u> 2.2	89.06 <u>+</u> 4.8

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TABLE 13

PAH Removal in 1-Liter Bioslurry Reactors

COMPOUND	PERCENT REMOVAL REACTOR 1	PERCENT REMOVAL REACTOR 2	PERCENT REMOVAL REACTOR 3
Naphthalene	84.23	84.36	93.39
Acenaphthylene	83.60	69.34	91.25
Acenaphthene	68.23	50.26	79.72
Fluorene	68.94	60.88	89.87
Phenanthrene	61.66	57.71	89.61
Anthracene	63.07	55.26	85.26
Fluoranthene	61.98	58.57	77.56
Pyrene	61.11	50.47	51.42
Benz(a)anthracene ¹	61.26	77.77	70.83
Chrysene ¹	61.05	63.11	65.66
Benz(b)anthracene ¹	62.20	77.85	73.42
Benz(k)anthracene ¹	60.98	76.83	70.07
Benzo(a)pyrene ¹	61.61	90.61	69.37
Dibenz(a,h)anthracene'	51.72	45.62	43.22
Benzo(g,h,i)perylene1	51.72	45.62	43.22
Indeno(1,2,3- c,d)pyrene ¹	51.72	45.62	43.22

¹ - CPAHs

Note:

Reactor 1 was unfertilized control.

Reactor 2 was fertilized plus surfactant and co-metabolite

Reactor 3 was fertilized plus surfactant, co-metabolite, and bioaugmented.

biodegradation kinetics, evaluation of in situ vadose-zone PAH bioremediation, and respirometric evaluation of the effect of surfactant addition on the removal of ultimate biological oxygen demand of PAH-impacted soils. All data generated during the course of these investigations was evaluated to determine the feasibility of applying specific remedial strategies to full-scale remediation.

All data generated during the course of the batch slurry and bioslurry reactor studies for the Moss-American site will be stored on a spreadsheet, with hard copies maintained in two separate filing areas. The batch slurry study data will be analyzed to determine the extent of biodegradation in the various treatments. The preliminary substrate utilization rates determined during the completion of the batch slurry study will be used to confirm the selection of hydraulic retention time (HRT) and biological solids retention time (BSRT) set points for bioslurry reactor operation. In addition, performance data will help estimate the quantity of substrate that can be applied to the bioslurry system and allow for maximum removal efficiency.

Performance data, generated during steady-state operations, will also be evaluated to determine the operating parameters which generate optimum effluent quality. Steady-state operation is defined as the period of operation during which the rate of contaminant removal is constant. The operating parameters considered during this investigation include HRT, BSRT, pH, dissolved oxygen, and agitation.

Reactor design based on performance data determined during bench- and pilot-scale investigations is routinely demonstrated in bioslurry reactor systems, as well as conventional activated sludge systems (Field and Wojtanowicz, 1989; Engelder, et al., 1990; Marks, et al., 1991). The BAC has effectively generated reliable treatability data required for design.

9.0 Health and Safety

9.1 Hazard Analysis

The creosote-impacted soils used in this study contain polycyclic aromatic hydrocarbons (PAH), including benzene and naphthalene. Appendix D contains the Material Safety Data Sheets (MSDS) for these two materials.

Creosote is a yellow to black liquid with a tarry odor. It is a combustible liquid with a flashpoint of approximately 160°F. Exposure to creosote vapors may cause moderate irritation of the nose and throat. Liquid contact may cause severe eye burns, and reddening and itching of skin. Prolonged contact with skin may cause second-degree burns.

The benzene soluble fraction of creosote is carcinogenic, and repeated exposure has been associated with an increased risk of developing cancer of the lungs, skin, bladder, and kidneys. Pregnant women may be especially susceptible to exposure effects associated with creosote volatiles.

The Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for creosote, benzene-soluble fraction, is 0.2 milligrams per cubic meter (mg/m^3) of air based on an 8-hour exposure. The major potential routes of exposure to PAH are respiratory via inhalation of vapors and skin absorption via skin contact with the waste or waste-contaminated equipment.

The task involving the greatest potential exposure to PAH is handling and transferring the waste into both the batch and bioslurry reactors. Other tasks involving potential exposure to PAH are sample collection/handling and decontamination of equipment. Engineering controls will be utilized to reduce or eliminate the potential for exposure to vapors. The engineering controls include use of laboratory exhaust hoods for all sample

preparation and the use of carbon adsorption on the exhaust of the sealed bioslurry reactor.

Copies of the MSDS will be distributed to all personnel working on this project for review. Additionally, the MSDS will be posted near the bioslurry reactor, where personnel have access to the hazard information before entering the project area.

9.2 Personal Protective Equipment

The level of personal protective equipment (PPE) used during the charging of the bioslurry reactor will be determined following an assessment of screening activities. If the exposure monitoring indicates that contaminants are present at one-half the PEL, then Level C PPE will be used. If the contaminant concentration is less than one-half the PEL or not detected, Level D PPE will be employed.

If Level C PPE is determined to be appropriate for handling and transferring waste into the reactor or decontaminating the interior of the reactor or equipment, then it will be employed. Level C PPE will consist of:

- Full-face air-purifying respirator with organic vapor high-efficiency particulate air (HEPA) cartridges
- · Viton gloves outer; latex gloves inner
- Rubber apron or polyethylene-coated Tyvek coveralls
- Work uniforms
- Steel-toed shoes.

Level D protection will be used for activities conducted within the laboratory exhaust hood. If Level D protection is shown to be appropriate for the treatability tasks, it will be employed. Level D PPE will include:

- Safety glasses with side shields (goggles when collecting liquid samples)
- Viton gloves outer; latex gloves inner (when collecting samples)
- Steel-toed shoes
- · Laboratory coat or polyethylene-coated Tyvek coveralls

9.3 Respiratory Protection Program

A comprehensive respiratory protection program has been established by IT Corporation (IT). This program is mandated in all locations where use of such equipment is intended to lessen the potential for adverse health affects to an employee.

As part of the respiratory training program, each employee is instructed in the following elements:

- Nature of the respiratory hazard on the work site and the appraisal of potential consequences if the respiratory protection is not utilized
- Use and proper fit of the respirator
- · Cleaning, disinfecting, inspecting, maintenance, and storing of the respirator
- · Proper selection, capabilities, and limitations of PPE.

Routinely used respiratory equipment will be inspected, cleaned, and disinfected daily to help ensure proper hygiene practices. An inspection of these breathing devices will include the following:

- Examination of the head straps for breaks, loss of elasticity, broken or malfunctioning buckles, and other attachments
- Examination of the facepiece for excessive dirt, cracks, tears, distortion, holes, or inflexibility
- Examination of the exhalation and inhalation valves for any foreign material, cracks, tears, or distortion in the valve. Additional checks will be made to inspect for proper insertion, defective valve covers, or improper installation
- Examination of air-purifying elements for incorrect cartridge, expired shelf-life of the cartridge, or cracks or dents in the cartridge or cartridge holder
- Examination of proper insertion of the cartridges into the facepiece and a check of the gaskets inside the cartridge holder.

When respiratory protection is required, respiratory cartridges will be changed daily. All respirators will be inspected prior to each day's use. If broken or malfunctioning parts are found during the cleaning process, these parts will be replaced or new respiratory equipment will be issued to the user.

The respiratory protective equipment will be stored in an area protected from any mechanical damage. The protection area will guard against dust, heat, excessive moisture, or damage by chemical contact. The storage area for the respirators should be in a readily accessible location.

The following guidelines apply to the use and storage of respirators.

- Only employees who have been trained to wear and maintain respirators properly will be allowed to use respiratory protection.
- Selection of respirators, as well as any decisions regarding upgrading or downgrading of respiratory protection, will be made by the health and safety officer or his designee.
- Positive and negative pressure tests will be performed each time the respirator is donned.
- Only employees who have been fit tested within the last 12 months will be allowed to work in atmospheres where respirators are required. Subcontractors will provide certificates of respirator fit tests completed within the last 12 months for each employee on site.
- · Respirator users will be instructed in the proper use and limitations of respirators.
- If an employee has difficulty in breathing during the fit test or during use, he will be evaluated medically to determine if he can wear a respirator safely while performing assigned tasks.
- No employee will be assigned to tasks requiring the use of respirators if, based upon the most recent examination, a physician determines that the health or safety of the employee will be impaired by respirator use.

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- · Contact lenses will not be worn while using any type of respiratory protection.
- · Respirators will be cleaned and sanitized daily after use.
- Respirators will be stored in a convenient, clean, and sanitary location on site.
- Respirators will be inspected during cleaning. Worn or deteriorated parts will be replaced.
- Facial hair that might interfere with a good facepiece seal or proper operation of the respirator is prohibited.
- The IT project manager will review the respiratory protection program to ensure that employees are properly wearing and maintaining their respirators and that the respiratory protection is adequately protecting the employees.
- The health and safety officer and the project manager will evaluate the respiratory protection program routinely to ensure the continuing effectiveness.
- Respirators used for emergency response will be inspected weekly by the health and safety coordinator.

9.4 General Work Practices

The following work practices will be adhered to during the course of project activities. At least one copy of these procedures will be available at the treatability study work site.

- Contaminantd protective equipment, such as respirators, hoses, boots, etc., will not be removed from the regulated work area until it has been cleaned or properly packaged and labeled.
- Legible and understandable precautionary labels that display identity and appropriate hazard warning will be prominently affixed to containers of contaminantd scrap, waste, debris, and clothing.
- Removal of PAH-contaminantd material from protective clothing or equipment by flowing, shaking, or any other means that disperse contaminantd material into the air is <u>prohibited</u>.

- No food or beverages will be present or consumed in the treatability study work area.
- No tobacco products will be present or used, and cosmetics will not be applied in the treatability study work area.
- Employees will wash their hands and face before eating, drinking, smoking or applying cosmetics.
- PAH-contaminantd materials will be stored in tightly-closed containers in wellventilated areas.
- Containers will be moved only with the proper equipment and will be secured to prevent dropping or loss of control during transport.
- Emergency equipment will be located outside storage areas in readily accessible locations that will remain minimally contaminantd with PAH.
- All areas that have been determined as uncontaminated inside the regulated area will be clearly marked as such. No personnel, equipment, etc. will be in these areas until they have been decontaminated.

9.5 Personnel Training

All personnel at the Biotechnology Applications Center (BAC) facility receive at least 40 hours of OSHA health and safety training. OSHA training includes a minimum of 24 hours of initial off-site training and a minimum of 8 hours annual refresher training. This includes instruction on exits, fire extinguishers, handwashing, safety showers, and eye wash stations. Supervisors receive an additional 8 hours of health and safety training. All personnel also receive 8 hours annual health and safety training, which meets the requirements of OSHA regulations included in 29 CFR 1910.120. Only personnel who have had qualitative fit tests and annual fit tests thereafter will be allowed to work in areas where respirators are required.

Upon BAC receipt of the creosote-impacted soils, a hazards communications meeting will be held to inform employees of project-specific contaminants and the project technical scope of work.

9.6 Medical Surveillance

A preassigned health assessment will be required for all personnel working with toxic substances. This examination will include a previous work medical history. It will be followed by annual medical examinations, which will update and document any accidental exposures. All IT employees participate in an annual medical surveillance program. This medical surveillance program meets the requirement of the OSHA regulations included in 29 CFR 1910.120.

9.7 Spill Prevention and Containment

The primary spill prevention method that will be enforced throughout this project will minimize the quantity of toxic materials used for experimentation. Any visible quantity of spilled liquid (slurry) waste from the reactor operations must be cleaned up immediately with spill-absorbing pads located in the work area. These pads will be collected in sealable cans and stored for disposal. After the visible quantity is absorbed, the contaminated work surface will be wiped repeatedly with water-soaked rags and dried. Spills on concrete will be absorbed with a sweeping compound.

Major spills, fire, or explosions will necessitate response in accordance with the IT BAC Chemical Hygiene Plan (CHP). The laboratory CHP is maintained in an area readily accessible to all employees. If an emergency situation arises, the first duty of project personnel is to alert all affected personnel and then contact the facility emergency coordinator.

At the end of the pilot-scale investigation, any remaining liquids or solids will be poured into the waste container supplied by project personnel. The waste container will be properly identified as a satellite waste collection container and labeled for the type of waste it contains with an appropriate hazard warning. Questions on the proper disposal method should be directed to the appropriate project personnel.

10.0 Residuals Management

All waste received or generated during the course of the investigation will be properly containerized, labeled, and shipped to the Moss-American site for storage with other predesign activity residuals, pending final disposal. Following the completion of the batch slurry study, all wastes will be combined and shipped to the site for storage.

The residual streams created during bioslurry remediation include treated solids, process water, and possible air emissions. Treated solids will be containerized, labeled, and stored at 4°C for shipment to the site where they will be stored with other predesign activity residuals, pending final disposal. The majority of the process water will be recycled during the study; excess amounts will also be containerized, labeled, and stored at 4°C for return to the site. Possible air emissions from the reactor will be controlled by carbon adsorption. Spent carbon adsorption materials will be containerized with the test matrix and returned to the Moss-American site for staging with other predesign wastes and residuals.

The shipment of all wastes and treatment residuals will be done in compliance with applicable Department of Transportation (DOT) regulations.

11.0 Reports

A Technical Memorandum (TM) will be issued describing the Phase I batch and bioslurry reactor treatability studies. The schedule for TM submittal is presented in Figure 4. Table 14 illustrates the tentative organization of the TM. The preparation of the TM will adhere to the standards described in United States Environmental Protection Agency (U.S. EPA), "Guide for Conducting Treatability Studies Under Comprehensive Environmental Response, Comprehensive and Liability Act (CERCLA)," EPA/540/2-89/058.

Moss-American Site Treatability Test Plan Date: 29 May 1992 Revision: 0

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FIGURE 4

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Project Schedule for Phase I Biological Slurry Treatability Studies Moss-American Site Milwaukee, Wi

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TABLE 14

Technical Memorandum Outline

- 1.0 Introduction
 - 1.1 Site Description
 - 1.1.1 Site Name and Location
 - 1.1.2 History of Operations
 - 1.1.3 Prior Removal and Remediation Activities
 - 1.2 Waste Description
 - 1.2.1 Waste Matrices
 - 1.2.2 Pollutants/Chemicals
 - 1.3 Remedial Technology Description
 - 1.3.1 Treatment Process and Scale
 - 1.3.2 Operating Features
- 2.0 Conclusions and Recommendations
 - 2.1 Conclusions
 - 2.2 Recommendations
- 3.0 Treatability Study Approach
 - 3.1 Test Objectives and Rationale
 - 3.2 Experimental Design and Procedures
 - 3.3 Equipment and Materials
 - 3.4 Sampling and Analysis
 - 3.4.1 Waste
 - 3.4.2 Treatment Process
 - 3.5 Data Management
 - 3.6 Deviations from the Test Plan
- 4.0 Results and Discussion
 - 4.1 Data Analysis and Interpretation
 - 4.1.1 Analysis of Waste Characteristics
 - 4.1.2 Analysis of Treatability Study Data
 - 4.1.3 Comparison to Test Objectives
 - 4.1 Quality Assurance/Quality Control
 - 4.2 Costs/Schedule for Performing the Treatability Study
 - 4.4 Key Contacts
- References

Appendices

- A Data Summaries
- B Standard Operating Procedures

12.0 Schedule

The project schedule for treatability activities is presented in Figure 4. The tasks with the greatest potential for time variance are test plan approval, agency documentation review, and the treatability tests.

12-1

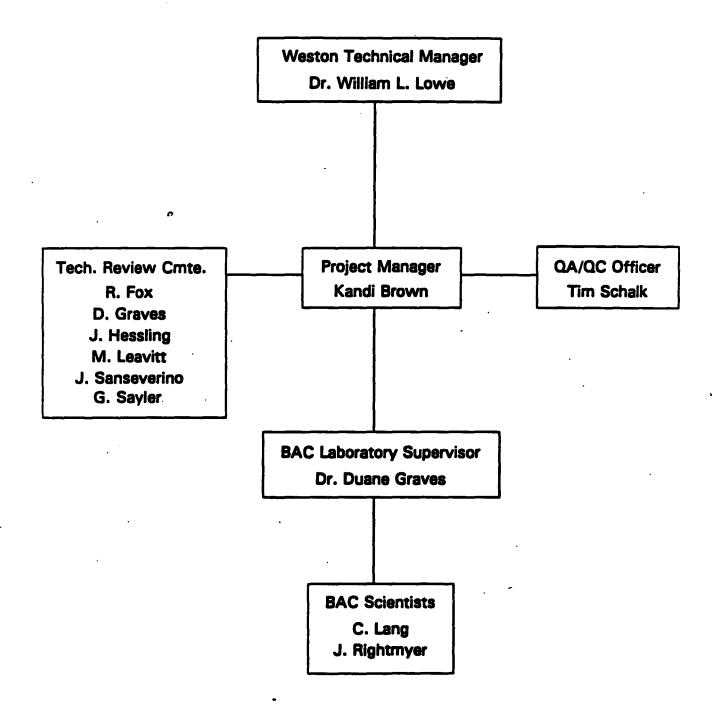
13.0 Management and Staffing

Figure 5 illustrates the organizational chart for this project. Kandi Brown, an IT Corporation (IT) Technical Associate, will serve as the project manager for the work based on her experience. She has had more than 5 years experience in designing and evaluating bioreactor systems. She has been project manager for all Biotechnology Applications Center (BAC) bioslurry reactor investigations utilizing slurry-phase reactors ranging in size from 1 to 64-liter (L). She is responsible to the client for performing the project according to requirements and conditions established in the contract. She is also responsible to IT for meeting specified safety, quality, and business goals, and to the project team members for defining their responsibility and authority within the project organization.

The BAC laboratory, where the treatability studies will be conducted, is managed by Dr. Duane Graves, an IT Technical Associate. Dr. Graves will be most actively involved in the preparation of the project Test Plan, review of project schedule, and allocation of required laboratory resources. Once the project is underway, he will routinely report treatability study activities to the Project Manager.

Primary technical personnel responsible for the execution of the treatability investigations will be Craig Lang and Janet Rightmyer. Craig Lang, an IT Technical Associate, is a project scientist with more than 8 years of experience in methods development, feasibility and treatability testing, conceptual design, and field implementation of bioremediation projects. He will be the primary technician responsible for the set-up, sampling and analysis, and demobilization of the batch slurry testing. In addition, he will be responsible for polycyclic aromatic hydrocarbons (PAH) and benzene, toluene, xylene (BTX) analyses.

Figure 5 Project Organization Chart



Ms. Rightmyer has more than 2 years experience in the execution of bioreactor investigations. In addition, Ms. Rightmyer was the primary technician on the IT-funded, pilot-scale bioreactor investigation utilizing two 64-L Eimco Biolift[™] slurry reactors (Figure 3). She is proficient in the mobilization, demobilization, operation and maintenance of Eimco reactors. She will be responsible for the bioslurry reactor set-up, operation and maintenance, sampling and analysis, and demobilization.

All data generated during the course of the treatability studies will be reported directly to the Project Manager following the completion of appropriate Quality Assurance/Quality Control (QA/QC) review by the project Quality Assurance Office (QAO), Tim Schalk. Ms. Brown will be assisted in the evaluation of this data by a technical review committee with 70 years of combined experience in bioremediation and hazardous waste treatment. Members of the review committee include Robert Fox, an IT Distinguished Technical Associate, Dr. Gary Sayler, a leader in environmental applications of molecular diagnostic techniques, and Maureen Leavitt, BAC Project Technical Coordinator.

The project Technical Memorandum will be prepared by the Project Manager and reviewed by the technical review committee.

Moss-American Site Test Plan - Bioslurry Treatability Date: 29 May 1992 Revision: 0

14.0 Literature Cited

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U.S. EPA, "Guide for Conducting Treatability Studies Under CERCLA," EPA/540/2-89/058.

U.S. EPA, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," QAMS-005/80.

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APPENDIX A

SHIPPING DOCUMENTATION



ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD*

Reference Document No. 32300: Page 1 of ____

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APPENDIX B

QA/QC DOCUMENTATION

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PREPARED BY: _____

LEGEND

- 1. A SAMPLE COLLECTION LOG IS TO BE COMPLETED FOR EACH SAMPLE.
- 2. ALWAYS COMPLETE BOTH SIDES. IF SECOND SIDE IS NOT USED, DRAW A LINE THROUGH IT AND MARK N/A. FILL IN CONTROL BLOCK AND PREPARED BY.
- 3. ALL ENTRIES ON LOG ARE TO BE COMPLETED, IF NOT APPLICABLE MARK N/A.
- 4. DATE: USE MONTH/DAY/YEAR; I.E., 10/30/85
- 5. TIME: USE 24-HOUR CLOCK; I.E., 1835 FOR 6:35 P.M.
- 6. PAGE: EACH SAMPLE TEAM SHOULD NUMBER PAGE _____ OF ____ FOR THE DAY'S ACTIVITIES FOR ALL SHEETS PREPARED ON A SINGLE DAY, I.E., IF THERE ARE A TOTAL OF 24 PAGES (INCLUDING FRONT AND BACK) NUMBER 1 OF 24, 2 OF 24. ETC.
- 7. SAMPLE LOCATION: USE BORING OR MONITORING WELL NUMBER, GRID LOCATION (TRANSECT), SAMPLING STATION I.D., OR COORDINATE TO PHYSICAL FEATURES WITH DISTANCES. INCLUDE SKETCH IN COMMENT SECTION IF NECESSARY.
- 8. SAMPLE TYPE: USE THE FOLLOWING SOIL; WATER (SURFACE OR GROUND); AIR (FILTERS, TUBES, AMBIENT, PERSONNEL); SLUDGE DRUM CONTENTS; OIL; VEGETATION; WIPE; SEDIMENT.
- 9. COMPOSITE TYPE: I.E., 24-HOUR, LIST SAMPLE NUMBERS IN COMPOSITE, SPATIAL COMPOSITE.
- 10. DEPTH OF SAMPLE: GIVE UNITS, WRITE OUT UNITS SUCH AS INCHES, FEET. DON'T USE ' OR ".
- 11. WEATHER: APPROXIMATE TEMPERATURE, SUN AND MOISTURE CONDITIONS.
- 12. CONTAINERS USED: LIST EACH CONTAINER TYPE AS NUMBER, VOLUME, MATERIAL (E.G., 2 IL GLASS; 4 40 ML GLASS VIAL: 1 400 ML PLASTIC; 1 3 INCH STEEL TUBE; 1 8 OZ. GLASS JAR).
- 13. AMOUNT COLLECTED: VOLUME IN CONTAINERS (E.G. 1/2 FULL).

APPENDIX C

BIOTECHNOLOGY APPLICATIONS CENTER STANDARD OPERATING PROCEDURES



IT Biotechnology Applications Center Microbial Enumeration Analysis Standard Operating Procedure

NUMBER: BAC009

Approved By:

3/92

Duane Graves, Ph.D. (Date) Process Development Supervisor

3/92 James J. King (Date)

General Manager/QA Officer

KLB/03-92/SMC/enumer.sop

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SOP No. BAC009 Revision No. 0 Date: 3-9-92 Page 1 of 3

STANDARD OPERATING PROCEDURE

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MICROBIAL ENUMERATION ANALYSIS

1.0 Principle

Viable bacterial cells of sufficient quantity are required for effective bioremediation. This test permits the quantification of bacteria from natural environments. Heterotrophs or specific contaminant degrading bacteria can be enumerated. Bacterial density is reported as colony-forming units (CFU) per milliliter (mL) of water or gram (g) of dry soil. A CFU is assumed to represent one bacteria.

2.0 Equipment

- Carbon-free mineral salts agar plates
- Dilute nutrient agar plates
- Sterile 1 percent sodium pyrophosphate and 0.1 percent polyvinylpyrrolidone-360 (PVP) in deionized (DI) water
- Sterile 10-mL dilution tubes
- 1-mL sterile disposable pipettes
- Waring blender with steel container
- Sterile 50-mL screw cap tubes
- Alcohol
- Glass plate spreader
- Petri dish turntable
- Dessicators

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Polynuclear aromatic hydrocarbon source

Quebec colony counter.

3.0 Procedure

- 1. Weigh 5 g of soil/sludge into the blender.
- 2. Add 45 mL of PVP.
- Homogenize by running the blender twice at high speed for 10 seconds with a 10-second rest interval between mixings. Decant mixture into 50-mL tube and seal.
- 4. Perform 10-fold serial dilutions on the homogenized mixture. Aqueous samples are not pretreated with PVP; they are plated as received. The dilution concentrations are determined by the anticipated concentration of bacterial concentrations within the sample. A five order of magnitude range of dilutions is plated.
- 5. The nutrient agar plates should be plated one order of magnitude higher than the corresponding mineral agar plates.
- 6. Using the glass plate spreader and turntable, 0.1 mL of the appropriate dilutions is plated on the two types of agar media .
- 7. Samples plated on the mineral salts agar are placed in the desiccators along with the appropriate carbon source. Samples plated on the nutrient agar are placed in a protected area away from additional carbon sources. Plates are incubated at 20°C.

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8. After the appropriate incubation time, the bacterial colonies are counted with the Quebec colony counter. Results are recorded as CFU per mL of groundwater or per gram of dry soil. The inoculated plates should be incubated the same number of days, approximately 3 to 7 days for nutrient agar and 7 to 14 days for mineral agar. The actual incubation time depends on the growth response of the bacteria.

4.0 Calculations

Water samples: Colony Count X Dilution Factor = CFU per mL Soil samples: (Colony Count X Dilution Factor) X Dry Wt)/Wet Wt = CFU/gm dry soil.

5.0 Interferences

None.

6.0 <u>OC Requirements</u>

Sterility testing of agar medium.



IT Biotechnology Applications Center Phosphate Analysis Standard Operating Procedure

NUMBER: BAC015

Approved By:

52

Duane Graves/ Ph.D. (Date) Process Development Supervisor

Jam Kina Date) General Manager/QA Officer

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SOP No. BAC015 Revision No. 0 Date: 3-8-92 Page 1 of 4

STANDARD OPERATING PROCEDURE

PHOSPHATE ANALYSIS

1.0 Principle

Ammonium molybdate and potassium tartrate react in an acid medium with dilute solutions of ortho-phosphate to form phosphomolybdic acid, which is reduced to the intensely-colored molybdenum blue by ascorbic acid. The phosphate analysis is utilized to determine the levels of phosphate present within the samples.

2.0 Equipment

- Bausch & Lomb Spectronic 1001 Spectrophotometer
- HACH PhosVer 3 powder pillows
- 20-milliliter (mL) vials
- 1-, 5-, and 10-mL pipettes
- Deionized (DI) water
- 2,000 parts per million (ppm) KH₂PO₄ standard
- 100-mL volumetric flasks.

3.0 Standards

Standards are generated from a potassium phosphate (KH_2PO_4) 2,000 ppm stock solution. This stock solution is prepared by dissolving 0.285 gram (g) of anhydrous KH_2PO_4 in 100-mL of DI water. This stock is then diluted as indicated in the table below to obtain the indicated concentrations.

KH₂P0₄	Stock DI	Water Conc. (ppm)
0.1 mL	100 mL	2.0
0.05 mL	100 mL	1.0
0.025 mL	100 mL	0.5

4.0 Procedure

- 1. Prepare a 0.5, 1.0, 2.0 ppm phosphate standards from the 2,000 ppm stock solution of KH₂PO₄.
- 2. Using a pipette, place 10 mL of each standard into a vial.
- 3. Add the contents of 1 PhosVer 3 powder pillow and swirl.
- 4. Allow color to develop for at least 10, but no longer than 30, minutes.
- 5. Measure the absorbance of a blank (10 mL of DI water with 1 PhosVer 3 powder pillow added) using the spectrophotometer at a wavelength of 700 nanometers (nm).
- 6. Measure the absorbance of the three standards on the spectrophotometer at a wavelength of 700 nm.
- 7. Calculate the linear regression curve of the standards and the blank using a programmed calculator.

8. If measuring a groundwater sample, take 10 mL of the sample and add the contents of 1 PhosVer powder pillow and swirl. If the absorbance is higher than the 2.0 ppm standard, then dilute as necessary with DI water. If the absorbance is lower than the 0.5 ppm standard, then the sample is below the

detection limit, and it should be reported as such.

- 9. If the sample is soil, then weigh out one dry scoop of soil in a glass jar with cap. Add 25 mL DI water and add 1 soil extractant pillow. Shake and let stand to separate. When separated, withdraw 1 mL of liquid and add 9 mL of DI water in a separate vial. Sample can be at higher dilutions, but the amount of sample needs to be 10 mL to react with the reagents added. Add 1 PhosVer 3 powder pillow, swirl, and run on spectrophotometer at 700 nm. This gives absorbance.
- 10. Use the curve generated to determine the concentration of the sample which is reported as mg/kg.

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5.0 Calculations

Calculation of the linear regression of the standards is required to determine concentrations of the samples. The curve generated from the standards is then used to determine sample concentrations.

6.0 Interferences

Interferences may be caused by chromium, nitrate, sulfide, and silicate. Interferences are determined by analyzing a 10-mL sample on the spectrophotometer that has been spiked with 0.01 mL of the 2,000 ppm phosphate stock solution. If a difference of greater than 10 percent is observed between the actual and calculated concentrations, interferences are present and dilution of the sample is required to obtain accurate data. Dilution ratios are as follows:

Sample Volume	Water Volume	Dilution Factor
2	8	5
1	9	10
0.5	9.5	20
0.1	9.9	100

7.0 Quality Control Requirements

Quality control (QC) requirements are satisfied through the preparation of blanks (10-mL DI water and 1 PhosVer powder pillow). If a series of samples are to be run, one out of every ten samples are to be blanks. If significant concentration are noted in the blank sample, this concentration is subtracted from the sample concentrations obtained.



IT Biotechnology Applications Center Carbon Analysis Using the Dohrmann Total Carbon Analyzer Standard Operating Procedure

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NUMBER: BACOO8

Approved By:

82

Duane Graves, Ph.D. (Date)
Process Development Supervisor

3/13/92 King James J (Date)

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SOP No. BAC008 Revision No. 0 Date: 3-9-92 Page 1 of 6

STANDARD OPERATING PROCEDURE

CARBON ANALYSIS USING THE DOHRMANN TOTAL CARBON ANALYZER (TCA)

1.0 Principle

Ultraviolet (UV) radiation promoted persulfate oxidation of organic compounds dissolved in acidic water results in the production of carbon dioxide. The carbon dioxide concentration is then measured as parts per million (ppm) by an infrared detector.

Sample Requirements and Preservation

2.0 Groundwater

- 10 milliliter (mL) of groundwater
- Sample preservation and containers: nonpreserved samples in nonsterile bottles, shipped on ice and stored at 4°C.

Holding time: no more than 3 days after receipt of groundwater samples.

3.0 Other Aqueous Samples

• Analyze at least 1 mL of samples as soon as possible after collection.

4.0 <u>Apparatus</u>

- Dohrmann TCA with automatic sampling unit
- Unimetrix side port syringes in 50-, 250-, and 1,000-microliters (µL) sizes.
- Use two syringes: one for samples and one for standards
- 5-mL Yale syringes
- 0.45-µm Gelman Acrodisc filters.

5.0 Reagents Acidified Persulfate Solution (reactor module fluid)

Add 1 mL of concentrated phosphoric acid to ~500 mL of deionized (DI) water in a 1-L volumetric and mix. Bring to 950 mL with DI water and mix. Add approximately 20 grams (g) (\pm 5 percent) of American Chemical Society (ACS)grade potassium persulfate to the acidified water, mix without heat, and bring to volume.

6.0 Carbon as Phthalate Standards

Add 0.5 mL of concentrated phosphoric acid to less than 500 mL of DI water; mix and bring to volume. Add 0.4255 g of ACS-grade potassium hydrogen phthalate into a 100-mL volumetric and bring to volume with acidified water. This is a 2,000 ppm C as phthalate standard. Add 20 mL of this solution to 80 mL of acidified water for 400 ppm carbon (C); add 1 mL of the 2,000 ppm C standard to 199 mL of acidified water for a 10 ppm C standard.

7.0 Carbon as Carbonate Standards

Dissolve 7.00 g of sodium bicarbonate (NaHCO₃) in less than 1 L of DI water and bring to volume. Do <u>not</u> use acidified water. This is a 1,000 ppm C as bicarbonate solution. Dilute 100X for a 10 ppm C standard.

8.0 Procedure

Total Carbon Analysis

- 1. Injection of the sample as received or unfiltered will result in a value known as the total carbon content. Sometimes it is desirable to know the dissolved carbon content instead, especially if the sample is particularly cloudy or if there is a layer of organics on the water. If total carbon content is desired, proceed to the next step. If dissolved carbon content is desired, flush a 0.45 μ m Gelman Acrodisc with 1-mL of DI water, purge with air, and then liter the sample prior to analysis.
- 2. Turn the power of the Dohrmann TCA on by first pressing the power button on the auto-sampler, then pressing the power button on the unit housing the infrared detector.
- 3. Engage the jaw clamp on the peristaltic pump. Place the feed tube (the thin tube with the black plastic tag) into a liter flask containing the acidified persulfate solution, and place the free end of the green tagged tube in the container marked waste. Then press, in the following order, the buttons marked POWER, PUMP, and LAMP. Ensure that the pump is drawing fluid from the flask containing the acidified persulfate.
- 4. Allow the instrument to warm up until baseline in reached. Baseline is established when the infrared detector, in DET mode, has ceased to change in reading. The baseline value may be adjusted by removing the front panel of the unit housing the detector, and turning the knob marked ZERO until the desired setting is reached (>0.05 and <0.10). Do not touch the knob marked SPAN.

- 5. Set the instrument to the desired injection volume, (40 μ L, 200 μ L, and 1 mL). Check to see if there a calibration on the instrument for that injection volume; the calibration button will be lighted if it is calibrated. If the instrument is calibrated go to Step 6; if it is not calibrated, go to Step 7.
- 6. To check the existing calibration, inject the proper volume of the appropriate standard (1 mL of 10 ppm C as phthalate, 200 μ L of 200 ppm C as phthalate, or 40 μ L of 2,000 ppm C as phthalate) into the injection port, immediately press the START button in the detector housing, and allow the oxidation to go to completion. The instrument will beep loudly when the run is complete. Compare the ppm C value that the instrument reports with that of the standard. If it is within 2 percent of the standard value, go to Step 8. Otherwise, the machine must be recalibrated. Decalibrate the instrument by pressing the calibration button for about 0.5 second (the light in the button will go out). Calibrate as per Step 7.
- 7. The instrument should be set on the desired injection volume. To calibrate the instrument, inject an aliquot of the proper concentration of standard, immediately press the start button, and obtain an area count value; this is given when the instrument is uncalibrated and in ppm C mode. (Note: This value is a raw data value; it will not be close to the ppm C value of the standard.) For a 1-mL injection, the raw data value should be 7.50 \pm 1.85 area counts; for a 200 μ L injection it should be 300 \pm 75 area counts; and for a 40 μ L injection it should be 1500 \pm 375 area counts. Repeat this process three times. The area count values obtained should be within 2 percent of each other, otherwise you must start the calibration over. This is done by pressing the calibration button twice-once for 0.5 second and then for 1.5 sec. If the calibration values are within 2 percent of each other, calibrate the instrument by pressing the calibration button for 1.5 sec or until the light in it comes on. The instrument is programmed to write "Suspect Data" when a calibration

value differs from its predecessors by more than 2 percent. The instrument calculates the deviation, but the user must check the data.

- 8. Once the instrument is calibrated, inject the proper amount of sample (based on the injection volume at which the instrument is set) into the injection port, immediately press the start button, and wait for the ready light to come on and the instrument to beep. If there is sufficient sample, repeat the injection.
- 9. After the sample analysis is completed, turn the instrument off by pressing buttons in the following order: lamp button, pump button and power button above the lamp button, power button on the detector housing, and the power button on the auto-sampler. Release the jaw clamp on the peristaltic pump, remove the feed tube, and cap the persulfate solution.

Inorganic Carbon Analysis

Inorganic carbon analyses are conducted in the same manner as total carbon analyses except the lamp is not turned on, and the instrument is calibrated with a 1 mL aliquot of a 10 ppm C as CO_{3-2} solution.

9.0 Calculations

None.

10.0 Interferences

Certain complex hydrocarbons are recalcitrant to UV-promoted persulfate oxidation. Dissolved heavy metals catalyze the decomposition of organic compounds to carbon dioxide and, therefore, may interfere with the analysis of inorganic carbon.

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11.0 Quality Control Requirements

Test accuracy of calibration by injecting an appropriate potassium phthalate standard. Recalibrate the instrument if error is greater than 25 percent.

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APPENDIX D

MATERIAL SAFETY DATA SHEETS

 $g_{1,1} = \frac{1}{2} g_{1,2} =$

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American B				••	•	
Material Sat	lety Data	_				
emergency telephone no		0 (American	Scientific	Products)		MATERIAL SAFETY
chemtrec telephone no	800/424-930					DATA SHEET
information telephone no.	616/726-317	1 (American	Burdick &	Jackson)		
I. Identification	Benzene			78.11		
chemical name	Aromatic Hy	drocarbon	molecular w	cigini		BENZENE
chemical family	Benzol	var ocar bon	formula	<u></u>		
synonyms DOT proper shipping name						
DOT hazard class	Flammable I	Liquid				
DOT identification no	UN1114		CAS no. 71	-43-2		
II. Physical and Che	mical Data				-	
boiling point, 760mm Hg.		_ freezing point	5.5	°C	evaporation rate	(BuAc=1) ca 3
vapor pressure at 20°C						@ 25°C 0.18%
% volatiles by volume					stability	
nazardous polymerization _		Not expect			· · · —	
azaluous porymenzation _				•		
					racteristic aro	matic odor.
oppearance and odor		Clear, colo Heat, spark	rless liquid s, open fla	with a char me, open co	ontainers, and	matic odor. poor ventilation.
appearance and odor conditions to avoid materials to avoid nazardous decomposition p		<u>Clear, colo</u> Heat, spark Strong oxid	rless liquid is, open flan izing agent combustion	with a chaine, open co	ontainers, and g acids.	
appearance and odor conditions to avoid materials to avoid	roducts	Clear, colo Heat, spark Strong oxid Incomplete toxic vapor	rless liquid is, open flat izing agent combustion s. closed cup	with a chain me, open co s and strong n can gener auto ignitic upper limit	ontainers, and g acids. ate carbon mo	poor ventilation.
appearance and odor conditions to avoid materials to avoid nazardous decomposition p III. Fire and Explosic lash point. (test method) larmable limits in air % by	roducts	<u>Clear, colo</u> Heat, spark Strong oxid Incomplete toxic vapor -11°C (Tag 1.3	rless liquid is, open flat izing agent combustion s. closed cup d flammable	with a chain me, open co s and strong n can gener auto ignitio upper limit a.	g acids. ate carbon mo	poor ventilation.
appearance and odor conditions to avoid materials to avoid hazardous decomposition p III. Fire and Explosion lash point. (test method) lammable limits in air % by inusual fire and explosion h	roducts	Clear, colo Heat, spark Strong oxid Incomplete toxic vapor -11°C (Tag 1.3 Volatile and Carbon diox Water will 1	rless liquid ss, open flat lizing agent combustion s. closed cup d flammable kide, dry ch	with a chain me, open co s and strong a can gener auto ignition upper limit emical or f ctive in ext	g acids. ate carbon mo	poor ventilation.
appearance and odor conditions to avoid naterials to avoid naterials to avoid nazardous decomposition p II. Fire and Explosion ash point. (test method) ammable limits in air % by nusual fire and explosion h	roducts	Clear, colo Heat, spark Strong oxid Incomplete toxic vapor -11°C (Tag 1.3 Volatile and Carbon diox Water will i spread it, b	rless liquid is, open flat izing agent combustion s. closed cup d flammable kide, dry ch not be effec ut a water	with a chain me, open co s and strong a can gener auto ignition upper limit a emical or f ctive in ext spray can b	ate carbon mo	poor ventilation.
appearance and odor conditions to avoid materials to avoid mazardous decomposition p II. Fire and Explosic lash point. (test method) lammable limits in air % by inusual fire and explosion h	roducts	Clear, colo Heat, spark Strong oxid Incomplete toxic vapor -11°C (Tag 1.3 Volatile and Carbon diox Water will i spread it, b containers.	rless liquid is, open flat izing agent combustion s. closed cup d flammable d flammable kide, dry ch not be effec ut a water Wear full	with a chain me, open constructions and strong can gener auto ignition upper limit a emical or f ctive in ext spray can b protective	g acids. ate carbon mo on temperature oam. inguishing a fi e used to cool clothing and s	poor ventilation. pnoxide and other 562°C 7.1 ire and may exposed elf-contained
appearance and odor conditions to avoid materials to avoid mazardous decomposition p II. Fire and Explosic lash point. (test method) lammable limits in air % by inusual fire and explosion h	roducts	Clear, colo Heat, spark Strong oxid Incomplete toxic vapor -11°C (Taq 1.3 Volatile and Carbon dios Water will i spread it, b containers. breathing a	rless liquid is, open flat izing agent combustion s. closed cup d flammable kide, dry ch not be effect ut a water Wear full pparatus.	with a char me, open co s and strong can gener auto ignition upper limit a emical or f ctive in ext spray can b protective leat will bu	ate carbon mo	poor ventilation. pnoxide and other 562°C 7.1 ire and may exposed elf-contained
appearance and odor conditions to avoid materials to avoid nazardous decomposition p III. Fire and Explosic lash point. (test method) tammable limits in air % by inusual fire and explosion h	roducts	Clear, colo Heat, spark Strong oxid Incomplete toxic vapor -11°C (Tag 1.3 Volatile and Carbon diox Water will i spread it, b containers.	rless liquid is, open flat izing agent combustion s. closed cup d flammable d flammable kide, dry ch not be effect ut a water Wear full pparatus. h age contain	with a char me, open co s and strong can gener auto ignition upper limit a emical or f ctive in ext spray can b protective leat will bu	g acids. ate carbon mo on temperature oam. inguishing a fi e used to cool clothing and s	poor ventilation. pnoxide and other 562°C 7.1 ire and may exposed elf-contained

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V. Health Hazards

<u>Occupatio</u>	nal Exposure Lir	nit	5	<u>Concentration Imm</u> to He	mediately Dangerous ealth
OSHA	8-hour PEL	-	10 ppm		
	Ceiling	-	25 ppm	OSHA/NIOSH	2,000 ppm
	Peak	-	50 ppm		
			••	Odor Thr	<u>eshold</u>
ACGIH	TLV-TWA	-	10 ppm		
	TLV-STEL	-	25 ppm	NSC	2 ppm
	(15-min)		••	NIOSH	not listed
				OHS	1.5-5 ppm
NIOSH	TLV-TWA	-	10 ppm		
	TLV-C	-	not listed		

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Carcinogenic, Mutagenic, Teratogenic Data

Human carcinogen (NTP, IARC) Suspect human carcinogen (ACGIH) Mutagenic and teratogenic data (RTEC) Animal carcinogen (IARC)

Primary Routes of Entry

Benzene may exert its effects through inhalation, skin absorption, and ingestion.

Industrial Exposure: Route of Exposure/Signs and Symptoms

- Inhalation: Exposure can cause dizziness, intoxication, excitement, headache, vomiting, delirium, drowsiness, and unconsciousness.
- Eye Contact: Liquid and high vapor concentration can cause irritation, neuritis, atrophy, visual impairment, edema, and cataracts.
- Skin Contact: Prolonged or repeated skin contact can cause irritation and dermatitis through defatting of skin.

Ingestion: Can cause gastrointestinal tract discomfort.

Effects of Overexposure

Benzene is a primary skin irritant, central nervous system depressant, bone marrow depressant, and leukemogen. Acute benzene exposure from inhalation or ingestion initially produces excitation and euphoria, followed by headache, drowsiness, dizziness, vomiting, delirium and unconsciousness. Respiratory irritation and pulmonary edema are possible. Severe exposure causes blurred vision, tremors, shallow and rapid respiration, ventricular fibrillation, paralysis, and convulsions. Liver and kidney damage may occur. Chronic exposure to benzene poses the most significant toxic effects. Symptoms are headache, anorexia, nervousness, weariness, anemia, pallor, bleeding under the skin and eyes, and reduced clotting ability. Bone marrow damage and leukemia may develop. Liver and kidney damage may occur.

Medical Condition Aggravated by Exposure

Preclude from exposure those individuals with diseases of the heart, lung, kidney, liver, nervous system, or the blood, and those susceptible to dermatitis.

Emergency First Aid

Inhalation: Immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse administer cardiopulmonary resuscitation (CPR). Contact physician immediately.

Eye Contact: Rinse with copious amounts of water for at least 15 minutes. Get emergency medical assistance.

Skin Contact: Flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothing and shoes. Wash clothing before re-use, and discard contaminated shoes. Get emergency medical assistance.

Ingestion: Call local Poison Control Center for assistance. Contact physician immediately. Aspiration Hazard - Do not induce vomiting.

VI. Safety Measures and Equipment_

Skin:

Ventilation: Adequate ventilation is required to protect personnel from exposure to chemical vapors exceeding the PEL and to minimize fire hazards. The choice of ventilation equipment, either local or general, will depend on the conditions of use, quantity of material, and other operating parameters.

Respiratory: Use approved respirator equipment. Follow NIOSH and equipment manufacturer's recommendations to determine appropriate equipment (air-purifying, air-supplied, or self-contained breathing apparatus).

Eyes: Safety glasses are considered minimum protection. Goggles or face shield may be necessary depending on quantity of material and conditions of use.

Protective gloves and clothing are recommended. The choice of material must be based on chemical resistance and other user requirements. Generally, Buna-N offers acceptable chemical resistance. Individuals who are acutely and specifically sensitive to benzene may require additional protective equipment. Storage: Benzene should be protected from temperature extremes and direct sunlight. Proper storage of benzene must be determined based on other materials stored and their hazards and potential chemical incompatibility. In general, benzene should be stored in an acceptably protected and secure flammable liquid storage room.

Other: Emergency eye wash fountains and safety showers should be available in the vicinity of any potential exposure. Ground and bond metal containers to minimize static sparks.

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VII. Spill and Disposal Data_

Spill Control:

Protect from ignition. Wear protective clothing and use approved respirator equipment. Absorb spilled material in an absorbent recommended for solvent spills and remove to a safe location for disposal by approved methods. If released to the environment, comply with all regulatory notification requirements. l

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Waste Disposal:

Dispose of benzene as an EPA hazardous waste. Hazardous waste numbers: U019(Ignitable, Toxic); D001(Ignitable).

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Revision Date: 6/85

<u>KEY</u>

са	Approximately	STEL	Short Term Exposure Level
па	Not applicable	TLV	Threshold Limit Value
С	Ceiling	TWA	Time Weighted Average
PEL	Permissable Exposure Level	BuAc	Butyl Acetate
	· · ·		

NSC National Safety Council ("Fundamentals of Industrial Hygiene", 1983) OHS Occupational Health Services ("Hazardline")

chemists helping chemists in research & industry aldrich chemical co. P.O. Box 355, Milweukee, Wisconsin 53201 USA	Telephone: (414) 273-3850 TWX: (910) 262-3052 Aldrichem MI Telex: 26 843 Aldrich MI FAX: (414) 273-4979
ATTN: SAFETY DIRECTOR IT CORPORATION TECHNOLOGY CEVELOPMENT LABORATORY 304 DIRECTORS ORIVE KNOXVILLE TN 37923 DAVID PRALT	₽₩ DATE: 04/11/ CUST#: 427195 'P']#:
MATERIAL SAFETY DATA	SHEET PAGE 1
PRODUCT #: 14714-1 NAME: NAPHTHALENE, 993 CAS #:91-20-3 MF: C10H8	- · ·
SYNCNYMS CAMPHER TAR * MIGHTY 150 * MIGHTY RD1 * MOTH BALLS * MOTH FLAKES * NAFTALEN (POLISH) * NAPHTHALENE (AC NAPHTHALIN * NAPHTHALIN (DCT) * NAPHTHALINE * NAPHT MOLTEN (DOT) * NCI-C52904 * RCRA WASTE NUMBER U165 1334 (DDT) * UN 2304 (DDT) * WHITE TAR *	GIH,OOT,CSHA) + HENE * NAPTHALENE,
RTECS NO: 0J0525000 NAPHTYALENE IRRITATION DATA	
SKN-RBT 495 MG OPEN MLD UCDS* EYE-RBT 100 MG MLD BIOFX TLICITY DATA	** 1/11/68 (* 16-4/70
UNR-HMN LDLD:29 MG/KG YKYUA UNR-MAN LDLD:74 MG/KG 85DCA URR-MAN LDLD:74 MG/KG 85DCA URL-RAT LD50:490 MG/KG 85GMA URL-MUS LD50:533 MG/KG FAATD IPR-MUS LD50:150 MG/KG NTIS* SCU-MUS LD50:1969 MG/KG TCIZA IVN-MUS LD50:100 MG/KG CSLNX URL-SPG LD50:1200 MG/KG GISAA	Q -,228,60 6 31,1499,80 1 2,73,70 1 -,89,82 0 4,406,84 4 AD691-490 G 20,772,73 4 NX#00203 A 47(11),78,82
REVIEWS, STANCARDS, AND REGULATIONS ACGIH TLV-TWA 10 PPM; STEL 15 PPM 85INA8 5,420,86 EPA FIFRA 1988 PESTICIDE SUBJECT TO REGISTRATION OR FEREAC 54,7740,89	
MSHA STANDARD-AIR:TWA 10 PPM (50 MG/M3) DTLVS# 3,17 DSHA PEL:8H TWA 10 PPM (50 MG/M3) FEREAC 54,2923,89 DSHA PEL FINAL:8H TWA 10 PPM (50 MG/M3);STEL 15 PPM 54,2923,89	
NOHS 1974: HZD 49600; NIS 71; TNF 4341; NDS 68; TNE NDES 1983: HZD 4960000; TNF 83; NIS 7209; NCS 87; 5220	TNE 112696; TFE
EPA GENETOX PROGRAM 1988, NEGATIVE: CELL TRANSFORM. EPA GENETOX PROGRAM 1988, NEGATIVE: CELL TRANSFORM. EMBRYD EPA GENETOX PROGRAM 1988, NEGATIVE: HISTIDINE REVER	-RLV F344 RAT
EPA TSCA CHEMICAL INVENTORY, JUNE 1990 EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, DECEMB	

CONTINUED ON NEXT PAGE

Telephone Belgium 114747 Holland 060224748 Telex 62302 Alchem B FAX 022428216

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Aldnch-Ch D-7924 Sti Co ¤G D-7924 Steinheim Telephone 7329870 Telex 714838 Aldri D FAX 0732987139/239



Telephone: (414) 273-3850 TWX: (910) 262-3052 Aldrichem MI Telex: 26 843 Aldrich MI FAX: (414) 273-4979

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MATERIAL SAFETY DATA SHEET PAGE

CUST#: 427195 PC#:

PRODUCT #: 14714-1 CAS #:91-20-3 MF: C1048

• • • •

NAME: NAPHTHALENE, 99%

----- TOXICITY HAZAPDS ------

NIOSH ANALYTICAL METHODS: SEE HYDROCARBONS, ARCMATIC, 1501; NIDSH ANALYTICAL METHODS: SEE POLYNUCLEAR ARCNATIC HYDROCARBONS (HPLC) 5506: (GC), 5515 NTP CARCINCGENESIS STUDIES:ON TEST (TWO YEAR STUDIES), OCTOBER 1990 OSHA ANALYTICAL METHOD #35 TARGET ORGAN DATA SENSE URGANS AND SPECIAL SENSES (PTOSIS) BEHAVIERAL (SUMNOLENCE) BEHAVIERAL (CHANGE IN MOTOR ACTIVITY) BEHAVIERAL (ATAXIA) LUNGS, THERAX ER RESPIRATION (RESPIRATORY DEPRESSION) LUNGS, THURAX UK RESPIRATION (RESPIRATORY DEPRESSION) TIONAL INFORMATION NAPHTHALENE IS RETINOTOXIC AND SYSTEMIC ABSORPTION OF ITS VAPOR MAY RESULT IN CATARACTS, OPTICAL NEURITIS, INJURIES TO THE CORNEA AND MARKED FYE IRRITATION ABOVE 15 PPM. INGESTION OF LARGE QUANTITIES HAS BEEN REPORTED TO CAUSE SEVERE HEMOLYTIC ANEMIA AND HEMOGLOBINURIA. ADDITIONAL ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES (RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR COMPLETE INFORMATION. ----- HEALTH HAZARD DATA -----ACUTE EFFECTS HARMFUL IF SWALLDWED, INHALED, OR ABSORBED THROUGH SKIN. CAUSES EYE AND SKIN IRRITATION. MATERIAL IS IRPITATING TO MUCOUS MEMBRANES AND UPPER RESPIRATORY TRACT. SYMPTOMS OF EXPOSURE MAY INCLUDE BURNING SENSATION, COUGHING, HHEEZING, LARYNGITIS, SHORTNESS OF BREATH, HEADACHE, NAUSEA AND VUMITING. ABSORPTION INTO THE BODY LEADS TO THE FORMATION OF METHEMOGLOBIN WHICH IN SUFFICIENT CONCENTRATION CAUSES CYANOSIS. ONSET MAY BE DELAYED 2 TO 4 HOURS OR LONGER. MAY CAUSE ALLERGIC SKIN REACTION. TARGET ORGAN(S): ĒŸĒŠ BLÖDD, KIDNEYS FIRST ALD IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES OR SKIN WITH COPIDUS

CONTINUED ON NEXT PAGE

Adinch Chemie Bautevard Lambermont 140 b6 B-1030 Bruzelev/Russet Feleptione Beigum 114747 Holland Go524748 Teles 6302 Accem B FAX 022426216 & \$ 5, 4

Aldinch-Chimie S.a.rl 27. Fosse des Treze F-67000 Strasbourg Telephone: 8527010 Telephone: 85227010 Telex: 890076 Aldinch FAX: 88751283 Naly Aldrich Chimics S.r. Vis Pietro Toestil, 4 20127 Millano Telephone 022813/ Telez 330882 Aldm FAX 022896301 Japan Aldrich Japan Kyodo Bidg, Shinkanda 10 Kanda-Miliuracho Chiyoda-Ku, Tokyo Telephone 032560155 Salt Angenoide 032560155 Booin Alginen Guirthica Agit de Correas 161 20100 Alcobendas (Madrid Temphone 916639977 Tetes 22169 SAG3-E FAX 91653006

United Kingdom Addreh Chamical Co., Ltd The Old Brickyard, New Road Gillingham, Dorbet SPB 4./L Telephone 074782211 Telephone 074782211 Telephone 417238 Aldreh G FAX 0747823779 West Commany Alghch Chamie GribH & Co. KG D-7924 Steinneim Telephone. 7329870 Teles. 714838 Algh D FAX. 0732887139/239



Telephone: (414) 273-3850 TWX: (910) 262-3052 Aldrichem MI Telex: 26 843 Aldrich MI FAX: (414) 273-4979

MATERIAL SAFETY DATA SHEET PAGE 3

CUST#: 427195 PO#:

PRODUCT #: 14714-1 CAS #:91-20-3 MF: C1048

NAME: NAPHTHALENE, 99%

----- HEALTH HAZARD DATA ------

ANOUNTS OF WATER FOR AT LEAST 15 MINUTES WHILE REMOVING CONTAMINATED ANDONIS OF WATER FOR AT LEAST IS MINUTES WHILE REMOVING CONTAMINATED CLOTHING AND SHOES. ASSURE ADEQUATE FLUSHING OF THE EYES BY SEPARATING THE EYELIDS WITH FINGERS. IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE DXYGEN. IF SWALLOWED, WASH OUT MOUTH WITH WATER PROVIDED PERSON IS CONSCIOUS. CALL & PHYSICIAN. DISCARD. CONTAMINATED CLOTHING AND SHOES. DISCARD CONTAMINATED CLOTHING AND SHOES.

----- PHYSICAL DATA ------

MELTING PT: 80 C TO 82 C NELTING FIL VAPUR DENSITY: 4.4 VAPUR PRESSURE: 03 MM & 25 C 1 MM & 53 C TARANCE AND ODDR WHITE CRYSTALLINE FLAKES

----- FIRE AND EXPLOSION HAZARD DATA -----

FLASHPOINT: 174 F AUTOIGNITION TEMPERATURE: 978 F LOWER EXPLOSION LEVEL: .92 UPPER EXPLOSION LEVEL: .92 EXTINGUISHING MEDIA CARBON DIDXIDE. DRY CHEMICAL POWDER. FOAM AND WATER SPRAY ARE EFFECTIVE BUT MAY CAUSE FROTHING. SPECIAL FIREFIGHTING PROCEDURES WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES. FLAMMABLE SOLID. UNUSUAL FIRE AND EXPLOSIONS HAZARDS FLASHPOINT: 174 F

UNUSUAL FIRE AND EXPLOSIONS HAZARDS THIS MATERIAL, LIKE MOST MATERIALS IN POWDER FORM, IS CAPABLE OF CREATING A DUST EXPLOSION.

INCOMPATIBILITIES OXIDIZING AGENTS

CONTINUED ON NEXT PAGE

----- REACTIVITY DATA ------

114747 224748 X 022428216

6160

FAX 0747823779

H & Co KG

	mists helping chemists in research & industry Idrich Chemical CO. Nox 355, Milwaukee, Wisconsin 53201 USA	Telephone: (414) 273-3850 TWX: (910) 262-3052 Aldrichem Mi Telex: 26 843 Aldrich Mi FAX: (414) 273-4979
	MATERIAL SAFETY DATA	A SHEET PAGE 4
		CUST#: 427195 P3#:
PRUDUCT #: CAS #:91-2 MF: C1048	14714-1 NAME: NAPHTHALENE, 995 0-3	8
-	REACTIVITY DATA	
TUXIC FUME	USTICN OR DECOMPOSITION PRODUCTS S OF: CXIDE, CARBON DIOXIDE	F
STEPS TO BE TA EVACUATE À	KEN IF MATERIAL IS RELEASED OR SPILLED	•
WEAR SELF- RUBBER GLC SWEEP UP AVUID RAIS WASTE DISPOSAL DISSOLVE C CHEMICAL I	CONTAINED BREATHING APPARATUS, RUBBER BO VES. PLACE IN A BAG AND HOLD FOR WASTE DISPON ING DUST. METHOD R MIX THE MATERIAL WITH A COMBUSTIBLE SO NCINERATOR EQUIPPED WITH AN AFTERBURNER L FEDERAL, STATE AND LOCAL ENVIRONMENTAL	SAL. DLVFNT AND BURN IN A AND SCRUBBER. L REGULATIONS.
COMPATIBLE NICSH/MSHA SAFETY SHO USE UNLY I DD NOT BRE AVOID CONT DO NOT GET AVDID PROL WASH THURD TOXIC. IRRITANT. KEEP TIGHT KEEP AWAY HYGROSCOPIC STORE IN A REGULATORY INFO	ACT WITH FUMES. IN EYES. ON SKIN, ON CLOTHING. CNGED DR REPEATED EXPOSURE. UGHLY AFTER HANDLING. LY CLOSED. FROM HEAT, SPARKS, AND OPEN FLAME. C COOL DRY PLACE. ERMATION CT IS SUBJECT TO SARA SECTION 313 REPERT	TING REQUIREMENTS.
ALL INCLUSIVE LIABLE FOR ANY ABOVE PRODUCT.	RMATION IS BELIEVED TO BE CORRECT BUT DO AND SHALL BE USED ONLY AS A GUIDE. ALDRI DAMAGE RESULTING FROM HANDLING OR FROM SEE REVERSE SIDE OF INVOICE OR PACKING ITIONS OF SALE.	ICH SHALL NOT BE HELD CONTACT WITH THE
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 Belghum / Holland
 Prance
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 Algrich Chemie
 Bullrich C

Aldrich Japan Kyodo Bidg. Shinkanda 10 Kanda-Mikuracho Chyoda-Ku, Tokyo Tolophone 032560155 FAX 032560157

Agrico Química Agrico Correge 161 28100 Alcobendas (Magnd) Telepicos 916530977 Telepi 22189 SAQIS-E FAX 916538084

United Kingdom Adheh Chemical Co., Lid The Old Brezystal, New Road Gillinghom, Dontet SPS 44, Tatephone 0747622211 Telax 417238 Aldreft G FAX. 0747823779

West Germany Alginch-Chame GmoH & Co. KG 0-7924 Steinheim Telephone 7320970 Telephone 7320970 Telex 714838 Algin D FAX. 0732967139/239

CREOSOTE, COAL TAR

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Common Byna Crocosta el	ANTER LEADE	Vellow to black Tarry odor	6. FIRE KAZARDS	IR. HAZARD ASSESSMENT CODE
Dage el	1407 0 000 or 1	ris as water.	6.1 Plash Point > 1077 G.C. 6.2 Planmatic Limits in April 101 Surfaces 6.3 Pro Estimation Agents: Dry chancel, califien danals of team 6.4 Pro Estimated in Sector Not to be	(Boo Heland Announnan Handbout) A-T-U-X-Y
Stop decisinge 4 Dossible Call the oscientment Isoste and remove decharged material Isoste and remove decharged material Nooly ocal health and poly non control agencies.		Lo Producting Angene risk to an United Water may be antifective. 4.6 Speaks Heatrice of Combuston Productor to films Meany, making black ansee is formed. 4.7 Synthes Tempertaine 67°F 4.4 Execting Heatry Rist services	II. NAZARO CLASSIFICATIONS ILI Code of Federal Republicana Computers ford ILS MAS Hazard Rethy for Bulk Water Transportation: Compary Rethy	
Fire	Continuenten. Extinguen win div chemicals, losm di Carbon dioxide Water may be ineffective on tre		Cardinal Plant Character Fait personnel Cardinal Plant Character Comparisons Cardinal Plant Comparisons Cardinal Comparisons	Fire
Exposure	IF SWALLOWED and victor or main and have victor IF SWALLOWED and victor	Ning and shoes aprily of water pen and fush with benny of water in COINSCIUS, have victor onne water induces vortering in UNCCIVESCIUS SOM MAYING COM- eacept Meet victor water	7. CHEMICAL REACTIVITY 7.1 Resolvity Wile Water: No reaction 7.2 Resolvity with Common Statements for reaction 7.3 Statement for remember Added and Constant for Added and Constant Not perman 7.6 Performance of Performance 7.8 Indef rate of Resonant to Presentify Common to remember 7.1 Heater Rate (Resonant to Presentify Common 21	Anarous Elect
Water Pollution	Effect of two concentration Fouring to shortwine. May be dangerous if it enter Notify local nearth and wide Notify local nearth or nearth in	ris weiter Fillman. In clicture		12. Physical And Chemical Properties 12.1 Physical Subs at IS'C and 1 ame Little Weight Meture 12.3 Weight Weight Meture 12.5 Define Paint at 1 ame >3557 = >10°C = >355°K 12.6 Physical Research Control (1) 12.6 Physical Research Control (1) 12.7 Physical Research Control (1) 12.8 Physical Research Control (1) 12.9 Physical Research Control (1) 13.9 Physical Research Control (1
ESPORSE TO DISCHARGE See Response Manabashi Security water construction Manabashi Should be refinered Charmoni and physical treatment		K. WATER POLLITION L1 Aqueste Testelty: Cole not evaluate L2 Waterteel Testelty: Cole not evaluate L3 Biological Caygon Comand (BCD): Date not evaluate L4 Food Chain Concentration Potential: None	12.4 Pressing Paint: Not partners 12.5 Critical Transportance: Not partners 12.7 Specific Carusty: 1.05-1.09 at 15°C Dauks 12.4 Liquid Water Interstatel Tension: (est.) 15 Ornacors = 0.015 N/m at 20°C 12.9 Liquid Water Interstatel Tension: (est.) 20 Ornacors = 0.020 N/m at 20°C 12.19 Vapor (Gas) Specific Constry: Not Constraint	
	Man: 9/1983 3	4. GBSERVABLE CHARACTERISTICS 4.1 Physical Blinto (as shippool): Louid 4.2 Cater: Yellow to brown to black 4.3 Cater: Creasure or terry; aromasc		12.11 Rote of Specific Hosts of Veper (Ges); Not partnerst 12.12 Latent Hest of Vepersations Not partnerst 12.13 Hest of Canasatters (est.) —12.500 Bts/Ib = -4.900 ca/g = —200 X 10 ⁵ J/kg 12.14 Hest of Desenteestory Not partnerst 12.15 Hest of Desenteestory Not partnerst
S. HEALTH HAZARDS Second Processes Equipment: Al-servers cannot make index; chemical eatery gogges and/or has sheet overals or a necence afran, benn drove, chemical eatery gogges and/or has sheet overals or a necence afran, benn drove, chemical eatery gogges and/or has sheet overals or a necence afran, benn drove, cause symptems Pelicenty Expensive Veors cause moderns means of nose and thost, Louid cause sovere burns of oyae and reddening and ficting of alm. Proorgad contect with san can cause sovere burns of oyae and reddening and ficting of alm. Proorgad contect with san can cause sovere burns of expensive veolations, and others, proorgad contect with san can cause sovere of Expensive Medul.ATCh: remove vectors to heat hat if he is not instanting, give articles respirate sheet.ACTCh: remove vectors to heat affinal, give oxyger; cat a physicae. EYES: fuch monagements with plantly of weath for at lease 15 ms, and cat a physicael. SKIRE upps with vegetable of or mergenne, then wath with scap and weater. INGESTICN: here victim drok, water or mit; do NOT induce vomiting. 4. Threened Liait Values 0.2 mg/m ²		 SHIPPING INFORMATION Grease of Purity: White creaters or vanue inscions, desenang on boiling pare, All have samer properse. Strange Temperature: Ambern L3 Instratamethrer: No requirement L4 Vending: Open (Rame arrease) 	12.18 Heat of Palymentarian Not person 12.28 Heat of Passon: Data not evaluate 12.28 Lämiting Value: Ose not evaluate 12.27 Reid Vaper Pressure: Low	
 Short Term Inhibition Limits: Data not internated Short Term Inhibition Limits: Data not available Tassetty by Ingestion; Grade 2: LDus = 0.5 to 5 g/bg Late Testesty: Reparated explosures may cause cancer of shin. Vaper (Bas) inhibit Characteristics: Vapor Cause moderate mitation such that personnal and find may cancertrainting unpresented. The officit a tendoraty. Lipited or Selb) inflant Characteristics: Fanty severe skin mitatis. May cause pain and second-degree barry alter a few minutes. Lipited or Travelokt. Data not evaluate 		мот	NOTES	
L11 IOLH Value: 400				

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CREOSOTE, COAL TAR

12.17 SATURATED LIQUID DENSITY		12.18 LIQUID HEAT CAPACITY		12.19 LIQUID THERMAL CONDUCTIVITY		12.20 LIQUID VISCOSITY	
(degrees F)	Pounds per cubic foot (estimate)	Temperature (degrees F)	British thermal unit per pound-F (estimate)	Temperature (degrees F)	British thermai unit-inch per hour- square toot-F	Temperature (degrees F)	Centipoise
51	67.379	51	.400		N	67.73	12.000
52	67.349	52	.400		Ö		
53	67.309	53	.400		Ť	[
54	67.280	54	.400				
55	67.240	55	.400		P		
56	67.209	56	.400		E		
57	67.169	56 57	.400		R		
58	67.139	58 '	400		T		
59	67.099	59 , h			1 1		
60	67.070	.60	.400		l n ł		
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64	66.929	64	.400	•	· · · · · · · · · · · · · · · · · · ·		
65	66.900	65	.400	•	I. I	E E	
66	66.860	66	.400			1	
67	66.830	· 67	.400	•	·	1	
68	66,790	168		• •,	ъ. ·	1	
69	66.759	69	.400			· [-
70	66.719	70	.400			1	
71	66,690	71	.400				
72	66.650	. 72,	.400				
73	66.620	73	.400		.i		
74	66.580	74	.400				
75	68.549	75	.400				
76	66.509	76	.400			l l	

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12.24 BAS HEAT CAPACITY	12.23 12 SATURATED VAPOR DENSITY IDEAL GAS HEA		SATURATED V	2.22 POR PRESSURE	SATURATED VA	12.21 SOLUBILITY IN WATER	
ure British thermal unit F) per pound-F	Temperature (degrees F)	Pounds per cubic foot	Temperature (degrees F)	Pounds per square inch	Temperature (degrees F)	Pounds per 100 pounds of water	Temperature (degrees F)
N O T		N O T		N O T	.35.	I N S	
P E R		P E A	•	P E R		O L U B	
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Occupational Health Guideline for Coal Tar Pitch Volatiles

INTRODUCTION

This guideline is intended as a source of information for employees, employers, physicians, industrial hygienists, and other occupational health professionals who may have a need for such information. It does not attempt to present all data; rather, it presents pertinent information and data in summary form.

SUBSTANCE IDENTIFICATION

Anthracene

- Formula: C1.H10
- Synonyms: None
- Appearance and odor: Pale green solid with a faint aromatic odor.

Phenanthrene

- Formula: C14H10
- Synonyms: None
- Appearance and odor: Colorless solid with a faint aromatic odor.

Pyrene

- Formula: C10H10
- Synonyms: None
- Appearance: Bright yellow solid

Carbazole

- Formula: C12H,N
- Synonyms: None
- Appearance and odor: Colorless solid with a faint aromatic odor.

Benzo(a)pyrene

- Formula: C20H12
- Synonyms: BaP, 3,4-benzopyrene

• Appearance and odor: Colorless solid with a faint aromatic odor.

PERMISSIBLE EXPOSURE LIMIT (PEL)

The current OSHA standard for coal tar pitch volatiles is 0.2 milligram of coal tar pitch volatiles per cubic meter of air (mg/m³) averaged over an eight-hour work shift. NIOSH has recommended that the permissible exposure limit for coal tar products be reduced to 0.1 mg/m³ (cyclohexane-extractable fraction) averaged over a work shift of up to 10 hours per day, 40 hours per week, and that coal tar products be regulated as occupational carcinogens. The NIOSH Criteria Document for Coal Tar Products and NIOSH Criteria Document for Coke Oven Emissions should be consulted for more detailed information.

HEALTH HAZARD INFORMATION

• Routes of exposure

Coal tar pitch volatiles can affect the body if they are inhaled or if they come in contact with the eyes or skin.

• Effects of overexposure

Repeated exposure to coal tar pitch volatiles has been associated with an increased risk of developing bronchitis and cancer of the lungs, skin, bladder, and kidneys. Pregnant women may be especially susceptible to exposure effects associated with coal tar pitch volatiles. Repeated exposure to these materials may also cause sunlight to have a more severe effect on a person's skin. In addition, this type of exposure may cause an allergic skin rash.

• Reporting signs and symptoms

A physician should be contacted if anyone develops any signs or symptoms and suspects that they are caused by exposure to coal tar pitch volatiles.

• Recommended medical surveillance

The following medical procedures should be made available to each employee who is exposed to coal tar pitch volatiles at potentially hazardous levels:

These recommendations reflect good industrial hygiene and medical surveillance practices and their implementation will . assist in achieving an effective occupational health program. However, they may not be sufficient to achieve compliance with all requirements of OSHA regulations.

U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES

Public Health Service Centers for Disease Control National Institute for Occupational Safety and Health

U.S. DEPARTMENT OF LABOR

Occupational Safety and Health Administration

1. Initial Medical Examination:

-A complete history and physical examination: The purpose is to detect pre-existing conditions that might place the exposed employee at increased risk, and to establish a baseline for future health monitoring. Examination of the oral cavity, respiratory tract, bladder, and kidneys should be stressed. The skin should be examined for evidence of chronic disorders, for premalignant and malignant lesions, and evidence of hyperpigmentation or photosensitivity.

---Urinalysis: Coal tar pitch volatiles are associated with an excess of kidney and bladder cancer. A urinalysis should be obtained to include at a minimum specific gravity, albumin, glucose, and a microscopic on centrifuged sediment, as well as a test for red blood cells.

-Urinary cytology: Coal tar pitch volatiles are associated with an excess of kidney and bladder cancer. Employees having 5 or more years of exposure or who are 45 years of age or older should have a urinary cytology examination.

-Sputum cytology: Coal tar pitch volatiles are associated with an excess of lung cancer. Employees having 10 or more years of exposure or who are 45 years of age or older should have a sputum cytology examination.

-14" x 17" chest roentgenogram: Coal tar pitch volatiles are associated with an excess of lung cancer. Surveillance of the lungs is indicated.

-FVC and FEV (1 sec): Coal tar pitch volatiles are reported to cause an excess of bronchitis. Periodic surveillance is indicated.

—A complete blood count: Due to the possibility of benzene exposure associated with coal tar pitch volatiles, a complete blood count is considered necessary to search for leukemia and aplastic anemia.

-Skin disease: Coal tar pitch volatiles are defatting agents and can cause dermatitis on prolonged exposure. Persons with pre-existing skin disorders may be more susceptible to the effects of these agents.

2. Periodic Medical Examination: The aforementioned medical examinations should be repeated on an annual basis, and semi-annually for employees 45 years of age or older or with 10 or more years' exposure to coal tar pitch volatiles.

Summary of toxicology

Coal tar pitch volatiles (CTPV) are products of the destructive distillation of bituminous coal and contain polynuclear aromatic hydrocarbons (PNA's). These hydrocarbons sublime readily, thereby increasing the amounts of carcinogenic compounds in working areas. Epidemiologic evidence suggests that workers intimately exposed to the products of combustion or distillation of bituminous coal are at increased risk of cancer at many sites. These include cancer of the respiratory tract, kidney, bladder, and skin. In a study of coke oven workers, the level of exposure to CTPV and the length of time exposed were related to the development of cancer. Coke oven workers with the highest risk of cancer were those employed exclusively at topside jobs for 5 or more years, for whom the increased risk of

dying from lung cancer was 10-fold; all coke oven workers had a 7-1/2-fold increase in risk of dying from kidney cancer. Although the causative agent or agents of the cancer in coke oven workers is unidentified, it is suspected that several PNA's in the CTPV generated during the coking process are involved. Certain industrial populations exposed to coal tar products have a demonstrated risk of skin cancer. Substances containing PNA's which may produce skin cancer also produce contact dermatitis; examples are coal tar, pitch, and cutting oils. Although allergic dermatitis is readily induced by PNA's in guinea pigs, it is only rarely reported in humans from occupational contact with PNA's; these have resulted largely from the therapeutic use of coal tar preparations. Components of pitch and coal tar produce cutaneous photosensitization; skin eruptions are usually limited to areas exposed to the sun or ultraviolet light. Most of the phototoxic agents will induce hypermelanosis of the skin; if chronic photodermatitis is severe and prolonged, leukoderma may occur. Some oils containing PNA's have been associated with changes of follicular and sebaceous glands which commonly take the form of acne. There is evidence that exposures to emissions at coke ovens and gas retorts may be associated with an increased occurrence of chronic bronchitis. Coal tar pitch volatiles may be associated with benzene, an agent suspected of causing leukemia and known to cause aplastic anemia.

CHEMICAL AND PHYSICAL PROPERTIES

• Physical data—Anthracene

- 1. Molecular weight: 178.2
- 2. Boiling point (760 mm Hg): 340 C (644 F)
- 3. Specific gravity (water = 1): 1.24

4. Vapor density (air = 1 at boiling point of anthracene): 6.15

- 5. Melting point: 217 C (423 F)
- 6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg

7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble

8. Evaporation rate (butyl acetate = 1): Not applicable

• Physical data—Phenanthrene

1. Molecular weight: 178.2

2. Boiling point (760 mm Hg): 340 C (644 F)

3. Specific gravity (water = 1): 1.18

4. Vapor density (air = 1 at boiling point of phenanthrene): 6.15

5. Melting point: 100.5 C (213 F)

6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg

7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble

8. Evaporation rate (butyl acetate = 1): Not applicable

• Physical data—Pyrene

1. Molecular weight: 202.3

2. Boiling point (760 mm Hg): Greater than 360 C (greater than 680 F)

1

3. Specific gravity (water = 1): 1.28

4. Vapor density (air = 1 at boiling point of pyrene):
6.9

5. Melting point: 150.4 C (303 F)

6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg

7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble

8. Evaporation rate (butyl acetate = 1): Not applicable

• Physical data—Carbazole

1. Molecular weight: 167.2

2. Boiling point (760 mm Hg): 355 C (671 F)

3. Specific gravity (water = 1): Greater than 1 4. Vapor density (air = 1 at boiling point of carbazole): 5.8

5. Melting point: 246 C (475 F)

6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg

7. Solubility in water, g/100 g water at 20 C (68 F):

Insoluble 8. Evaporation rate (butyl acetate = 1): Not applica-

ble

Physical data—Benzo(a)pyrene

1. Molecular weight: 252.3

2. Boiling point (760 mm Hg): Greater than 360 C (greater than 680 F)

3. Specific gravity (water = 1): Greater than 1 4. Vapor density (air = 1 at boiling point of benzo(a)pyrene): 8.7

5. Melting point: 179 C (354 F)

6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg

7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble

8. Evaporation rate (butyl acetate = 1): Not applicable

• Reactivity

1. Conditions contributing to instability: None hazardous

2. Incompatibilities: Contact with strong oxidizers may cause fires and explosions.

3. Hazardous decomposition products: None

4. Special precautions: None

• Flammability

1. Flash point: Anthracene: 121 C (250 F) (closed cup); Others: Data not available

2. Autoignition temperature: Anthracene: 540 C (1004 F); Others: Data not available

3. Flammable limits in air, % by volume: Anthracene: Lower: 0.6; Others: Data not available

4. Extinguishant: Foam, dry chemical, and carbon dioxide

• Warning properties

Grant states that "coal tar and its various crude fractions appear principally to cause reddening and squamous eczema of the lid margins, with only small erosions of the corneal epithelium and superficial changes in the stroma, which disappear in a month following exposure. Chronic exposure of workmen to tar fumes and dust has been reported to cause conjunctivitis and discoloration of the cornea in the palpebral fissure, either near the limbus or, in extreme cases, across the whole cornea. Occasionally, epithelioma of the lid margin has been attributed to contact with coal tar."

MONITORING AND MEASUREMENT PROCEDURES

General

Measurements to determine employee exposure are best taken so that the average eight-hour exposure is based on a single eight-hour sample or on two four-hour samples. Several short-time interval samples (up to 30 minutes) may also be used to determine the average exposure level. Air samples should be taken in the employee's breathing zone (air that would most nearly represent that inhaled by the employee).

Method

Coal tar products may be sampled by collection on a glass fiber filter with subsequent ultrasonic extraction and weighing. An analytical method for coal tar pitch volatiles is in the *NIOSH Manual of Analytical Methods*, 2nd Ed., Vol. 1, 1977, available from the Government Printing Office, Washington, D.C. 20402 (GPO No. 017-033-00267-3).

RESPIRATORS

 Good industrial hygiene practices recommend that engineering controls be used to reduce environmental concentrations to the permissible exposure level. However, there are some exceptions where respirators may be used to control exposure. Respirators may be used when engineering and work practice controls are not technically feasible, when such controls are in the process of being installed, or when they fail and need to be supplemented. Respirators may also be used for operations which require entry into tanks or closed vessels, and in emergency situations. If the use of respirators is necessary, the only respirators permitted are those that have been approved by the Mine Safety and Health Administration (formerly Mining Enforcement and Safety Administration) or by the National Institute for Occupational Safety and Health.

• In addition to respirator selection, a complete respiratory protection program should be instituted which includes regular training, maintenance, inspection, cleaning, and evaluation.

PERSONAL PROTECTIVE EQUIPMENT

Employees should be provided with and required to use impervious clothing, gloves, face shields (eight-inch minimum), and other appropriate protective clothing necessary to prevent skin contact with condensed coal tar pitch volatiles, where skin contact may occur.
If employees' clothing may have become contaminated with coal tar pitch volatiles, employees should change into uncontaminated clothing before leaving the work premises.

Clothing contaminated with coal tar pitch volatiles

should be placed in closed containers for storage until it can be discarded or until provision is made for the removal of coal tar pitch volatiles from the clothing. If the clothing is to be laundered or otherwise cleaned to remove the coal tar pitch volatiles, the person performing the operation should be informed of coal tar pitch volatiles's hazardous properties.

• Employees should be provided with and required to use splash-proof safety goggles where condensed coal tar pitch volatiles may contact the eyes.

SANITATION

Workers subject to skin contact with coal tar pitch volatiles should wash with soap or mild detergent and water any areas of the body which may have contacted coal tar pitch volatiles at the end of each work day.
Employees who handle coal tar pitch volatiles should

wash their hands thoroughly with soap or mild detergent and water before eating, smoking, or using toilet facilities.

• Areas in which exposure to coal tar pitch volatiles may occur should be identified by signs or other appropriate means, and access to these areas should be limited to authorized persons.

COMMON OPERATIONS AND CONTROLS

The following list includes some common operations in which exposure to coal tar pitch volatiles may occur and control methods which may be effective in each case:

Operation

Controls

Liberation from extraction and packaging from coal tar fraction of coking

Use as a binding agent in manufacture of coal briquettes used for fuel; use as a dielectric in the manufacture of battery electrodes, electric-arc furnace electrodes, and electrodes for alumina reduction

Use in manufacture of roofing felts and papers and roofing

ventilation; general dilution ventilation; personal protective equipment Process enclosure; local exhaust ventilation; general

Process enclosure:

local exhaust

dilution ventilation; personal protective equipment

Process enclosure; local exhaust ventilation; general dilution ventilation; personal protective equipment

Operation

Use for protective coatings for pipes for underground conduits and drainage; use as a coating on concrete as waterproofing and corrosion-resistant material; use in road paving and sealing

Use in manufacture and repair of refractory brick; use in production of foundry cores; use in manufacture of carbon ceramic items Controis Process enclosure; local exhaust

Process enclosure; local exhaust ventilation; general dilution ventilation; personal protective equipment

Process enclosure; local exhaust ventilation; general dilution ventilation; personal protective equipment

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EMERGENCY FIRST AID PROCEDURES

In the event of an emergency, institute first aid procedures and send for first aid or medical assistance.

Eye Exposure

If condensed coal tar pitch volatiles get into the eyes, wash eyes immediately with large amounts of water, lifting the lower and upper lids occasionally. If irritation is present after washing, get medical attention. Contact lenses should not be worn when working with these chemicals.

Skin Exposure

If condensed coal tar pitch volatiles get on the skin, wash the contaminated skin using soap or mild detergent and water. Be sure to wash the hands before eating or smoking and to wash thoroughly at the close of work.

Breathing

If a person breathes in large amounts of coal tar pitch volatiles, move the exposed person to fresh air at once. If breathing has stopped, perform artificial respiration. Keep the affected person warm and at rest. Get medical attention as soon as possible.

• Rescue

Move the affected person from the hazardous exposure. If the exposed person has been overcome, notify someone else and put into effect the established emergency rescue procedures. Do not become a casualty. Understand the facility's emergency rescue procedures and know the locations of rescue equipment before the need arises.

SPILL AND DISPOSAL PROCEDURES

• Persons not wearing protective equipment and clothing should be restricted from areas of releases until cleanup has been completed.

• If coal tar pitch volatiles are released in hazardous concentrations, the following steps should be taken: 1. Ventilate area of spill.

RESPIRATORY PROTECTION FOR COAL TAR PITCH VOLATILES

Condition	Minimum Respiratory Protection* Required Above 0.2 mg/m ³		
Particulate and Vapor Concentration			
2 mg/m³ or less	A chemical cartridge respirator with an organic vapor cartridge(s) and with a fume or high-efficiency filter.		
	Any supplied-air respirator.		
	Any self-contained breathing apparatus.		
10 mg/m³ or less	A chemical cartridge respirator with a full facepiece and an organic vapor cartridge(s) and with a fume or high-efficiency filter.		
	A gas mask with a chin-style or a front- or back-mounted organic vapor canister and with a full facepiece and a fume or high-efficiency filter.		
	Any supplied-air respirator with a full facepiece, helmet, or hood.		
	Any self-contained breathing apparatus with a full facepiece.		
200 mg/m² or less	A Type C supplied-air respirator operated in pressure-demand or other positive pressure or continuous-flow mode.		
	A powered air-purifying respirator with an organic vapor cartridge and a high- efficiency particulate filter.		
400 mg/m³ or less	A Type C supplied-air respirator with a full facepiece operated in pressure- demand or other positive pressure mode or with a full facepiece, helmet, or hood operated in continuous-flow mode.		
Greater than 400 mg/m³ or entry and escape from unknown concentrations	Self-contained breathing apparatus with a full facepiece operated in pressure- demand or other positive pressure mode.		
	A combination respirator which includes a Type C supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure or continu- ous-flow mode and an auxiliary solf-contained breathing apparatus operated in pressure-demand or other positive pressure mode.		
Fire Fighting	Self-contained breathing apparatus with a full facepiece operated in pressure- demand or other positive pressure mode.		
Еѕсаре	Any gas mask providing protection against organic vapors and particulates, including pesticide respirators which meet the requirements of this class.		
	Any escape self-contained breathing apparatus.		

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*Only NIOSH-approved or MSHA-approved equipment should be used.

2. Collect released material in the most convenient and safe manner for reclamation or for disposal in sealed containers in a secured sanitary landfill.

• Waste disposal method:

Coal tar pitch volatiles may be disposed of in sealed containers in a secured sanitary landfill.

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Appendix E

Protocol for Collection and Characterization of Treatability Study Test Matrix

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PROTOCOL FOR COLLECTION AND CHARACTERIZATION OF TREATABILITY STUDY TEST MATRIX

Soil samples for the Phase I laboratory-scale treatability study will be collected from the Moss-American site. One sampling event will be conducted. Sufficient quantities of soils for all planned Phase I bioslurry and soil washing treatability tests will be obtained during this sampling event and placed into drums for transport, and/or intermediate storage at the site awaiting transport to the testing laboratories.

SAMPLING OBJECTIVE

The objective of this sampling event is to collect sufficient quantities of CPAHcontaminated soils from the site to conduct the planned Phase I treatability studies on the bioslurry and soil washing technologies. These soil portions will be characterized prior to treatability testing, for parameters which are important in the treatability study program. Analytical data from this characterization will be used to support analyses and interpretation of treatability study results.

SOIL SAMPLE (TEST MATRIX) REQUIREMENTS

Two soil composites will be collected from the site. One composite is intended to provide soils exhibiting "average" CPAH concentrations in the range of 300-600 mg/kg. The second composite is intended to provide "high" CPAH concentrations in the range of 1,000-1,500 mg/kg. The selection of soil sampling locations to meet these criteria will be based upon existing RI/FS site characterization data and other predesign activities as these data may become available. The areas from which these samples will be taken include the former processing area and the former treated storage areas of the Moss-American site.

Due to possible RCRA restrictions on storage of soil quantities at the treatability test facility, soil quantities in excess of the permitted amount will be stored in tarp-covered

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drums and staged on the Moss-American site pending transport to the designated testing facilities.

SOIL SAMPLING PROCEDURE

The area selected for site sampling will be marked with pin flags by the field sampling team. Within this area, the required volume of soil will be excavated using hand tools. The excavated soils will be placed temporarily on plastic sheeting located adjacent to the excavated area. Large debris, rocks, and turf will be manually separated from the soils. The excavated soils will be manually mixed using hand tools to provide a relatively homogeneous mixture. Following mixing, the soils will be placed into drums and sample containers as appropriate, sealed, labeled, and moved to the temporary staging area while awaiting shipment. Large debris, rocks, and turf will be returned to the excavation. Additional borrow soil will be used as necessary to fill the excavated area. The "average" concentration soil composite will be collected first and the "high" concentration composite collected second in a similar manner.

Equipment and personnel decontamination procedures presented in the Interim Health and Safety Plan and the Predesign Phase Quality Assurance Project Plan will be followed.

SOIL SAMPLE (TEST MATRIX) CHARACTERIZATION

Soil composites collected from the site will be characterized in order to evaluate properties or conditions that may affect or determine the results of the treatability test. Properties or conditions that will be considered include the following:

- CPAH concentration, which could affect treatability performance and the statistical interpretation of treatability test results.
- Physical/chemical properties, such as particle size distribution, organic carbon content and the presence of other contaminants, that may interfere with the treatment processes.

• Variables that may affect biological activity, such as macro- and micro-nutrient levels and pH.

Indigenous microbial activity levels in the soil samples/composites will be characterized to determine the potential need for microbial acclimation or stimulation. This effort will include an estimation of microbial population/viability and determination of PAH degradation capabilities, which will be accomplished by using aerobic plate counts or most probable number (MPN) methods.

At the time of the site sampling event, one portion (approximately 5 kg) of each composite will be shipped to WESTON's Environmental Technology Laboratory (ETL) in West Chester, PA for initial physical/chemical characterization. An additional portion will be aseptically transferred to sterile containers and transmitted to ETL for microbial enumeration. The initial characterization program is summarized in Table 2, while analytical methods and holding times are summarized in Table 3.

Soil composites and analytical samples will be shipped to the ETL and treatability testing laboratories by certified commercial carrier.

HEALTH AND SAFETY

The soil collection and compositing sampling event will be conducted in accordance with the Interim Predesign Health and Safety Plan, as amended by HASP Amendment No. 1.¹

¹Roy F. Weston, Inc., Draft Predesign Work Plan, Moss-American Site, Milwaukee, Wisconsin, 28 April 1992.

Table 1

Soil Composite Quantities

	Bioslurry Treatability Test (lb.)	Soil Washing Test (lb.)	Total
Average Soil Composite	100	110	210
High Soil Composite	100	110	210

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Table 2

Initial Characterization Test Matrix

Parameter	Laboratory ¹	Average Soils	"High" Soils	Total
Microbial Enumeration	FE	1	1	2
Particle Size Distribution	ETL	1	1	2
Porosity (Bulk Density/Specific Gravity)	ETL	1	2	2
Moisture Content	ETL	1	1	2
Liquid/Plastic Limits	ETL	1	1	2
Percent Solids	ETL	1	1	2
рН	WA	1	1	2
Total Organic Carbon (TOC)	WA	1	1	2
СРАН	WA	1	1	2
BTX	WA	1	1	2

 ¹ FE - WESTON Fate and Effects Laboratory ETL - WESTON Environmental Technology Laboratory WA - WESTON Analytics (Lionville) Laboratory

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Table 3

Analytical Methods

Parameter	Method	Sample Requirements	Preservation
Microbial Enumeration	Plate Count	100 g./ Sterile glass	Cool, 4°C
Particle Size Distribution	ASTM D422	1 l.	
Porosity (Bulk Density/ Specific Gravity)		1 l.	None
Moisture Content	ASTM D2216	1 l .	None
Atterberg Limits	ASTM D423/D424	1 1.	None
Percent Solids	CLP SOW	250 ML/amber glass	Cool, 4°C
рН	9040	250 ML/amber glass	Cool, 4°C
Total Organic Carbon (TOC)	Method 415.1	250 ML/amber glass	Cool, 4°C
СРАН	EPA Method 8310	250 ML/amber glass	Cool, 4°C
BTX	EPA Method 8020	2-125 ML/amber glass	Cool, 4°C