DOE/MC/29467 -- 5670

Decontamination Systems Information and Research Program

Quarterly Report

July 1 - August 31, 1996

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For

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Office of Environmental Management
Office of Technology Development
1000 Independence Avenue
Washington, DC 20585

U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

By

West Virginia University Research Corp.
West Virginia University
P. O. Box 6103
Morgantown, West Virginia 26506-6103

Disclaimer

ABSTRACT

This project concerns investigation of the feasibility of establishing voluntary remediation programs in the Kanawha Valley, WV.

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EXECUTIVE SUMMARY

The United States contains numerous hazardous waste sites. Many sites are on private land near operating units of various companies. An effort is being made to determine the conditions under which such sites can be remediated voluntarily. Such voluntary remediation would result in an accelerated clean-up of U.S. sites and a significant cost savings. Meetings are being held with U.S. companies to evaluate the potential for actually initiating voluntary clean-ups.

1.0 INTRODUCTION

The objective of the project will be to first assess the interest and willingness of industry in the Kanawha River Valley to participate in discussions that would lead toward voluntary cleanup activities. The second will be to implement the activities agreed upon by the interested parties. The project will first involve individual discussions with the industrial, government, and other organized groups in the area. These discussions will help determine the feasibility of organizing voluntary efforts. If the discussions indicate that conditions may be favorable for developing individual or group voluntary cleanup projects, a working group will be convened to establish the environmental goals of the project as well as the technical approach for achieving those goals.

2.0 WORK PERFORMED THIS PERIOD

This period involved only planning work, since many contacts were on vacation and the Brownfields legislation is being developed under the leadership of one of the principals of this project.

3.0 WORK PLANNED FOR NEXT PERIOD

Begin MOU development and discuss implementation with the companies. Link companies to DOE.

4.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES - None.

EXECUTIVE SUMMARY

The projects for the 1996 WVU Cooperative Agreement are categorized into three task focus areas: Task 1.0 Contaminant Plume Containment & Remediation, Task 2.0 Cross Cutting Innovative Technologies, and Task 3.0 Small Business Support Program. Summaries of the accomplishments for the subtasks reporting under these categories during the third quarter, 1 July 96 through 30 September 96, are presented below.

TASK 1.0 CONTAMINANT PLUME CONTAINMENT & REMEDIATION

Subtask 1.1 Analysis of the Vortec Cyclone Melting System (VMS) for Remediation of PCB Contaminated Soils Using CFD: Work continued this period on the analysis of the recuperator base (residence chamber) and the recuperator itself. Analysis of the temperature distribution and species concentrations inside the residence chamber have been calculated. An approximate kinetic model has been constructed to calculate the concentration of specified hydrocarbon or PCBs as a function of time. The calculations performed using the FLUENT computer code produced PCB destruction efficiencies of 99.9999% in the flue gas. Current work incorporates radiation and species transport modeling of the recuperator, and work will begin on the analysis of the separator/reservoir.

Subtask 1.2 Drain Enhanced Soil Flushing (DESF) Using Prefabricated Vertical Drains: Work continued this period on the field pilot scale test. The testing was performed at a closed Ashland Petroleum gas station site having soil and groundwater contaminated with BTEX. The pilot scale test used five prefabricated vertical drains (PVDs) spaced three feet apart. The PVDs were tied into a pipe header system that permitted application of either a positive or negative head pressure. The extraction testing was performed over two days and showed reduced levels of BTEX concentrations in groundwater grab samples. Injection tests were also proven to be successful. This project continues to mark progress and planning has begun to expand installation of the PVDs for a scaled-up test phase. Work for the next period will include installation of the PVDs at the Weston, WV site by the cost-sharing partner the Nilex Corp.

Subtask 1.3 *Performance and Characteristics Evaluation of Acrylates as Grout Barriers*: During this period research was directed into four areas; 1) Viscosity tests on vinyl monomer mixtures, 2) Injection of urethane grout into the 12 inch diameter columns, 3) Flexible-wall permeability tests on urethane grout samples, and 4) Compaction tests on various ratios of sand / kaolinite soil mixtures. Permeability test results on the urethane grout specimens yielded hydraulic conductivities on the order of magnitude of 10^{-9} cm/s for water as the permeant; hydraulic conductivity results using TCLP solutions were reported on the order of magnitude of 10^{-5} cm/s. For the next quarter, research will continue with long-term permeability testing, refining the grout injection process, and testing the vinyl monomer grout in the large diameter columns.

Subtask 1.4 *Development of Standard Test Protocols Barrier Design Models for Desiccation Barriers*: Accomplishments over the past quarter include: METC SARS committee approval of the proposed operating permit; Continued review of existing publications concerning air-water mass transfer in porous media; Relocation of the experimental apparatus to METC Building B-17 and equipment setup; Experiments on sand pack effective porosity and drying characteristics; and the design and experimental plans for the scaled-up Circulating Air Test Cell were initiated. Planned activities for the fourth quarter include preparation of the construction and operating permits for the large scale testing proposed for the B-17 facility, continue testing of the porosity and permeability on sand pack specimens, and continue drying testing.

Subtask 1.5 *Development of Standard Test Protocols & Barrier Design Models for In Situ Formed Barriers - Technical Support*: Accomplishments reported this quarter include: Obtaining the METC Operating and Construction permits for the Circulating Air Barrier and Chemical Grout projects; Development and submittal of the QA/QC guidelines for the Circulating Air Barrier tests and obtaining the construction permit for the scale-up Circulating Air Test Cell apparatus; and completing the paper work for permitting and preparation of QA/QC guidelines for the Chemical

Grout project. Planned work for the next period will include preparation of a report on planning for testing Freeze Barrier and Dual Barrier systems.

Subtask 1.6 *In Situ Bioremediation of Chlorinated Solvents*: During this quarter the following accomplishments were reported: partial site data from the DOE Portsmouth Gaseous Diffusion Plant was obtained from INTERA and reviewed; experimentation on existing columns continued to operate in the sequential and simultaneous modes; and microcosm studies were begun. The second simultaneous column was constructed followed with initiation of tracer and integrity testing. Microcosm work has proven the ability of methogens to completely degrade TCE and a previously unknown acclimation phase has been discovered. This discovery has led to the creation of a new maintenance reactor for the express purpose of acclimating methogens to TCE. Other microcosms have been created to study effects of a sodium surfactant on biodegradation.

Subtask 1.7A *Development and Implementation of a Decision Support System for Management of the EM50 Technology Development Program:* During this reporting period work concentrated on: Collecting and entering data sets into the database; Refining database development; and Developing Decision Support System tools. Project personnel have met with representatives from DOE's METC and Savannah River facilities, and with personnel from the United States Air Force at Brooks AFB. Interim prototype demonstrations of the software showed that the Decision Support System provides functionality and flexibility to the user. The project is expected to be completed during the next quarter.

Subtask 1.7B *Development of a Prototype Database and Decision Support System for Management of the EM50 Technology Development Program:* During this period, progress was made with development of two on-line help systems. These systems will be used by the database design team supported under Subtask 1.7A. Work for the next period will consist of designing an on-line hep system to provide context-sensitive help for users of the EM-50 database.

Task 2.0 CROSS CUTTING INNOVATIVE TECHNOLOGIES

Subtask 2.1 *A GIS-Based Infrastructure for Site Characterization and Remediation*: Progress has been reported on the three major project tasks. Work has continued on development of the prototype interactive tutorial system and with expanded GIS application to the US Forest Service. Difficulties were reported with obtaining information from the US Corps of Engineers at the Huntington district for mapping and modeling the WV Ordinance site. This was due to delays with implementing the site aquifer pump-and-treat remediation cleanup operation. This task will be extended through to the end of December 1996.

Subtask 2.2 *Treatment of Mixed Wastes via Fluidized Bed Steam Reforming*: During this period the experimental program continued investigating the destruction efficiency (conversion) of organics in a steam reforming atmosphere. Experimental results using toluene and dichlorobenzene show that destruction efficiencies of 99% and greater are possible in a steam reforming atmosphere. The results of this work suggest that thermal destruction will be sufficient to obtain destruction efficiencies of four nines or better. The research continues into the next quarter and focuses on further destruction testing of the two compounds.

Subtask 2.3 *Use of centrifugal Membrane Technology with Novel Membranes to Treat Hazardous/Radioactive Waste*: In the third quarter the effect of membrane type (polymeric versus ceramic) on the performance of the SpinTek technology was investigated. Testing on an industrial coolant waste that had an oil content of approximately 5% was performed. Based on data obtained during this portion of the research, it appears that the SpinTek centrifugal system equipped with a ceramic membrane can treat a concentrated oil/grease waste stream satisfactorily. The ceramic membrane was found superior to the polymeric membrane tested for the following reasons: 1) the ceramic membrane had a much higher flux, 2) the ceramic membrane had a higher operating temperature, thus producing a larger flux, and 3) the polymeric membrane continued to foul compared with no fouling occurring for the ceramic membrane. For the final quarter of this year, work will begin on acquisition of oil/grease samples from DOE's RMI facility in Ashtabula, Ohio.

Subtask 2.4 Environmental Pollution Control Devices Based on Novel Forms of Carbon: Accomplishments reported this quarter include completion of an extensive literature review concerning heavy metal removal, waste water treatment, and water remediation using carbonaceous material to find applications of this technology to radionuclides. Continued testing of carbonaceous material for removal of cadmium and lead from aqueous samples was performed, removal of nickel from rinse solutions used in the plating industry was demonstrated, and removal of strontium and uranium from liquid waste streams was examined. Evaluation of the experimental data obtained from scaled-up equipment testing has identified areas requiring continued design iteration. Work will continue during the next quarter to improve design of the scaled-up remediation cell, removal of uranium will be studied, and removal of nickel from industrial samples is planned.

Subtask 2.5 Development of Instrumental Methods for Characterization and Analysis of Nuclear Wastes and Environmental Contaminants: Work in the third quarter focused on development of a viable ion exchange capillary electrochromatography (IE-CEC) system, to be refined and improved on in the fourth quarter. A packed capillary format was used to provide the maximum sample loading capacity and thereby yield optimal limits of detection. Relative to capillary zone electrophoresis (CZE), IE-CEC could yield both higher efficiency and higher sample capacity in the separation of ionic compounds. The fluorescence detector constructed earlier in the project year will be used in the fourth quarter in an effort to push limits of detection even lower. The project is on schedule and results continue to be encouraging.

Subtask 2.6A Production and Evaluation of Biosorbents and Cleaning Solutions for Use in Decontamination and Decommissioning: The objective of work this quarter dealt with identification of commercial surfactant formulations that will replace the ITG formulation IGT-FF-52 surfactant. The IGT surfactant functions as a cleaning solution to solubilize organic contaminants. Several candidate commercial surfactants were identified from stability tests performed in the presence of 25% ethanol. A sample of one to the commercial surfactants that performed best, Witcamide 85, was sent to West Virginia University for filtration tests. Additionally, metal binding tests were performed with several biosorbent preparations. The results will be reported next quarter.

<u>Subtask 2.6B</u> *Use of SpinTek Centrifugal Membrane Technology and Sorbents/Cleaning Solutions for D&D Work*: During this period four waste streams were treated using the SpinTek centrifugal ultrafiltration system: 1) IGT's biosorbent material, 2) thickened waste activated sludge, 3) kaolinite, and 4) IGT's amide-based surfactant. The SpinTek unit was operated under the following experimental parameters: flux, temperature, and turbidity. Results of this experiment show that the unit successfully separated a biosorbent for the permeate stream, and that turbidity was determined to be an appropriate indicator of micelle concentration in the IGT amide solution. The project continues to be successful with applying the SpinTek unit to waste streams having characteristics similar to waste streams produced during D&D activities. During the next quarter several surfactants will be separated from the aqueous phase.

Task 3.0 <u>SMALL BUSINESS SUPPORT PROGRAM</u>

<u>Subtask 3.1</u> *WVHTC - West Virginia High Tech Consortium Foundation - Environmental Technology Support Program*: Work the third quarter targeted three areas: 1) Environmental Technology Transfer from WVU via commercialization, 2) Establishing a track record as a viable commercialization service, and 3) Developing an Environmental Remediation Test bed site. Work during the next and final quarter will be directed at implementing key initiatives to support the commercialization focus.

<u>Subtask 3.2</u> *Small Business Interaction Opportunities*: Work performed this period included four new efforts: 1) development of a proposal for determining the effect of lithium hydroxide on glass quality in the Vortec vitrification process, 2) work with the Zenon Corp for providing novel membranes for testing on the SpinTek centrifugal membrane system, 3) Initiation of work on a new Subtask (Evaluation of a Supercritical Gravity Pressure Vessel), and 4) A site visit and proposal was developed to capitalize on the decontamination and decommissioning opportunities available at the RMI Extrusion plant at Ashtabula, Ohio.

Subtask 3.3 *Evaluation of a Supercritical Gravity Pressure Vessel (SGPV) for Supercritical Water Oxidation (SCWO)*: This project works with GeneSyst International on a technology evaluation to determine the applicability of the Supercritical Gravity Pressure Vessel (SGPV) to its applicability for treating low level mixed wastes within DOE. The project began at the end of the third quarter and therefore no quarterly report was submitted. The project will be reported on at the end of the next quarter.

Subtask 3.4 *Approach for Assessing Potential Voluntary Environmental Protection - Kanawha River Area*: During this period planning work was initiated and development of the Brownsfield legislation was developed under the leadership of one principal of the project.

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2.0 CROSS CUTTING INNOVATIVE TECHNOLOGIES

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3.0 SMALL BUSINESS SUPPORT PROGRAM

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1.1 ANALYSIS OF THE VORTEC CYCLONE MELTING SYSTEM (VMS) FOR REMEDIATION OF PCB CONTAMINATED SOILS USING CFD

I. Celik, Dept. of Mechanical & Aerospace Eng.

1.2 DRAINED ENHANCED SOIL FLUSHING (DESF) USING PREFABRICATED VERTICAL DRAINS

M. Gabr, Dept. of Civil & Environmental Eng.

1.3 PERFORMANCE AND CHARACTERISTICS EVALUATION OF ACRYLATES AS GROUT BARRIERS

M. Gabr, Dept. of Civil & Environmental Eng.

1.4 DEVELOPMENT OF STANDARD TEST PROTOCOLS & BARRIER DESIGN MODELS FOR DESICCATION BARRIERS

S. Ameri and K. Aminian, Dept. of Petroleum Eng.

1.5 DEVELOPMENT OF STANDARD TEST PROTOCOLS & BARRIER DESIGN MODELS FOR IN-SITU FORMED BARRIERS - TECHNICAL SUPPORT

B. Overby and C. Locke, BDM

1.6 IN SITU BIOREMEDIATION OF CHLORINATED SOLVENTS AT PORTS

P. Carriers, W. Sack, W. Lin, Dept. of Civil & Environmental Eng.

PROJECT TITLES WITH PRINCIPAL INVESTIGATORS (Cont.)

1.7A DEVELOPMENT AND IMPLEMENTATION OF A DECISION SUPPORT SYSTEM FOR MANAGEMENT OF THE EM-50 TECHNOLOGY DEVELOPMENT PROGRAM

R. Malinovsky, WPI

1.7B DEVELOPMENT OF A PROTOTYPE DATABASE & DECISION SUPPORT SYSTEM FOR MANAGEMENT OF THE EM-50 TECHNOLOGY DEVELOPMENT PROGRAM

J. Denton, College of Business and Economics

2.0 CROSS CUTTING INNOVATIVE TECHNOLOGIES

2.1 A GIS BASED INFRASTRUCTURE FOR SITE CHARACTERIZATION AND REMEDIATION

J. Hooper, Marshall University

2.2 TREATMENT OF MIXED WASTES VIA FLUIDIZED BED STEAM REFORMING - CAPTURE OF HAZARDOUS CHEMICAL WASTE UTILIZING FLUIDIZATION COATING TECHNOLOGY

R. Turton, Dept. of Chemical Eng.

2.3 USE OF CENTRIFUGAL MEMBRANE TECHNOLOGY WITH NOVEL MEMBRANES TO TREAT HAZARDOUS/RADIOACTIVE WASTE

B. Reed, Dept. of Civil & Environmental Eng.
W. Green, SpinTek

PROJECT TITLES WITH PRINCIPAL INVESTIGATORS (Cont.)

2.4 ENVIRONMENTAL POLLUTION CONTROL DEVICES BASED ON NOVEL FORMS OF CARBON

J. Zondlo, A. Stiller, A. Brennsteiner,
Dept. of Chemical Eng.

2.5 DEVELOPMENT OF INSTRUMENTAL METHODS FOR CHARACTERIZATION AND ANALYSIS OF NUCLEAR WASTES AND ENVIRONMENTAL CONTAMINANTS

V. Remcho, Dept. of Chemistry
N. Ballou, Battelle-Pacific Northwest National Laboratories

2.6A PRODUCTION AND EVALUATION OF BIOSORBENTS AND CLEANING SOLUTIONS FOR USE IN DECONTAMINATION AND DECOMMISSIONING

J. Kilbane and V. Srivastava, IGT

2.6B USE OF SPINTEK CENTRIFUGAL MEMBRANE TECHNOLOGY AND SORBENTS/CLEANING SOLUTIONS FOR DECONTAMINATION AND DECOMMISSIONING WORK

B. Reed, Dept. of Civil & Environmental Eng.
W. Green and R. Hays, SpinTek

3.0 <u>SMALL BUSINESS SUPPORT PROGRAM</u>

3.1 WVHTC - WEST VIRGINIA HIGH TECH CONSORTIUM FOUNDATION ENVIRONMENTAL TECHNOLOGY SUPPORT PROGRAM

J. Berkow, WVHTC

PROJECT TITLES WITH PRINCIPAL INVESTIGATORS (Cont.)

3.2 SMALL BUSINESS INTERACTION OPPORTUNITIES

E. Cook, Dept. of Civil and Environmental Engineering

3.3 TECHNOLOGY EVALUATION FOR SUPERCRITICAL WATER OXIDATION (SCWO) USING A SUPERCRITICAL GRAVITY PRESSURE VESSEL (SGPV)

Gerald C. Rappe, Genesyst International Inc.

CONTINUATION FROM 1995

Old **3.4** APPROACH FOR ASSESSING POTENTIAL VOLUNTARY ENVIRONMENTAL PROTECTION - KANAWHA RIVER AREA

R. Lovett, NRCCE

Administrative Summary

Background

West Virginia University (WVU) and the US Department of Energy, Morgantown Energy Technology Center (DOE/METC) entered a Cooperative Agreement in August 1992 titled "Decontamination Systems Information and Research Programs" (DOE Instrument No.: DE-FC21-92MC29467). Requirements stipulated by the Agreement require WVU to submit quarterly Technical Progress reports. This report contains the efforts of the research projects comprising the Agreement for the third (3rd) calender quarter of 1996.

Administrative Issues

Task 3.3, *"Evaluation of a Supercritical Gravity Pressure Vessel (SGPV) for Supercritical Water Oxidation (SCWO)"* was initiated this quarter with GeneSyst International Inc. This work focuses on a technology evaluation to determine the applicability of the Supercritical Gravity Pressure Vessel (SGPV) for treating low level mixed wastes within DOE. The project began at the end of the third quarter and so no quarterly report deliverables are included in this submission. The only deliverable for this project will be the final report that is due at the end of the fourth quarter.

Accounting Issues

A semi-accrued accounting system was adopted by the Department of Civil and Environmental Engineering (CEE) for 1996 budget reports. Each projects report contains a column for accrued expenses and obligated amounts that have not been disbursed by the WVU controller's office. All adjustments, except subcontractor expenses, are made in this column. Subcontractor expenditures are shown in the month incurred. For the 3rd calendar quarter of 1996 the following accrued adjustments were made to the controller's report:

Outlays:	$375,146
Obligated Encumbrances:	(103,392)
TOTAL ADJUSTMENT:	**$271,754**

ACCRUED ACCOUNTING OF AWARDED FUNDS

PERIOD COVERED 6/30/96 - 9/30/96

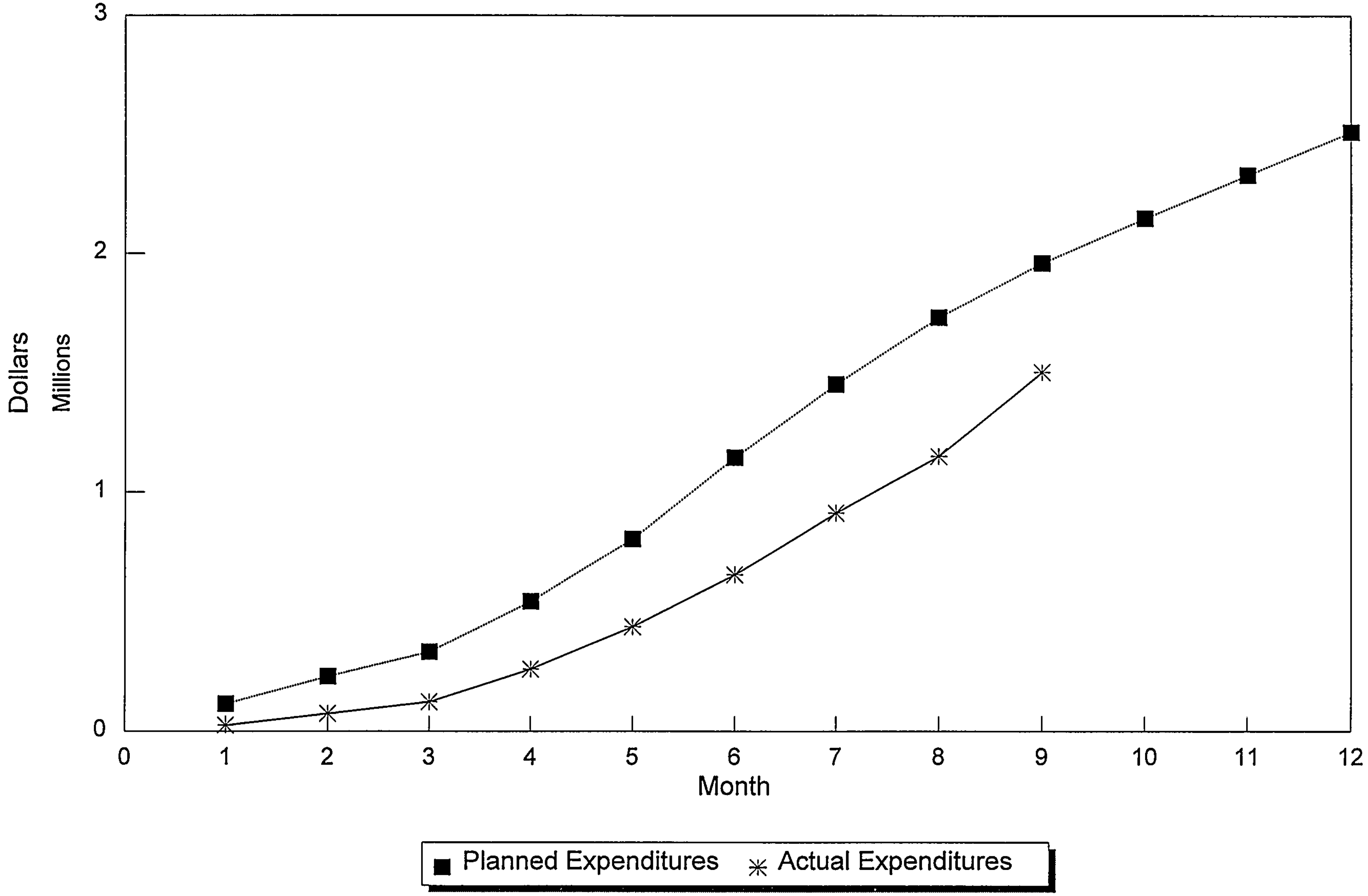
OUTLAYS

PREVIOUS OUTLAYS & ADJUSTMENTS (Grant Start - 3/31/96)

FUND 7631 - NRCCE				
Previous Outlays	3,525,462			
Current Adjustments				
FUND 8527 - WVU				
1995 Outlays	2,593,151			
Current Adjustments	1,034			
1996 Outlays (1/1 - 6/30/96)	654,582			
TOTAL PREVIOUS OUTLAYS		6,774,229		
CURRENT OUTLAYS (7/1/96-9/30/96)		848,861		
TOTAL PROJECTED OUTLAYS TO DATE 9/30/96			**7,623,090**	
UNLIQUIDATED OBLIGATIONS			759,376	
TOTAL SHARE OF OUTLAYS & UNLIQUIDATED OBLIGATIONS				**8,382,466**
FUNDS AUTHORIZED				
FUND 7631 (Prior to 1995)	4,749,048	4,749,048		
FUND 8527				
1995 Award	1,547,000			
Amendment No. A013	75,950			
1996 Award	1,900,000			
Amendment No. A015	361,605	3,884,555		
TOTAL FUNDS AUTHORIZED TO DATE				**8,633,603**
AMOUNT REMAINING				**$251,137**

Summary of Planned Versus Actual Expenses

1996 WVU Cooperative Agreement



METC TASK #	PLANNED ACTUAL	1 JAN	2 FEB	3 MAR	4 APR	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	TOTAL PLANNED	TOTAL ACTUAL
1.0 CONTAMINANT PLUME CONTAINMENT & REMEDIATION															
1.1	PLANNED	119	119	405	763	9,433	12,384	13,636	9,255	7,857	7,857	7,857	7,857	77,542	
	ACTUAL	0	0	286	914	7,016	15,853	7,873	2,074	3,003					37,019
1.2	PLANNED	13,170	13,400	13,400	14,830	13,400	21,914	20,484	20,484	13,400	13,400	13,400	13,400	184,682	
	ACTUAL	719	1,173	1,266	18,980	15,592	23,896	15,670	15,934	12,583					105,813
1.3	PLANNED	9,686	13,951	10,391	9,786	8,456	10,399	13,603	13,603	8,457	8,457	8,457	8,457	123,703	
	ACTUAL	0	3,167	3,207	3,311	2,567	5,967	18,988	14,400	16,466					68,073
1.4	PLANNED	9,787	9,787	10,502	10,502	10,502	27,508	27,508	27,508	9,887	9,887	9,887	9,887	173,152	
	ACTUAL	5,223	7,863	6,268	7,224	10,864	7,568	18,152	35,390	5,367					103,919
1.5	PLANNED	6,791	6,791	6,791	54,479	54,479	54,479	54,479	54,479	54,479	54,479	54,479	54,712	510,917	
	ACTUAL	4,793	4,793	4,793	44,223	61,445	52,488	47,118	59,522	6,458					285,633
1.6	PLANNED				0	5,532	18,508	14,007	8,821	6,851	5,421	4,706	4,706	68,552	
	ACTUAL				0	0	6,777	20,180	8,952	4,996					40,905
1.7A	PLANNED				0	65,976	57,221	57,822	57,221	57,221	9,533	10,134	9,533	324,661	
	ACTUAL				0	0	5,770	13,963	13,636	180,018					213,387
1.7B	PLANNED				0	5,856	6,607	5,856	6,070	5,713	2,566	2,137	2,137	36,942	
	ACTUAL				0	0	0	16,762	5,534	5,804					28,100
TOT 1.0	**PLANNED**	**39,553**	**44,048**	**41,489**	**90,360**	**173,634**	**209,020**	**207,395**	**197,441**	**163,865**	**111,600**	**111,057**	**110,689**	**1,500,151**	
	ACTUAL	**10,735**	**16,996**	**15,820**	**74,652**	**97,484**	**118,319**	**158,706**	**155,442**	**234,695**	**0**	**0**	**0**		**882,849**
2.0 CROSS CUTTING INNOVATIVE TECHNOLOGIES															
2.1	PLANNED	14,610	12,410	11,858	13,868	13,868	12,488	13,958	13,958	12,568	12,568	12,568	12,568	157,290	
	ACTUAL	8,979	8,340	11,111	9,282	12,844	12,409	16,409	17,163	15,920					112,457
2.2	PLANNED	4,469	3,056	2,527	1,669	7,972	15,134	1,669	1,669	1,812	1,669	1,669	1,812	45,127	
	ACTUAL	0	3,290	2,009	4,576	9,752	16,131	1,155	1,245	1,349					39,507
2.3	PLANNED	6,568	10,138	5,066	54,709	4,709	5,066	13,458	15,961	5,066	4,709	4,280	4,065	133,795	
	ACTUAL	0	1,126	1,233	3,264	5,035	8,808	19,558	16,722	57,353					113,099
2.4	PLANNED	11,343	10,343	7,274	6,773	6,773	11,312	10,955	10,955	7,131	5,987	5,343	5,701	99,890	
	ACTUAL	0	9,395	8,804	12,936	9,725	15,335	7,106	8,880	6,485					78,666
2.5	PLANNED	12,174	9,174	2,352	7,337	10,435	11,153	16,679	9,306	5,672	5,493	5,493	5,672	100,940	
	ACTUAL	0	4,019	2,609	10,996	11,678	14,323	12,850	8,910	6,219					71,604
2.6A	PLANNED				4,100	4,100	4,100	4,100	4,100	4,100	4,100	3,717	3,333	35,750	
	ACTUAL				0	2,776	736	2,990	6,018	3,043					15,563
2.6B	PLANNED				0	5,502	8,536	7,821	6,660	4,784	5,499	4,784	4,784	48,370	
	ACTUAL				0	0	48	0	794	7,334					8,176
TOT 2.0	**PLANNED**	**49,164**	**45,121**	**29,077**	**88,456**	**53,359**	**67,789**	**68,640**	**62,609**	**41,133**	**40,025**	**37,854**	**37,935**	**621,162**	
	ACTUAL	**8,979**	**26,170**	**25,766**	**41,054**	**51,810**	**67,790**	**60,068**	**59,732**	**97,703**	**0**	**0**	**0**		**439,072**
3.0 SMALL BUSINESS SUPPORT PROGRAM															
3.1	PLANNED	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	142,998	
	ACTUAL	4,145	4,145	4,145	11,407	11,407	17,406	14,038	14,574	15,109					96,376
**3.2	PLANNED	8,624	8,624	13,891	7,909	7,909	38,926	19,044	7,909	10,676	9,203	9,203	9,961	151,879	
	ACTUAL	950	1,033	1,816	11,039	13,100	13,385	10,965	7,778	4,867					64,933
*3.3	PLANNED								0	0	14,268	12,854	10,529	37,651	
	ACTUAL								0	0					0
TOT 3.0	**PLANNED**	**20,541**	**20,541**	**25,808**	**19,826**	**19,826**	**50,843**	**30,961**	**19,826**	**22,593**	**35,388**	**33,974**	**32,407**	**332,528**	
	ACTUAL	**5,095**	**5,178**	**5,961**	**22,446**	**24,507**	**30,791**	**25,003**	**22,352**	**19,976**	**0**	**0**	**0**		**161,309**
CONTINUATION FROM LAST YEAR															
old 3.4	PLANNED	5,307	5,307	5,307	13,675	13,675	13,675	0	0	0	0	0	0	56,946	
	ACTUAL	1,007	84	0	0	3,868	0	14,918	336	0					20,213
Unsettled Accounts															
GRAND TOTALS															
	PLANNED	**114,665**	**115,017**	**101,681**	**212,317**	**260,494**	**341,327**	**306,996**	**279,876**	**227,591**	**187,013**	**182,885**	**181,031**	**2,510,787**	
	ACTUAL	**25,816**	**48,428**	**47,547**	**138,152**	**177,669**	**216,900**	**258,695**	**237,862**	**352,374**	**0**	**0**	**0**		**1,503,443**

***Note: Project initiated this quarter**
****Note: Project adjusted this quarter**

General Note: The above monthly numbers are rounded to the nearest dollar.
Detailed information per project is included on the following pages.

METC TASK #	PROJECT TITLE	PLANNED ACTUAL	TOTAL PLANNED	TOTAL ACTUAL	OBLIGATED Encumbrances	AMOUNT REMAINING
1.0 CONTAMINANT PLUME CONTAINMENT & REMEDIATION						
1.1	Analysis of the Vortec Cyclone Melting System (VMS) for Remediation of contaminated Soils using CFD	PLANNED	77,542			
		ACTUAL		37,019	9,009	31,514
1.2	Drained Enhanced Soil Flusing (DESF) using Prefabricated Vertical Drains	PLANNED	184,682			
		ACTUAL		105,813	59,755	19,114
1.3	Performance and Characteristics Evaluation of Acrylates as Grout Barriers	PLANNED	123,703			
		ACTUAL		68,073	32,034	23,596
1.4	Development of Standard Test Protocols & Barrier Design Models for Dessication Barriers	PLANNED	173,152			
		ACTUAL		103,919	31,731	37,502
1.5	Development of Standard Test Protocols & Barrier Design Models for In-Situ Formed Barriers - Technical Support	PLANNED	510,917			
		ACTUAL		285,633	225,283	1
1.6	In Situ Bioremediation of Chlorinated Solvents at PORTS	PLANNED	68,552			
		ACTUAL		40,905	9,607	18,040
1.7A	Development and Implementation of a Decision Support System (DDS) for Management of the EM-50 Technology Development Program	PLANNED	324,661			
		ACTUAL		213,387	101,275	9,999
1.7B	Development of a Prototype Database and Decision Support System (DDS) for Management of the EM-50 Technology Development Program	PLANNED	36,942			
		ACTUAL		28,100	6,414	2,428
TOT 1.0	**TOTAL TASK 1.0**	PLANNED	1,500,151			
		ACTUAL		882,849	475,108	142,194
2.0 CROSS CUTTING INNOVATIVE TECHNOLOGIES						
2.1	A GIS Based Infrastructure for Site Characterization and Remediation	PLANNED	157,290			
		ACTUAL		112,457	43,001	1,832
2.2	Treatment of Mixed Wastes via Fluidized Bed Steam Reforming - Capture of Hazardous Chemical Waste Utilizing Fluidization Coating Technology	PLANNED	45,127			
		ACTUAL		39,507	3,404	2,216
2.3	Use of Centrifugal Membrane Technology with Novel Membranes to Treat Hazardous/Radioactive Waste	PLANNED	133,795			
		ACTUAL		113,099	21,965	(1,269)
2.4	Environmental Pollution Control Devices Based on Novel Forms of Carbon	PLANNED	99,890			
		ACTUAL		78,666	18,631	2,593
2.5	Development of Instrumental Methods for Characterization & Analysis of Nuclear Wastes & Environmental Contaminants	PLANNED	100,940			
		ACTUAL		71,604	31,560	(2,224)
2.6A	Production and Evaluation of Biosorbents and Cleaning Solutions for Use in Decontamination & Decommissioning	PLANNED	35,750			
		ACTUAL		15,563	20,187	0
2.6B	Use of SpinTek Centrifugal Membrane Technology and Sorbents/Cleaning Solutions for D & D Work	PLANNED	48,370			
		ACTUAL		8,176	18,121	22,073
TOT 2.0	**TOTAL TASK 2.0**	PLANNED	621,162			
		ACTUAL		439,072	156,869	25,221
3.0 SMALL BUSINESS SUPPORT PROGRAM						
3.1	WVHTC - West Virginia High Tech Consortium Foundation Environmental Technology Support Program	PLANNED	142,998			
		ACTUAL		96,376	43,783	2,839
**3.2	Small Business Interaction Opportunities	PLANNED	151,879			
		ACTUAL		64,933	18,741	68,205
*3.3	Technology Evaluation for Supercritical Water Oxidation Using a Supercritical Gravity Pressure Vessel	PLANNED	37,651			
		ACTUAL		0	37,650	1
TOT 3.0	**TOTAL TASK 3.0**	PLANNED	332,528			
		ACTUAL		161,309	100,174	71,045
CONTINUATION FROM LAST YEAR						
old 3.4	Approach for Assessing Potential Voluntary Environmental Protection - Kanawha River Area	PLANNED	56,946			
		ACTUAL		20,213	27,225	9,508
Unsettled Accounts						3,169
GRAND TOTALS		PLANNED	2,510,787			
		ACTUAL		1,503,443	759,376	251,137

*Note: Project initiated this quarter
**Note: Project adjusted this quarter

TASK 1.0

Contaminant Plume Containment & Remediation

PROJECT: Analysis of the Vortec Cyclone Melting System (CMS) for Remediation of PCB Contaminated Soils Using CFD

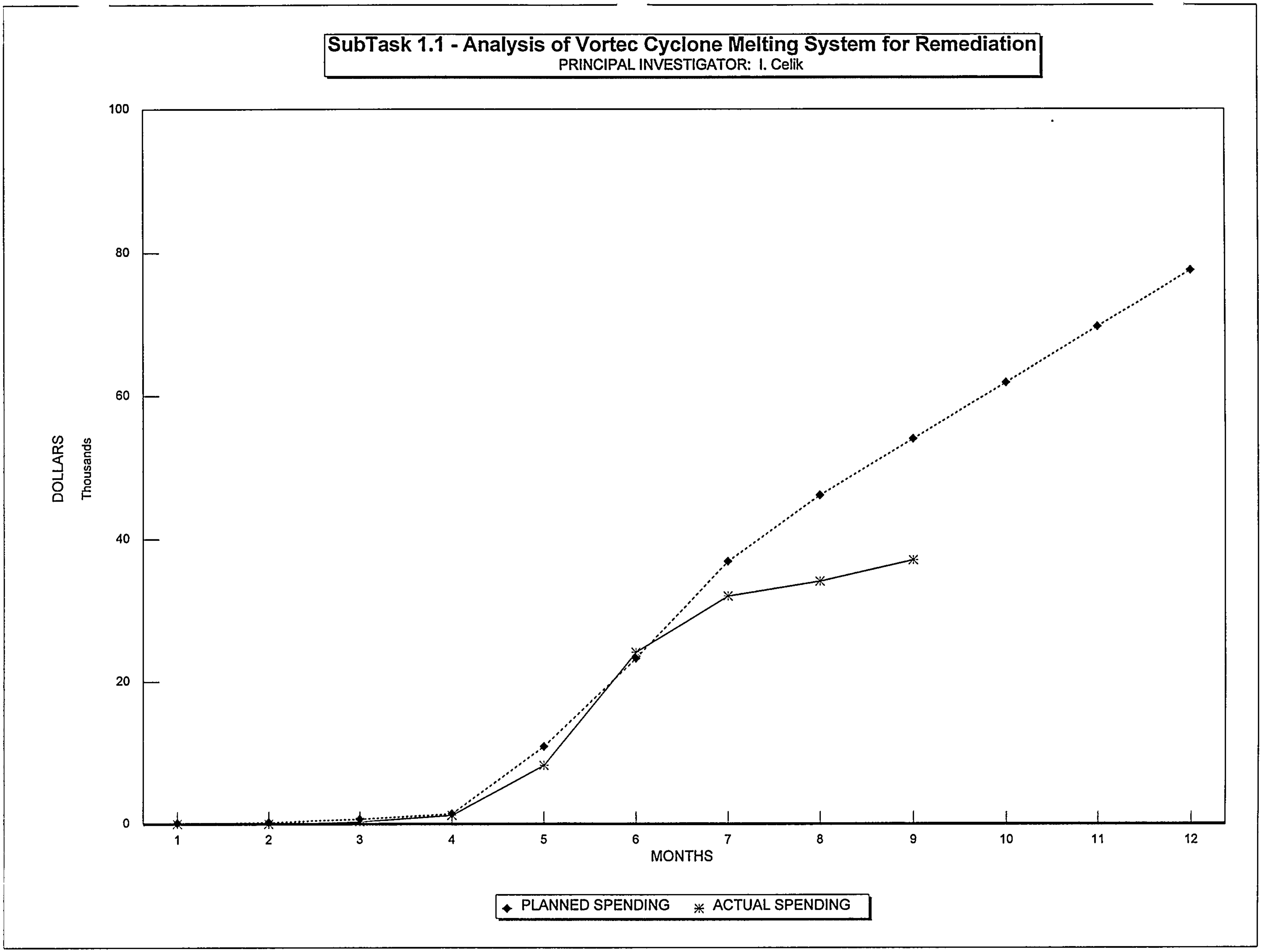
METC TASK NO.: 1.1

WVU ACCOUNT #: 8527-093-3536

PRINCIPAL INVESTIGATOR: I. Celik

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Full-time I. Celik	planned	0	0	0	0	3,150	6,300	6,300	3,150	2,393	2,393	2,393	2,393	$28,470			
	actual	0	0	0	0	3,145	6,289	3,144	0	0					$12,578	0	15,893
BENEFITS 29% of Full Time Salaries	planned	0	0	0	0	914	1,827	1,827	914	694	694	694	694	$8,256			
	actual	0	0	0	0	912	1,824	912	0	0	0	0	0		$3,647	0	4,609
STUDENTS Graduate	planned	0	0	200	200	200	200	1,200	2,200	2,200	2,200	2,200	2,200	$13,000			
	actual	0	0	200	100	850	1,600	1,450	1,450	2,100	0				$7,750	6,300	(1,050)
SUPPLIES	planned	0	0	0	250	250	250	125	125	125	125	125	125	$1,500			
	actual	0	0	0	0	0	0	0	0	0					$0	0	1,500
TRAVEL	planned	0	0	0	0	2,000	0	0	0	0	0	0	0	$2,000			
	actual	0	0	0	0	0	1,373	0	0	0					$1,373	0	627
OTHER	planned	83	83	83	83	83	83	83	83	83	83	83	83	$1,000			
	actual	0	0	0	539	0	0	0	0	0					$539	0	461
INDIRECT COSTS 43% of Direct Costs except *	planned	36	36	122	229	2,837	3,724	4,100	2,783	2,363	2,363	2,363	2,363	$23,317			
	actual	0	0	86	275	2,110	4,767	2,367	624	903	0	0	0		$11,131	2,709	9,477
PLANNED TOTALS: PER MONTH		119	119	405	763	9,433	12,384	13,636	9,255	7,857	7,857	7,857	7,857	$77,544			
ACTUAL TOTALS: PER MONTH		0	0	286	914	7,016	15,853	7,873	2,074	3,003	0	0	0		$37,018	9,009	31,516
CUMULATIVE																	
PLANNED TOTALS		119	238	644	1,406	10,840	23,224	36,859	46,114	53,972	61,829	69,686	77,544	$77,544			
ACTUAL TOTALS		0	0	286	1,200	8,216	24,069	31,942	34,015	37,018	37,018	37,018	37,018		$37,018		31,516
Budget Variance		$119	$238	$358	$206	$2,624	($845)	$4,917	$12,099	$16,953	$24,810	$32,668	$40,525				



SubTask 1.1 - Analysis of Vortec Cyclone Melting System for Remediation

PRINCIPAL INVESTIGATOR: I. Celik

PROJECT: Drained Enhanced Soil Flushing (DESF) Using Prefabricated Vertical Drains

METC TASK NO.: 1.2

WVU ACCOUNT NO.: 8527-090-3239

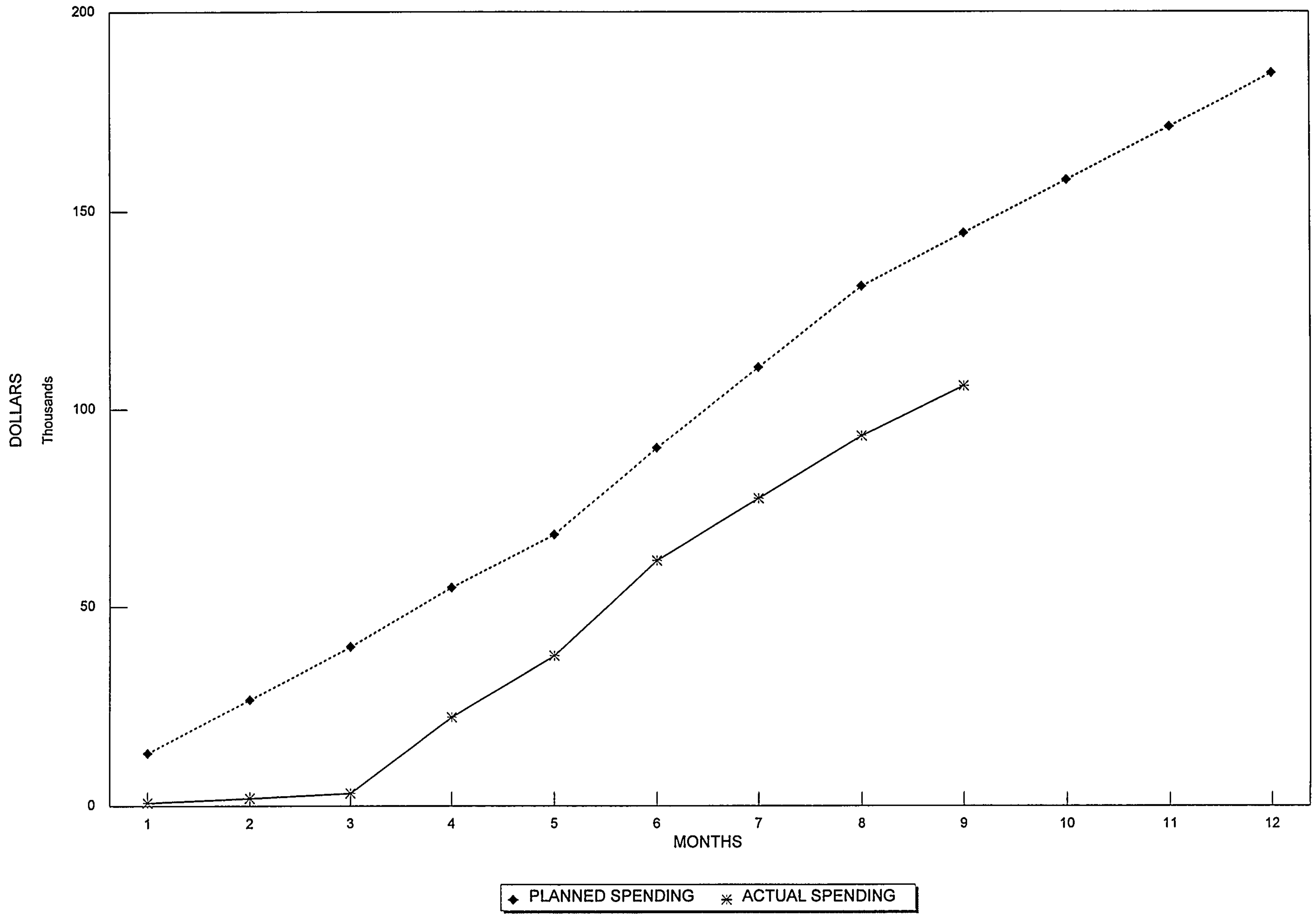
PRINCIPAL INVESTIGATOR: M.A. Gabr

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
M.A. Gabr	planned	0	0	0	0	0	3,840	3,840	3,840	0	0	0	0	$11,520			
S. Kiger	actual	0	0	0	2,495	4,551	6,607	0	4,162	2,081					$19,896	0	(8,376)
Research	planned	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	$12,870			
Associate	actual	0	0	0	5504.5	0	0	0	0	0					$5,505	0	7,366
Management	planned	609	609	609	609	609	609	609	609	609	609	609	609	$7,309			
	actual	0	0	0	1,217	2,129	2,129	0	0	0					$5,475	1,866	(32)
BENEFITS																	
29% of Full	planned	488	488	488	488	488	1,601	1,601	1,601	488	488	488	488	$9,193			
Time Salaries	actual	0	0	0	2,673	1,937	2,533	0	1,207	603	0	0	0		$8,954	541	(302)
STUDENTS																	
Graduate	planned	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	$23,920			
	actual	0	0	0	0	0	2,535	7,845	4,975	4,250					$19,605	12,750	(8,435)
Undergraduate	planned	750	750	750	750	750	750	750	750	750	750	750	750	$9,000			
	actual	0	0	285	369	828	600	837	639	564					$4,122	0	4,878
SUPPLIES	planned	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	$18,000			
	actual	319	705	439	1,014	971	1,393	272	43	359					$5,514	1,629	10,857
TRAVEL	planned	0	1,000	0	1,000	0	1,000	0	0	0	0	0	0	$3,000			
	actual	0	0	0	0	487	91	1,311	117	942					$2,948	0	52
OTHER	planned	458	458	458	458	458	458	458	458	458	458	458	458	$5,500			
	actual	184	0	162	0	0	0	0	0	0					$346	0	5,154
EQUIPMENT	planned	1,500	1,500	0	0	0	0	0	0	0	0	0	0	$3,000			
< $1,000	actual	0	116	0	0	0	305	92	0	0					$513		2,487
* > $1,000	planned	1,200	0	0	0	0	0	0	0	0	0	0	0	$1,200			
	actual	0	0	0	0	0	740	859	0	0					$1,599		(399)
SUB-CONTR.	planned	0	0	2,500	2,500	2,500	2,500	2,500	2,500	2,500	2,500	2,500	2,500	$25,000			
< $25,000	actual	0	0	0	0	0	0	0	0	0					$0	25,000	0
INDIRECT COSTS																	
43% of Direct	planned	3,599	4,029	4,029	4,459	4,029	6,590	6,160	6,160	4,029	4,029	4,029	4,029	$55,174			
Costs except *	actual	216	353	381	5,707	4,688	6,963	4,454	4,791	3,784	0	0	0		$31,337	17,968	5,869
PLANNED TOTALS: PER MONTH		13,170	13,400	13,400	14,830	13,400	21,914	20,484	20,484	13,400	13,400	13,400	13,400	$184,686			
ACTUAL TOTALS: PER MONTH		719	1,173	1,266	18,980	15,592	23,896	15,670	15,934	12,583	0	0	0		$105,812	59,755	19,119
CUMULATIVE																	
PLANNED TOTALS		13,170	26,571	39,971	54,802	68,202	90,116	110,600	131,084	144,485	157,885	171,286	184,686	$184,686			
ACTUAL TOTALS		719	1,892	3,158	22,138	37,730	61,626	77,296	93,230	105,812	105,812	105,812	105,812		$105,812		19,119
Budget Variance		$12,451	$24,679	$36,813	$32,664	$30,472	$28,490	$33,304	$37,855	$38,672	$52,073	$65,473	$78,873				

Sub-Task 1.2 - Drained Enhanced Soil Flushing Using Prefabricated Vertical Drains

PRINCIPAL INVESTIGATOR: M.A. Gabr



PROJECT: **Performance & Characteristics Evaluation of acrylates as Grout Barriers for In Situ Containment**

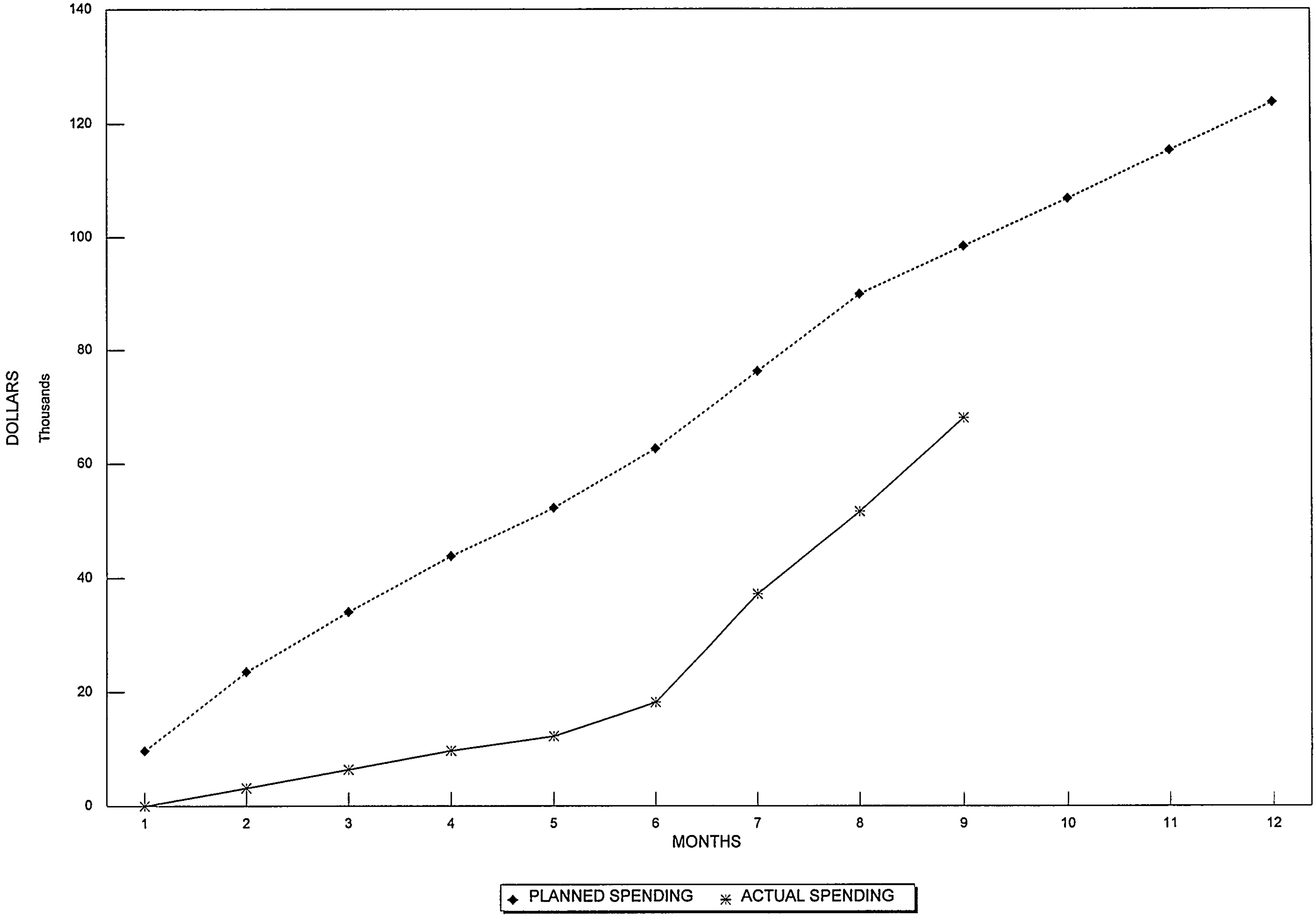
SUMMARY on/off Campus

METC TASK NO.: 1.3

PRINCIPAL INVESTIGATOR: M.A. Gabr

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Full-time	planned	0	1,500	1,500	0	0	0	3,000	3,000	0	0	0	0	$9,000			
M.A. Gabr	actual	0	0	0	0	0	0	5,874	2,937	3,690					$12,501	3,690	(7,191)
Research	planned	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	1,073	$12,870			
Associate	actual	0	0	0	0	0	0	0	0	0						0	12,870
Management	planned	609	609	609	609	609	609	609	609	609	609	609	609	$7,309			
	actual	0	0	0	0	915	915	686	686	686					$3,888	2,058	1,363
BENEFITS																	
29% of Full	planned	488	923	923	488	488	488	1,358	1,358	488	488	488	488	$8,462			
Time Salaries	actual	0	0	0	0	265	265	1,902	1,051	1,269	0	0	0		$4,753	1,667	2,042
STUDENTS																	
Graduate	planned	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	1,993	$23,920			
	actual	0	2,300	1,450	1,450	271	1,275	1,700	2,125	3,825					$14,396	11,475	(1,951)
Undergraduate	planned	1,040	1,040	1,040	1,040	1,040	1,040	1,040	1,040	1,040	1,040	1,040	1,040	$12,480			
	actual	0	168	120	120	0	1,227	3,957	2,064	1,479					$9,135	2,764	581
SUPPLIES	planned	2,500	2,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	1,500	$20,000			
	actual	0	107	798	1,122	186	150	461	1,241	1,083					$5,148	2,701	12,151
TRAVEL	planned	0	1,000	0	1,000	0	500	0	0	0	0	0	0	$2,500			
	actual	0	0	0	0	0	245	0	161	369					$775	200	1,525
OTHER	planned	42	42	42	42	42	42	42	42	42	42	42	42	$500			
	actual	0	0	239	0	258	582	111	0	842					$2,032	0	(1,532)
EQUIPMENT																	
* > $1,000	planned	0	1,000	0	0	0	0	0	0	0	0	0	0	$1,000			
	actual	0	0	0	0	0	0	0	1,226	0					$1,226	1,300	(1,526)
INDIRECT COSTS																	
Variable																	
43% / 23%	planned	1,942	2,272	1,712	2,042	1,712	3,155	2,989	2,989	1,713	1,713	1,713	1,713	$25,665			
On / Off Campus	actual	0	592	600	619	672	1,308	4,298	2,909	3,224					$14,222	6,179	5,264
PLANNED TOTALS: PER MONTH		9,686	13,951	10,391	9,786	8,456	10,399	13,603	13,603	8,457	8,457	8,457	8,457	$123,706			
ACTUAL TOTALS: PER MONTH		0	3,167	3,207	3,311	2,567	5,967	18,989	14,400	16,467	0	0	0		$68,076	32,034	23,597
CUMULATIVE																	
PLANNED TOTALS		9,686	23,638	34,029	43,815	52,271	62,671	76,274	89,877	98,334	106,792	115,249	123,706	$123,706			
ACTUAL TOTALS		0	3,167	6,374	9,685	12,252	18,219	37,209	51,608	68,076	68,076	68,076	68,076		$68,076		23,597
Budget Variance		$9,686	$20,471	$27,655	$34,130	$40,019	$44,451	$39,065	$38,269	$30,259	$38,716	$47,173	$55,631				



Sub-Task 1.3 - Evaluation of Acrylates as Grout Barriers for In-Situ Containment
PRINCIPAL INVESTIGATOR: M.A. Gabr

PROJECT: Development of Standard Test Protocols and Design Models for Dessication Barriers

PRINCIPAL INVESTIGATOR (S): K. Aminian & S. Ameri

METC TASK NO. 1.4

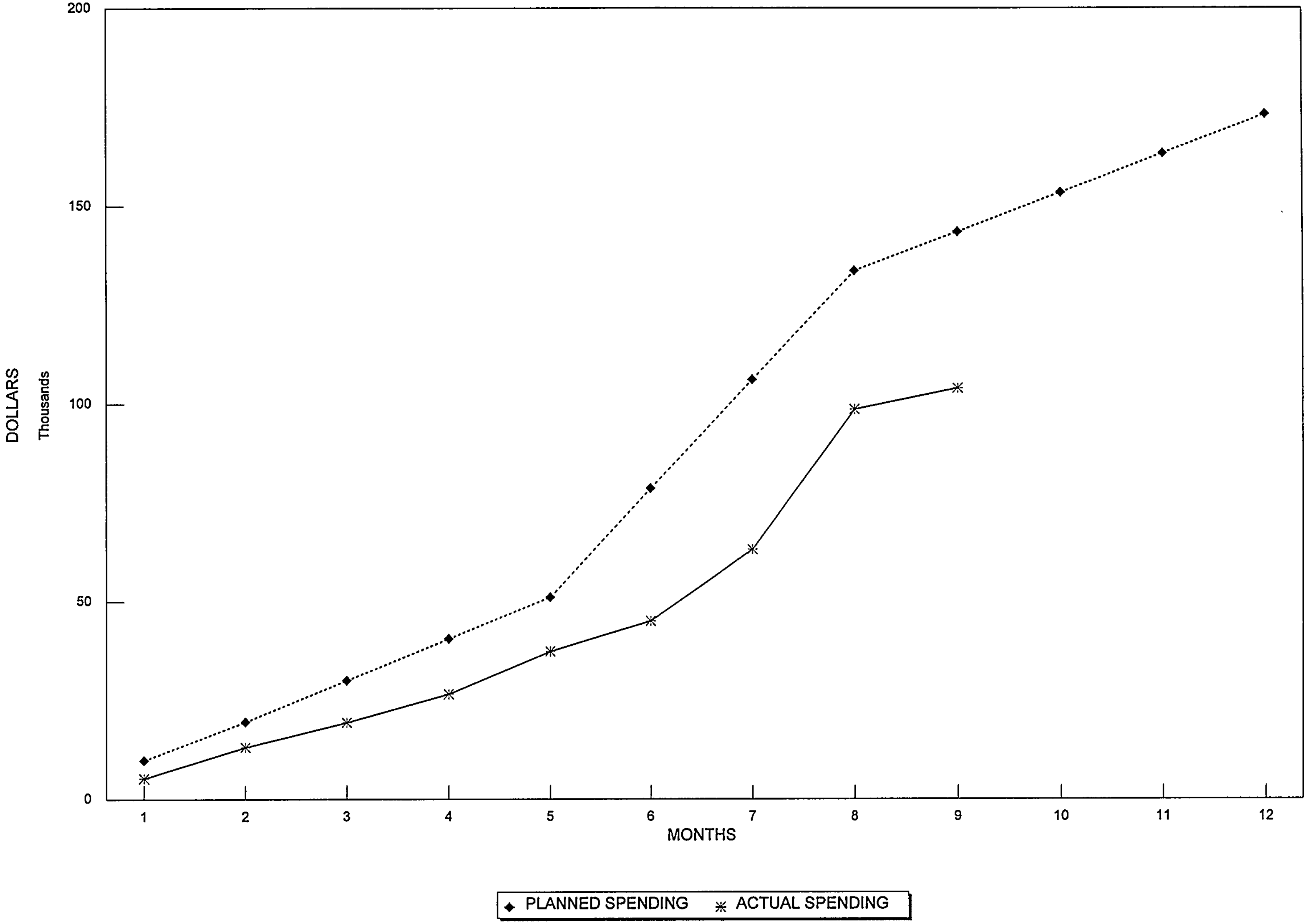
**** Summary on/off Campus**

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Full-time	planned	2,617	2,617	2,617	2,617	2,617	12,885	12,885	12,885	2,617	2,617	2,617	2,617	$62,208			
	actual	2,617	2,617	2,617	2,617	2,617	2,617	6,646	17,099	0					$39,447	9,000	13,761
Management	planned	609	609	609	609	609	609	609	609	609	609	609	609	$7,309			
	actual	0	0	0	0	1,824	0	873	873	873					$4,443	2,619	247
BENEFITS 29% of Full Time Salaries	planned	936	936	936	936	936	3,913	3,913	3,913	936	936	936	936	$20,160			
	actual	759	759	759	759	1,288	759	2,181	5,212	253	0	0	0		$12,728	3,370	4,062
STUDENTS Graduate	planned	3,000	3,000	3,000	3,000	3,000	3,000	3,000	3,000	3,000	3,000	3,000	3,000	$36,005			
	actual	870	2,570	1,720	1,720	1,920	2,120	4,795	3,865	2,925					$22,505	10,260	3,240
SUPPLIES	planned	125	125	125	125	125	125	125	125	125	125	125	125	$1,500			
	actual	0	447	0	777	801	610	80	0	129					$2,844	0	(1,344)
TRAVEL	planned	0	0	500	500	500	500	500	500	500	500	500	500	$5,000			
	actual	0	0	0	0	0	0	0	0	0					$0	0	5,000
OTHER	planned	42	42	42	42	42	42	42	42	42	42	42	42	$500			
	actual	0	0	0	0	0	47	0	0	0					$47	0	453
EQUIPMENT < $1,000	planned	500	500	500	500	500	0	0	0	0	0	0	0	$2,500			
	actual	0	0	0	0	0	0								$0	0	2,500
INDIRECT COSTS Variable Rate See On/Off Campus Detail	planned	1,958	1,958	2,173	2,173	2,173	6,434	6,434	6,434	2,058	2,058	2,058	2,058	$37,969			
	actual	977	1,470	1,172	1,351	2,414	1,415	3,577	8,342	1,187					$21,905	6,483	9,581
PLANNED TOTALS: PER MONTH		9,787	9,787	10,502	10,502	10,502	27,508	27,508	27,508	9,887	9,887	9,887	9,887	$173,151			
ACTUAL TOTALS: PER MONTH		5,223	7,863	6,268	7,224	10,864	7,568	18,152	35,391	5,367	0	0	0		$103,919	31,732	37,501
CUMULATIVE																	
PLANNED TOTALS		9,787	19,573	30,075	40,577	51,079	78,587	106,096	133,604	143,491	153,377	163,264	173,151	$173,151			
ACTUAL TOTALS		5,223	13,086	19,353	26,577	37,441	45,009	63,161	98,552	103,919	103,919	103,919	103,919		$103,919		37,501
Budget Variance		$4,564	$6,488	$10,722	$14,000	$13,637	$33,578	$42,935	$35,052	$39,572	$49,459	$59,345	$69,232				

Sub-Task 1.4 - Dessication Barriers *Summary on/off campus work*

PRINCIPAL INVESTIGATOR (S): K. Aminian & S. Ameri



PROJECT: Development of Standard Test Protocols and Design Models for Insitu Formed Barriers
<<<Technical Support - Cost Summary >>>

METC TASK NO.: 1.5

WVU ACCOUNT NO.: 8527-090-3237
PERIOD ENDING: September 30, 1996

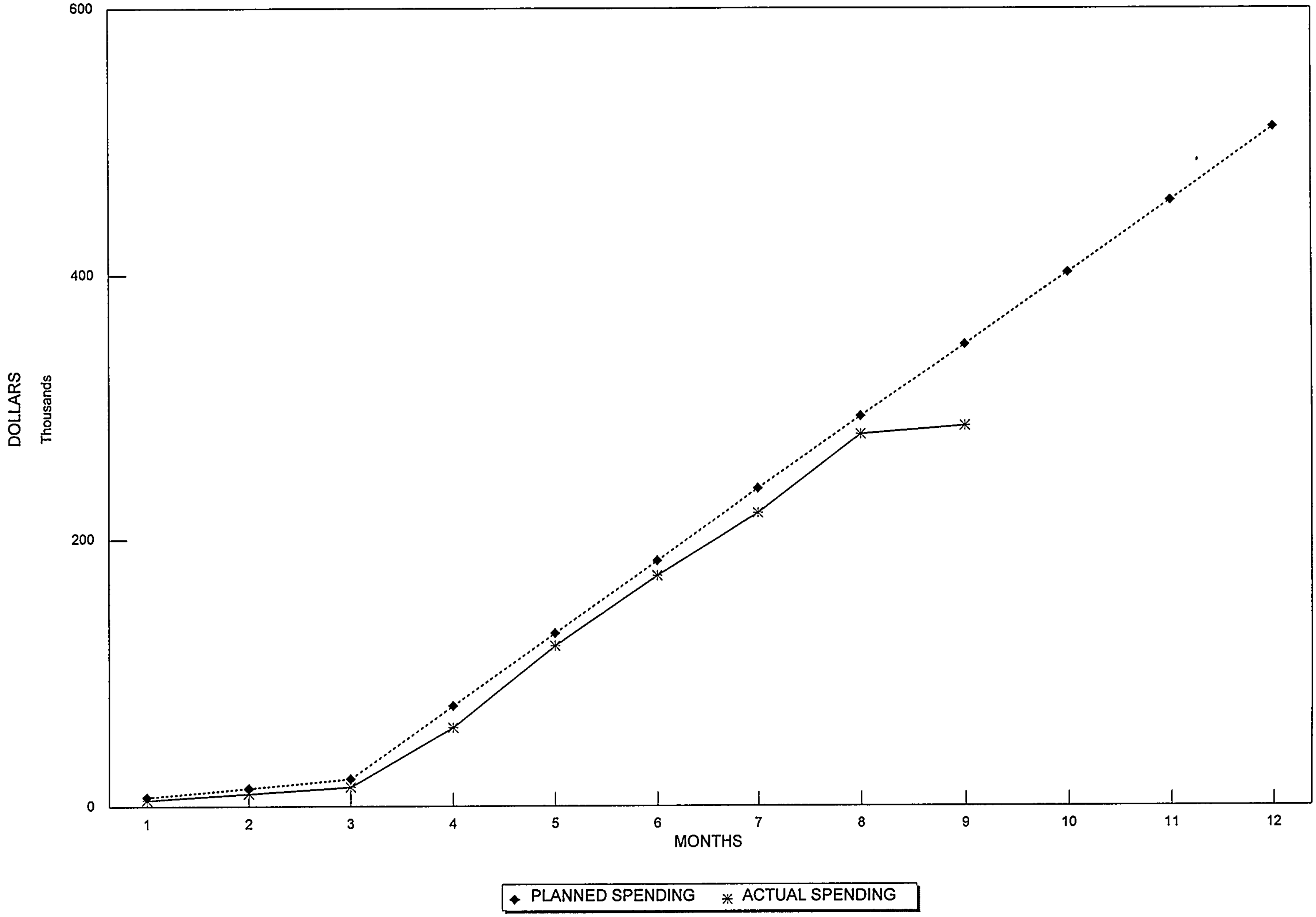
DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
Management SALARIES																	
Director	planned	950	950	950	950	950	950	950	950	950	950	950	950	$11,400			
	actual	475	475	475	1,742	1,742	1,742	0	533	1,065					$8,247	3,195	(42)
Assistants to the Director	planned	2,132	2,132	2,132	2,132	2,132	2,132	2,132	2,132	2,132	2,132	2,132	2,258	$25,707			
	actual	1,523	1,523	1,523	3,959	3,959	3,959	1,561	1,561	1,561					$21,129	4,683	(105)
Clerical Support	planned	600	600	600	600	600	600	600	600	600	600	600	600	$7,197			
	actual	600	600	600	0	0	0	875	875	875					$4,425	2,625	147
BENEFITS 29% of Full Time Salaries	planned	1,068	1,068	1,068	1,068	1,068	1,068	1,068	1,068	1,068	1,068	1,068	1,104	$12,848			
	actual	753	753	753	1,653	1,653	1,653	706	861	1,015	0	0	0		$9,802	3,046	0
SUB-CONTR. * > $25,000	planned	0	0	0	47,687	47,687	47,687	47,687	47,687	47,687	47,687	47,687	47,687	$429,187			
	actual	0	0	0	33,707	50,929	41,973	42,624	54,046	0					$223,279	205,908	0
INDIRECT COSTS 43% of Direct Costs except *	planned	2,042	2,042	2,042	2,042	2,042	2,042	2,042	2,042	2,042	2,042	2,042	2,112	$24,576			
	actual	1,441	1,441	1,441	3,162	3,162	3,162	1,351	1,647	1,942	0	0	0		$18,749	5,826	0
PLANNED TOTALS: PER MONTH		6,791	6,791	6,791	54,479	54,479	54,479	54,479	54,479	54,479	54,479	54,479	54,712	$510,916			
ACTUAL TOTALS: PER MONTH		4,793	4,793	4,793	44,223	61,445	52,488	47,118	59,522	6,458	0	0	0		$285,632	225,283	1
CUMULATIVE																	
PLANNED TOTALS		6,791	13,583	20,374	74,853	129,332	183,810	238,289	292,768	347,247	401,725	456,204	510,916	$510,916			
ACTUAL TOTALS		4,793	9,585	14,378	58,601	120,045	172,534	219,651	279,173	285,632	285,632	285,632	285,632		$285,632		1
Budget Variance		$1,999	$3,998	$5,996	$16,252	$9,286	$11,277	$18,638	$13,595	$61,615	$116,094	$170,573	$225,284				

** Sub-Contractor Detail Follows

Note: Budget Increased by $126 from last quarter to reflect salary Increases for fiscal year 1997

SubTask 1.5 - Standard Test Protocols & Design Models - Insitu Barriers

PRINCIPAL INVESTIGATOR (S): B. Overby and C. Locke - *** SUMMARY ***



PROJECT: Development of Standard Test Protocols and Design Models for Insitu Formed Barriers
<<<Technical Support >>>

METC TASK NO.: 1.5 *** Sub-Contractor Detail ***
BDM Federal

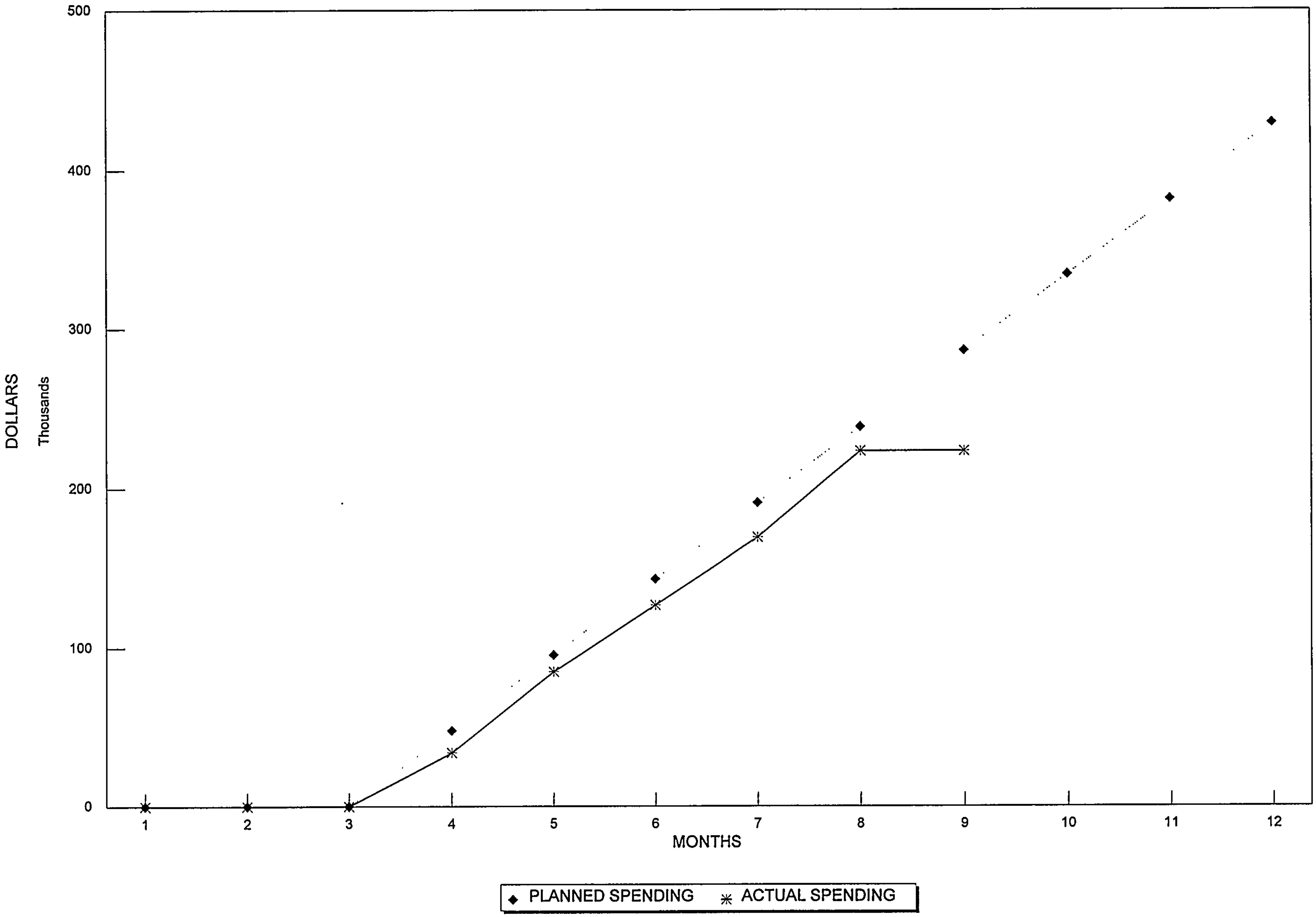
PRINCIPAL INVESTIGATOR (S): B. Overby & C. Locke

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Amount Remaining
Direct Labor and Loadings	planned	0	0	0	35,264	35,264	35,264	35,264	35,264	35,264	35,264	35,264	35,264	$317,373		
	actual				15,821	38,815	31,796	32,486	41,284						$160,202	157,171
EB Expense	planned	0	0	0	7,778	7,778	7,778	7,778	7,778	7,778	7,778	7,778	7,778	$70,000		
	actual				2,907	6,982	5,707	5,843	7,414						$28,853	41,147
Direct Materials	planned	0	0	0	0	0	0	0	0	0	0	0	0	$0		
	actual				0	0	0	0	0						$0	0
Freight & Postage	planned	0	0	0	0	0	0	0	0	0	0	0	0	$0		
	actual				0	0	0	0	0						$0	0
Publications Support	planned	0	0	0	0	0	0	0	0	0	0	0	0	$0		
	actual				0	0	0	0	0						$0	0
TRAVEL	planned	0	0	0	559	559	559	559	559	559	559	559	559	$5,035		
	actual				0	0	240	0	225						$465	4,570
SUB-TOTAL	planned	0	0	0	43,601	43,601	43,601	43,601	43,601	43,601	43,601	43,601	43,601	$392,408		
	actual	0	0	0	18,728	45,797	37,743	38,329	48,923	0	0	0	0		$189,520	202,888
Fee	planned	0	0	0	4,086	4,086	4,086	4,086	4,086	4,086	4,086	4,086	4,086	$36,778		
	actual	0	0	0	14,979	5,132	4,230	4,295	5,123	0	0	0	0		$33,759	3,019
PLANNED TOTALS: PER MONTH		0	0	0	47,687	47,687	47,687	47,687	47,687	47,687	47,687	47,687	47,687	$429,186		
ACTUAL TOTALS: PER MONTH		0	0	0	33,707	50,929	41,973	42,624	54,046	0	0	0	0		$223,279	205,907
CUMULATIVE																
PLANNED TOTALS		0	0	0	47,687	95,375	143,062	190,749	238,437	286,124	333,811	381,499	429,186	$429,186		
ACTUAL TOTALS		0	0	0	33,707	84,636	126,609	169,233	223,279	223,279	223,279	223,279	223,279		$223,279	205,907
Budget Variance		$0	$0	$0	$13,980	$10,738	$16,453	$21,516	$15,158	$62,845	$110,532	$158,220	$205,907			

Sub-Task 1.5: BDM Federal - Technical Support for Insitu Barriers

PRINCIPAL INVESTIGATOR (S): B. Overby & C. Locke



PROJECT: In Situ Bioremediation of Chlorinated Solvents at PORTS

METC TASK NO.: 1.6

WVU ACCOUNT NO.: 8527-090-3252

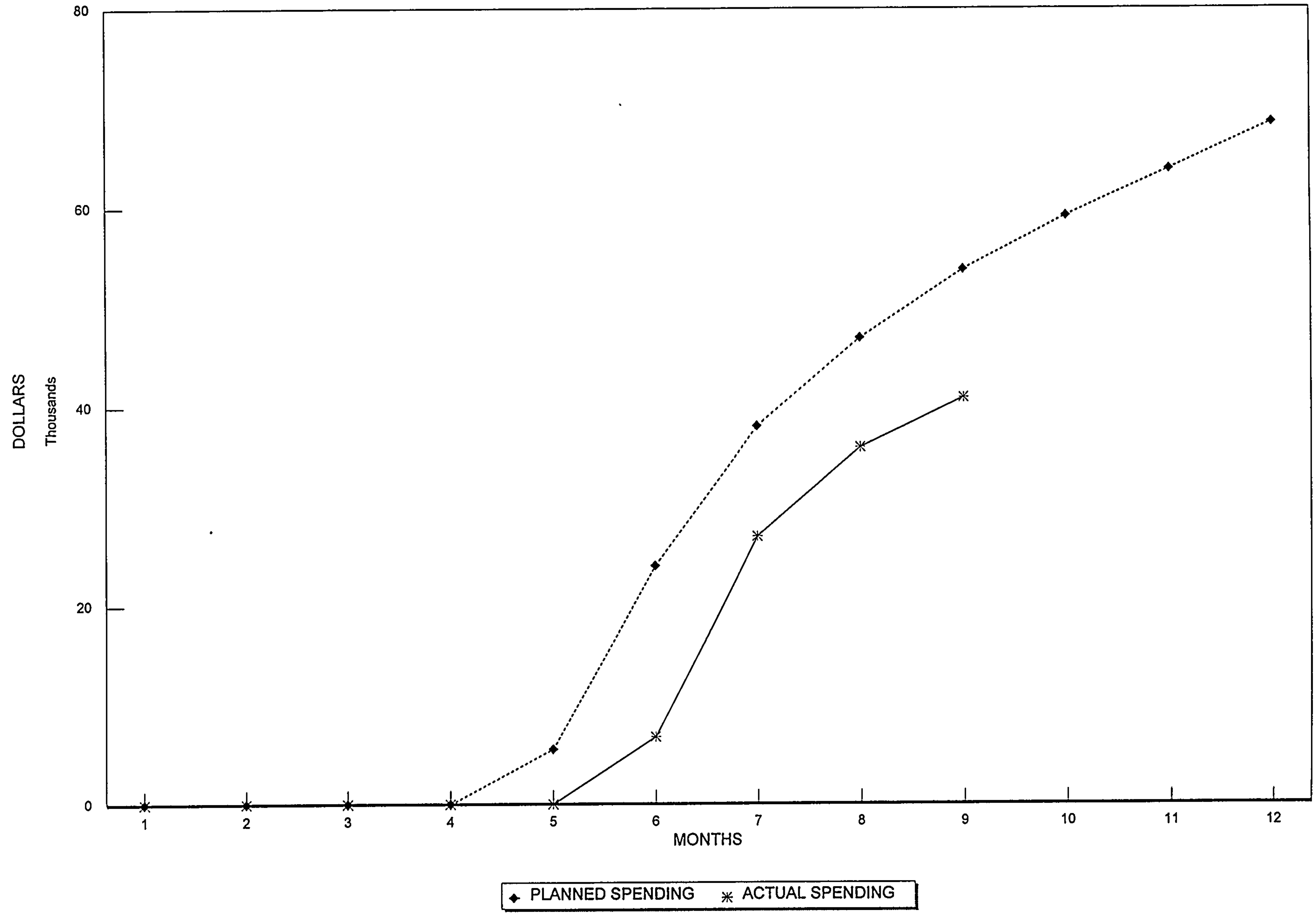
PRINCIPAL INVESTIGATOR (S): P. Carriere, W. Sack & W. Lin

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
P. Carrier	planned	0	0	0	0	973	1,947	1,947	973	0	0	0	0	$5,840			
	actual					0	0	3,893	1,947	0					$5,840	0	1
W. Sack	planned	0	0	0	0	870	1,740	1,740	870	0	0	0	0	$5,220			
	actual	0	0	0	0	0	3480	1856.27	0	0					$5,336	0	(116)
W. Lin	planned	0	0	0	0	429	429	429	429	429	429	429	429	$3,432			
	actual	0	0	0	0	0	0	0	0	0					$0	0	3,432
BENEFITS 29% of Full Time Salaries	planned	0	0	0	0	659	1,194	1,194	659	124	124	124	124	$4,203			
	actual	0	0	0	0	0	1,009	1,667	564	0	0	0	0		$3,241	0	962
STUDENTS Graduate	planned	0	0	0	0	0	1,800	1,800	1,800	1,800	1,800	1,800	1,800	$12,600			
	actual	0	0	0	0	0	250	2,683	1,700	1,700					$6,333	5,100	1,167
Undergraduate	planned	0	0	0	0	0	0	0	0	0	0	0	0	$0			
	actual	0	0	0	0	0	0	0	0	0					$0	0	0
SUPPLIES	planned	0	0	0	0	875	875	875	875	875	875	875	875	$7,000			
	actual					0	0	4,012	2,049	854					$6,915	1,618	(1,533)
TRAVEL	planned	0	0	0	0	0	700	0	500	0	500	0	0	$1,700			
	actual					0	0	0	0	0					$0	0	1,700
OTHER	planned	0	0	0	0	63	63	63	63	63	63	63	63	$500			
	actual					0	0	0	0	0					$0	0	500
EQUIPMENT < $1,000	planned	0	0	0	0	0	0	0	0	0	0	0	0	$0			
	actual					0	0	0	0	940					$940	0	(940)
* > $1,000	planned	0	0	0	0	0	6,000	2,500	0	0	0	0	0	$8,500			
	actual					0	0	0	0	0					$0	0	8,500
SUB-CONTR. < $25,000	planned	0	0	0	0	0	0	0	0	1,500	0	0	0	$1,500			
	actual					0	0	0	0	0					$0	0	1,500
INDIRECT COSTS 43% of Direct Costs except *	planned	0	0	0	0	1,664	3,761	3,460	2,653	2,060	1,630	1,415	1,415	$18,058			
	actual	0	0	0	0	0	2,038	6,068	2,692	1,502	0	0	0		$12,300	2,889	2,869
PLANNED TOTALS: PER MONTH		0	0	0	0	5,532	18,508	14,007	8,821	6,851	6,421	4,706	4,706	$68,552			
ACTUAL TOTALS: PER MONTH		0	0	0	0	0	6,777	20,180	8,952	4,996	0	0	0		$40,904	9,607	18,041
CUMULATIVE																	
PLANNED TOTALS		0	0	0	0	5,532	24,040	38,047	46,868	53,719	59,140	63,846	68,552	$68,552			
ACTUAL TOTALS		0	0	0	0	0	6,777	26,957	35,909	40,904	40,904	40,904	40,904		$40,904		18,041
Budget Variance		$0	$0	$0	$0	$5,532	$17,263	$11,090	$10,960	$12,815	$18,236	$22,942	$27,648				

Sub-Task 1.6: In Situ Bioremediation of Chlorinated Solvents

PRINCIPAL INVESTIGATOR (S): P. Carriere & W. Sack



PROJECT: Development and Implementation of a Decision Support System for Management of the EM-50 Technology Development Program

METC TASK NO.: 1.7A

WVU ACCOUNT NO.: 8527-090-3255

**** Summary of Costs ***

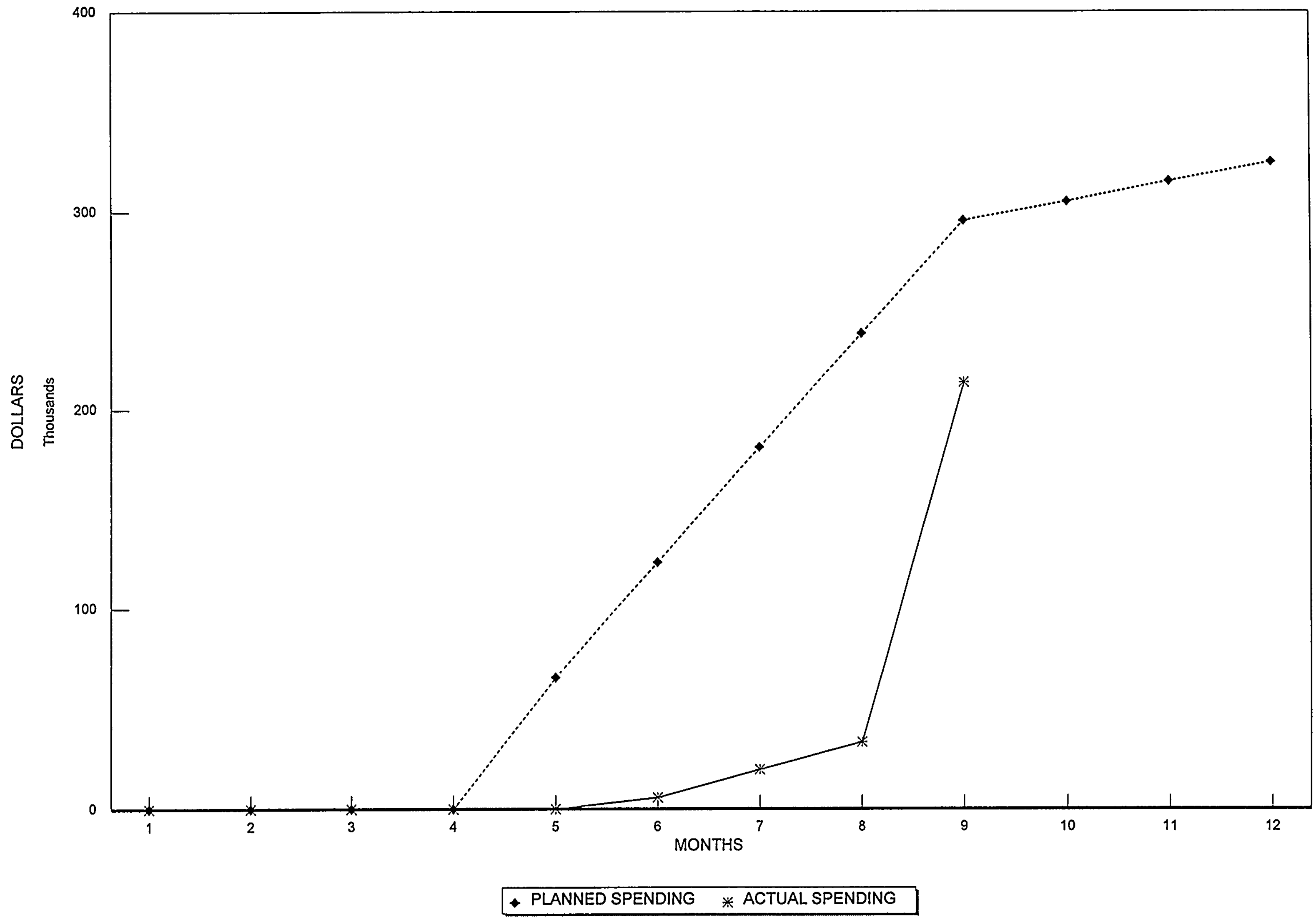
PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Management Salaries	planned	0	0	0	0	0	0	0	0	0	1,807	1,807	1,807	$5,421			
	actual	0	0	0	0	0	0	0	0	0					$0	0	5,421
BENEFITS 29% of Full Time Salaries	planned	0	0	0	0	0	0	0	0	0	524	524	524	$1,572			
	actual	0	0	0	0	0	0	0	0	0	0	0	0		$0	0	1,572
Sub-Contractor < $25,000	planned	0	0	0	0	25,000	0	0	0	0	0	0	0	$25,000			
	actual	0	0	0	0	0	4,691	11,352	8,957	0					$25,000	0	0
Sub-Contractor > $25,000	planned	0	0	0	0	35,226	57,221	57,822	57,221	57,221	6,200	6,801	6,200	$283,912			
	actual	0	0	0	0	0	0	0	2,619	180,018					$182,637	101,275	0
INDIRECT COSTS Variable (On/Off)	planned	0	0	0	0	5,750	0	0	0	0	1,002	1,002	1,002	$8,757			
	actual	0	0	0	0	0	1,079	2,611	2,060	0	0	0	0		$5,750	0	3,007
PLANNED TOTALS: PER MONTH		0	0	0	0	65,976	57,221	57,822	57,221	57,221	9,533	10,134	9,533	$324,662			
ACTUAL TOTALS: PER MONTH		0	0	0	0	0	5,770	13,963	13,636	180,018	0	0	0		$213,387	101,275	10,000
CUMULATIVE																	
PLANNED TOTALS		0	0	0	0	65,976	123,197	181,019	238,240	295,461	304,994	315,129	324,662	$324,662			
ACTUAL TOTALS		0	0	0	0	0	5,770	19,733	33,369	213,387	213,387	213,387	213,387		$213,387		10,000
Budget Variance		$0	$0	$0	$0	$65,976	$117,427	$161,286	$204,871	$82,074	$91,607	$101,742	$111,275				

*Sub-Contractor Detail Follows

Sub-Task 1.7A: DDS for Management of the EM-50 Technology

Development Program - Budget Summary



PROJECT: Development and Implementation of a Decision Support System for Management of the EM-50 Technology Development Program

METC TASK NO.: 1.7A

*** Sub-Contractor Detail **

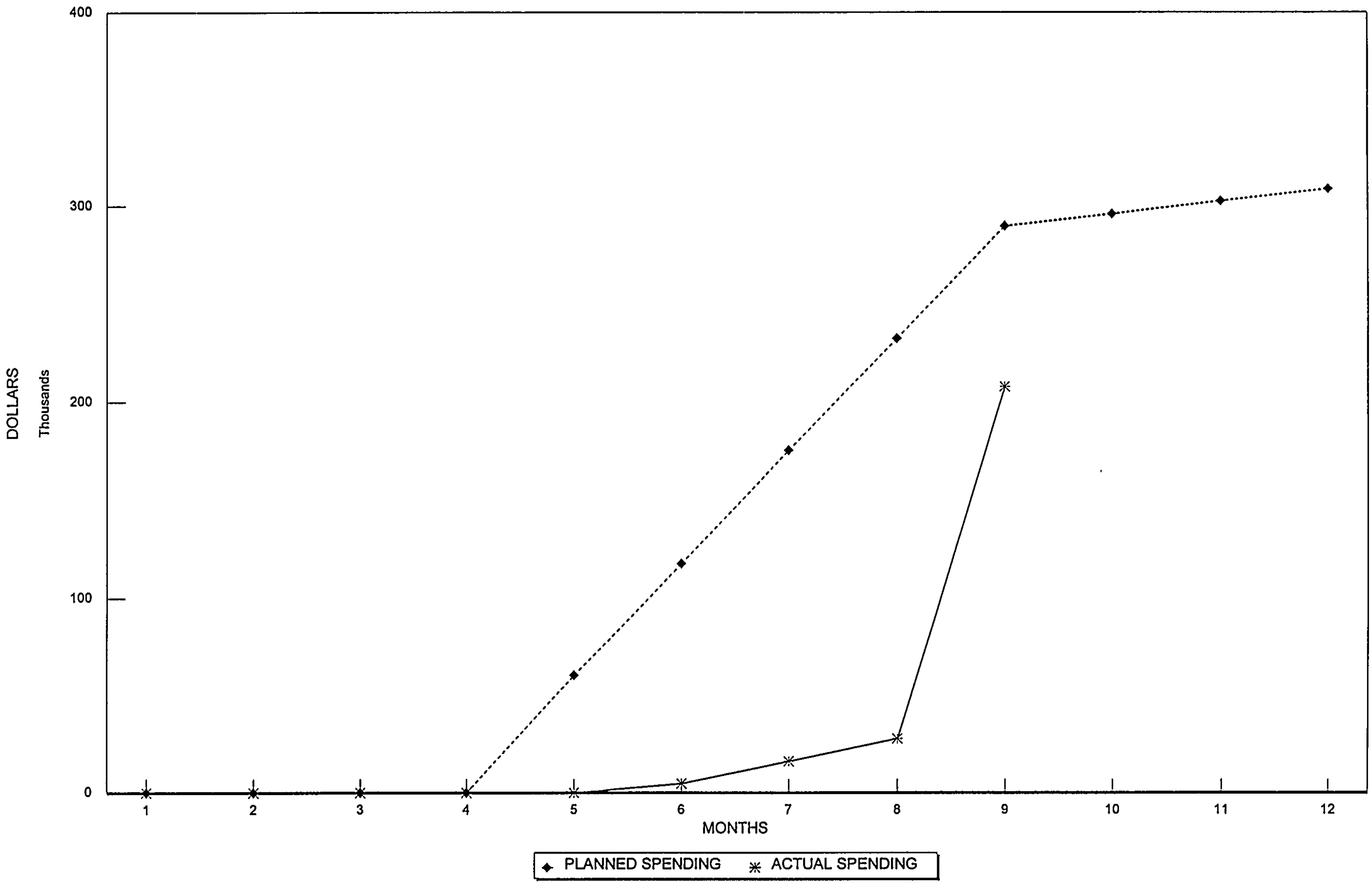
PRINCIPAL INVESTIGATOR: Ray Malinovsky
Wast Policy Institute

PERIOD ENDING: 9/30/96

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Amount Remaining
SALARIES																
Principal Invest. R. Malinousky	planned	0	0	0	0	5,500	5,500	5,500	5,500	5,500	556	556	556	$29,167		
	actual	0	0	0	0	0	1,360	370	4,352	942					$7,024	22,143
Fringe and Overhead	planned	0	0	0	0	4,103	4,103	4,103	4,103	4,103	415	415	415	$21,759		
	actual	0	0	0	0	0	1,015	749	2,690	649	0	0	0		$5,103	16,656
Data Entry Support	planned	0	0	0	0	500	500	500	500	0	0	0	0	$2,000		
	actual	0	0	0	0	0	0	0	0	0					$0	2,000
Programmer Support	planned	0	0	0	0	7,500	7,500	7,500	7,500	7,500	2,500	2,500	2,500	$45,000		
	actual	0	0	0	0	0	9	6,226	2,458	4,697					$13,390	31,610
Software	planned	0	0	0	0	2,000	0	0	0	0	0	0	0	$2,000		
	actual	0	0	0	0	0	0	865	0	0					$865	1,135
Travel	planned	0	0	0	0	500	0	500	0	500	0	500	0	$2,000		
	actual	0	0	0	0	0	1,519	654	0	1,832					$4,005	(2,005)
Virginia Tech SubContract	planned	0	0	0	0	30,000	30,000	30,000	30,000	30,000	1,688	1,688	1,688	$155,064		
	actual	0	0	0	0	0	0	0	0	139,973					$139,973	15,091
General and Administrative	planned	0	0	0	0	7,265	6,893	6,965	6,893	6,893	747	819	747	$37,212		
	actual	0	0	0	0	0	565	1,947	1,597	24,894					$29,003	8,209
FEE (5%)	planned	0	0	0	0	2,868	2,725	2,753	2,725	2,725	295	324	295	$14,710		
	actual	0	0	0	0	0	223	541	479	7,031					$8,274	6,436
PLANNED TOTALS: PER MONTH		0	0	0	0	60,226	57,221	57,822	57,221	57,221	6,200	6,801	6,200	$308,912		
ACTUAL TOTALS: PER MONTH		0	0	0	0	0	4,691	11,352	11,576	180,018	0	0	0		$207,637	101,275
CUMULATIVE																
PLANNED TOTALS		0	0	0	0	60,226	117,446	175,268	232,489	289,710	295,910	302,711	308,912	$308,912		
ACTUAL TOTALS		0	0	0	0	0	4,691	16,043	27,619	207,637	207,637	207,637	207,637		$207,637	101,275
Budget Variance		$0	$0	$0	$0	$60,226	$112,756	$159,226	$204,870	$82,073	$88,273	$95,075	$101,275			

Sub-Task 1.7A: DDS for Management of the EM-50 Technology

Development Program - *Sub-Contractor Detail* - PRINCIPAL INVESTIGATOR: R. Malinousky, Waste Policy Institute



PROJECT: Development of a Prototype Database and Decision Support System for Management of the EM-50 Technology Development Program

METC TASK NO.: 1.7B

WVU ACCOUNT NO.: 8527-078-0304

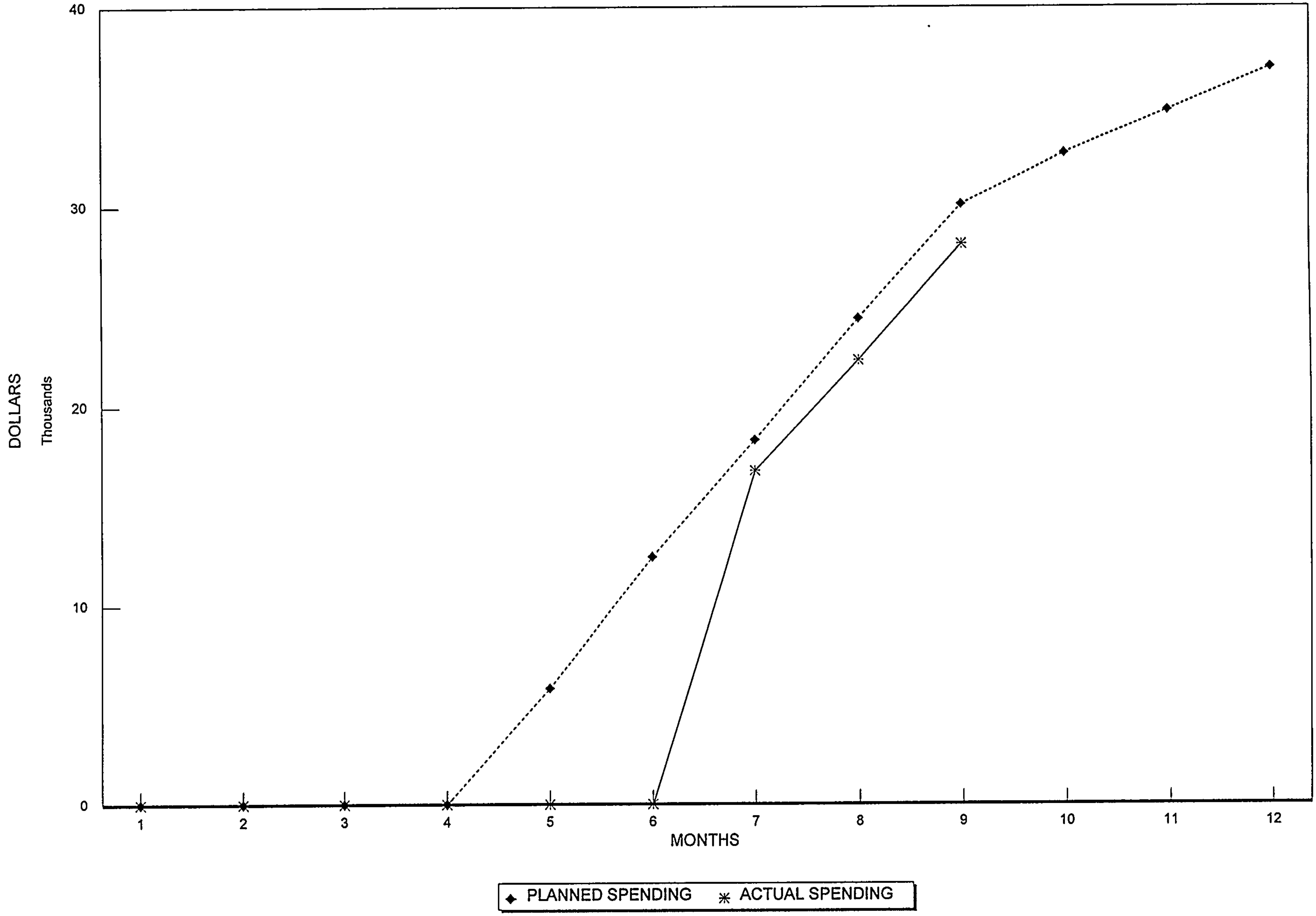
PRINCIPAL INVESTIGATOR: James Denton
College of Business

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Principal Invest.	planned	0	0	0	0	3,000	3,000	3,000	3,000	3,000	1,159	1,159	1,159	$18,476			
James Denton	actual	0	0	0	0	0	0	9000	3000	3000					$15,000	3,477	(1)
BENEFITS																	
29% of Full	planned	0	0	0	0	870	870	870	870	870	336	336	336	$5,358			
Time Salaries	actual	0	0	0	0	0	0	2,610	870	870	0	0	0		$4,350	1,008	(0)
SUPPLIES	planned	0	0	0	0	200	200	200	100	100	0	0	0	$800			
	actual	0	0	0	0	0	0	0	0	0					$0	0	800
TRAVEL	planned	0	0	0	0	0	500	0	250	0	250	0	0	$1,000			
	actual	0	0	0	0	0	0	111	0	189					$300		700
OTHER	planned	0	0	0	0	25	50	25	25	25	50	0	0	$200			
	actual	0	0	0	0	0	0	0	0	0					$0	0	200
INDIRECT COSTS																	
43% of Direct	planned	0	0	0	0	1,761	1,987	1,761	1,825	1,718	772	643	643	$11,109			
Costs except *	actual	0	0	0	0	0	0	5,040	1,664	1,745	0	0	0		$8,450	1,929	730
PLANNED TOTALS: PER MONTH		0	0	0	0	5,856	6,607	5,856	6,070	5,713	2,566	2,137	2,137	$36,943			
ACTUAL TOTALS: PER MONTH		0	0	0	0	0	0	16,762	5,534	5,804	0	0	0		$28,100	6,414	2,429
CUMULATIVE																	
PLANNED TOTALS		0	0	0	0	5,856	12,462	18,318	24,389	30,102	32,668	34,805	36,943	$36,943			
ACTUAL TOTALS		0	0	0	0	0	0	16,762	22,296	28,100	28,100	28,100	28,100		$28,100		2,429
Budget Variance		$0	$0	$0	$0	$5,856	$12,462	$1,557	$2,093	$2,002	$4,568	$6,706	$8,843				

Sub-Task 1.7B: Prototype Database & Decision Support System

for Management of the EM-50 Technology Development Program - PRINCIPAL INVESTIGATOR: James Denton



TASK 2.0

Cross Cutting Innovative Technologies

PROJECT: A GIS Based Infrastructure for Site Characterization & Remediation

METC TASK NO.: 2.1

WVU Account No.: 8527-090-3247

PERIOD ENDING: September 30, 1996

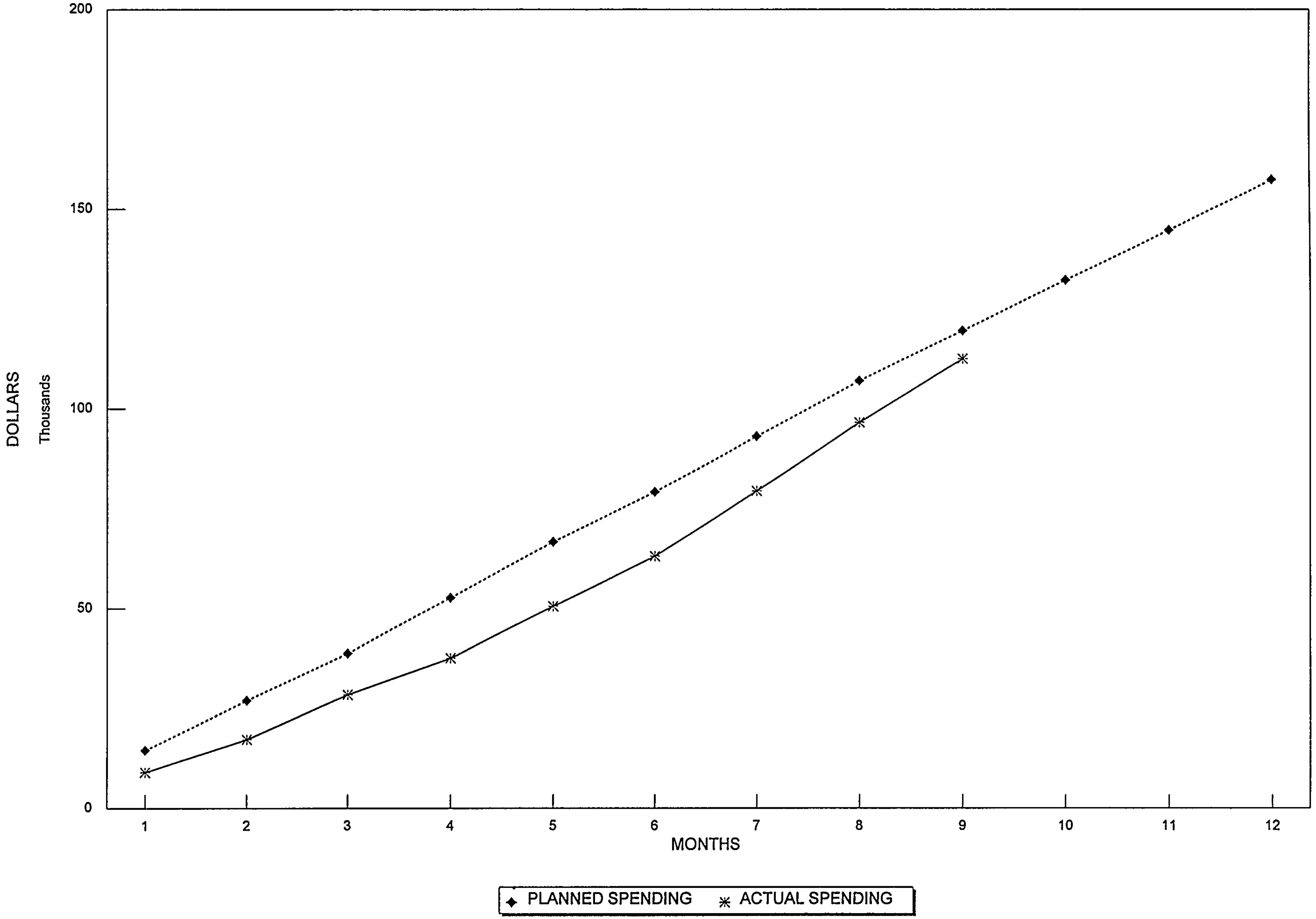
** Cost Summary **

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Director	planned	475	475	475	475	475	475	475	475	475	475	475	475	$5,700			
	actual	0	0	0	0	0	1,425	0	291	581					$2,297	1,743	1,661
Assistant Director	planned	305	305	305	305	305	305	305	305	305	305	305	305	$3,655			
	actual	0	0	0	0	0	0	979	835	622					$2,436	1,886	(667)
BENEFITS 29% of Full Time Salaries	planned	226	226	226	226	226	226	226	226	226	226	226	226	$2,713			
	actual	0	0	0	0	0	413	284	326	349	0	0	0		$1,372	1,052	288
SUB-CONTR. * > $25,000	planned	13,172	10,972	10,420	12,430	12,430	11,050	12,520	12,520	11,130	11,130	11,130	11,130	$140,034			
	actual	8,979	8,340	11,111	9,282	12,844	9,780	14,603	15,087	13,701					$103,727	36,307	0
INDIRECT COSTS 43% of Direct Costs except *	planned	432	432	432	432	432	432	432	432	432	432	432	432	$5,189			
	actual	0	0	0	0	0	790	543	624	667	0	0	0		$2,625	2,013	551
PLANNED TOTALS: PER MONTH		14,610	12,410	11,858	13,868	13,868	12,488	13,958	13,958	12,568	12,568	12,568	12,568	$157,291			
ACTUAL TOTALS: PER MONTH		8,979	8,340	11,111	9,282	12,844	12,409	16,409	17,163	15,920	0	0	0		$112,457	43,001	1,832
CUMULATIVE																	
PLANNED TOTALS		14,610	27,020	38,878	52,746	66,614	79,102	93,060	107,019	119,587	132,155	144,723	157,291	$157,291			
ACTUAL TOTALS		8,979	17,319	28,430	37,712	50,556	62,965	79,374	96,537	112,457	112,457	112,457	112,457		$112,457		1,832
Budget Variance		$5,631	$9,701	$10,448	$15,034	$16,058	$16,138	$13,687	$10,482	$7,130	$19,698	$32,266	$44,834				

* Sub Contractor Detail Follows

SubTask 2.1 - A GIS Based Infrastructure for Characterization & Remediation

Overall Summary



PROJECT: A GIS Based Infrastructure for Site Characterization and Remediation

METC TASK NO.: 2.1

*** Sub-Contractor Detail ***

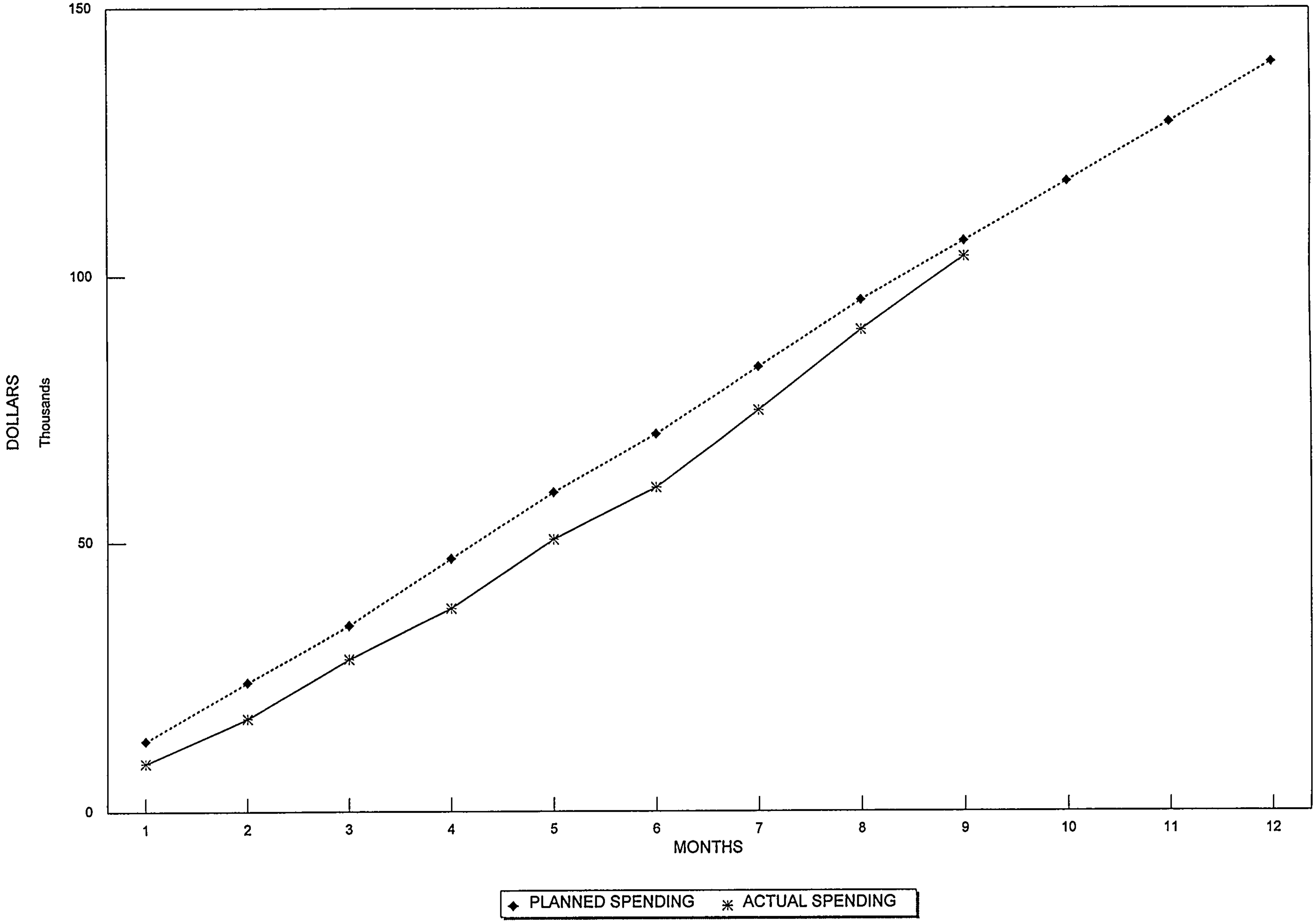
PRINCIPAL INVESTIGATOR (S): J. Hooper & A. Lodgher
Marshall University

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Amount Remaining
SALARIES	planned	5,432	5,432	5,432	5,432	5,432	5,432	5,432	5,432	5,432	5,432	5,432	5,432	$65,182		
	actual	5,641	5,110	5,368	5,497	5,206	5,461	6,710	6,367	6,134					$51,493	13,689
BENEFITS	planned	869	869	869	869	869	869	869	869	869	869	869	869	$10,429		
	actual	862	768	1,088	1,089	1,033	1,098	1,379	1,299	1,268					$9,884	545
STUDENT Salaries	planned	0	0	0	1,456	1,456	1,456	1,456	1,456	1,456	1,456	1,456	1,456	$13,108		
	actual	0	0	0	0	1,473	1,483	1,946	1,753	757					$7,412	5,696
GENERAL EXPENSE	planned	250	250	250	250	250	250	250	250	250	250	250	250	$3,000		
	actual	5	165	13	82	294	10	31	134	157					$891	2,109
TRAVEL	planned	0	0	1,000	1,000	1,000	0	1,000	1,000	0	0	0	0	$5,000		
	actual	0	0	418	0	0	0	0	616	0					$1,034	3,966
EQUIPMENT < $1,000	planned	1,400	1,400	0	0	0	0	0	0	0	0	0	0	$2,800		
	actual	0	0	1,165	58	1,301	(965)	440	685	1,541					$4,225	(1,425)
* > $1,000	planned	2,200	0	0	0	0	0	0	0	0	0	0	0	$2,200		
	actual	0	0	0	0	0	0	0	0	0					$0	2,200
INDIRECT COSTS Average 38.5% Direct Costs Except *	planned	3,021	3,021	2,869	3,423	3,423	3,043	3,513	3,513	3,123	3,123	3,123	3,123	$38,318		
	actual	2,473	2,296	3,060	2,556	3,537	2,693	4,097	4,233	3,844	0	0	0		$28,789	9,529
PLANNED TOTALS: PER MONTH		13,172	10,972	10,420	12,430	12,430	11,050	12,520	12,520	11,130	11,130	11,130	11,130	$140,037		
ACTUAL TOTALS: PER MONTH		8,979	8,340	11,111	9,282	12,844	9,780	14,603	15,087	13,701	0	0	0		$103,727	36,310
CUMULATIVE																
PLANNED TOTALS		13,172	24,145	34,565	46,995	59,425	70,475	82,996	95,516	106,646	117,776	128,907	140,037	$140,037		
ACTUAL TOTALS		8,979	17,319	28,430	37,712	50,555	60,336	74,939	90,026	103,727	103,727	103,727	103,727		$103,727	36,310
Budget Variance		$4,193	$6,826	$6,135	$9,283	$8,870	$10,140	$8,057	$5,490	$2,919	$14,049	$25,179	$36,310			

Sub-Task 2.1: GIS Infrastructure for Characterization & Remediation

PRINCIPAL INVESTIGATOR (S): J. Hooper & A. Lodgher - <<< Marshall University Detail >>>



PROJECT: Treatment of Mixed Wastes via Fluidized Bed Steam Reforming

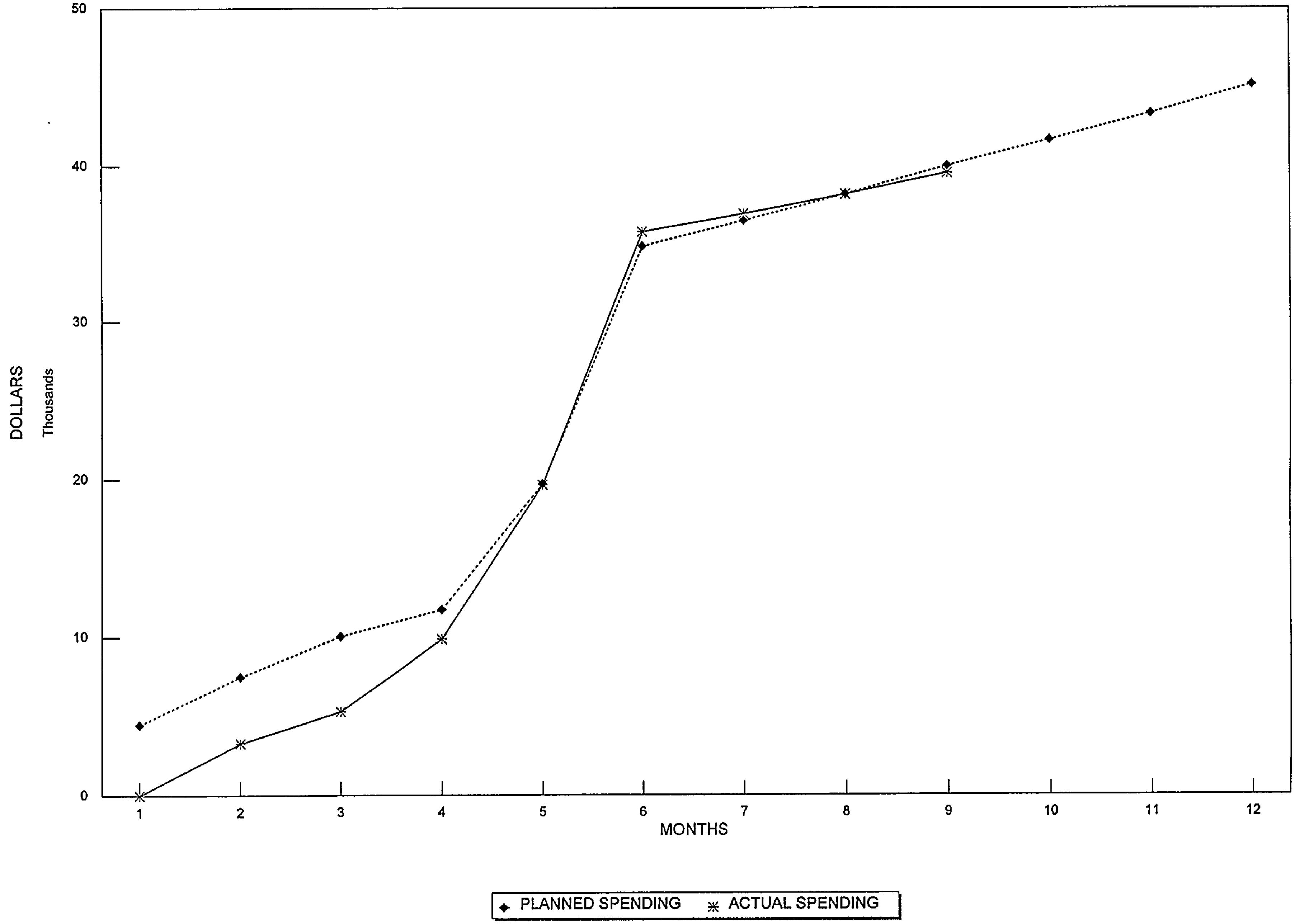
PRINCIPAL INVESTIGATOR: R. Turton

METC TASK NO.: 2.2
WVU ACCOUNT NO.: 8527-089-2406
PERIOD ENDING: 9/30/96

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
R. Turton	planned	0	0	0	0	3,417	6,835	0	0	0	0	0	0	$10,252			
	actual	0	0	0	0	3,417	6,835	0	0	0					$10,252	0	(0)
BENEFITS 29% of Full Time Salaries	planned	0	0	0	0	991	1,982	0	0	0	0	0	0	$2,973			
	actual	0	0	0	0	991	1,982	0	0	0	0	0	0		$2,973	0	(0)
STUDENTS Graduate	planned	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	$12,002			
	actual	0	2,200	1,100	1,100	1,740	640	760	749	778					$9,067	2,334	601
SUPPLIES	planned	167	167	167	167	167	167	167	167	167	167	167	167	$2,000			
	actual	0	101	305	193	671	1,824	48	122	40					$3,304	0	(1,304)
TRAVEL	planned	0	0	500	0	0	500	0	0	0	0	0	0	$1,000			
	actual	0	0	0	127	0	0	0	0	125					$253	47	700
OTHER	planned	0	0	100	0	0	100	0	0	100	0	0	100	$400			
	actual	0	0	0	0	0	0	0	0	0					$0	0	400
EQUIPMENT < $1,000	planned	0	970	0	0	0	0	0	0	0	0	0	0	$970			
	actual	0	0	0	0	0	0	0	0	0					$0	0	970
* > $1,000	planned	2,800	0	0	0	0	0	0	0	0	0	0	0	$2,800			
	actual	0	0	0	2,545	0	0	0	0	0					$2,545	0	255
INDIRECT COSTS 43% of Direct Costs except *	planned	502	919	760	502	2,397	4,551	502	502	545	502	502	546	$12,727			
	actual	0	989	604	611	2,933	4,851	347	374	406	0	0	0		$11,115	1,024	588
PLANNED TOTALS: PER MONTH		4,469	3,056	2,527	1,669	7,972	15,134	1,669	1,669	1,812	1,669	1,669	1,812	$45,123			
ACTUAL TOTALS: PER MONTH		0	3,290	2,009	4,576	9,752	16,131	1,155	1,245	1,349	0	0	0		$39,508	3,404	2,211
CUMULATIVE																	
PLANNED TOTALS		4,469	7,524	10,051	11,719	19,692	34,826	36,495	38,163	39,975	41,643	43,312	45,123	$45,123			
ACTUAL TOTALS		0	3,290	5,299	9,876	19,628	35,759	36,914	38,159	39,508	39,508	39,508	39,508		$39,508		2,211
Budget Variance		$4,469	$4,234	$4,751	$1,843	$63	($933)	($420)	$4	$466	$2,135	$3,803	$5,615				

SubTask 2.2 - Trmt of Mixed Waste via Fluidized Bed Steam Reforming

PRINCIPAL INVESTIGATOR: R. Turton



PROJECT: Use of Centrifugal Membrane Technology w/ Novel Membranes to Treat Hazardous/Radio Active Waste

METC TASK NO.: 2.3
WVU ACCOUNT NO.: 8527-090-3236

PRINCIPAL INVESTIGATOR: B. Reed

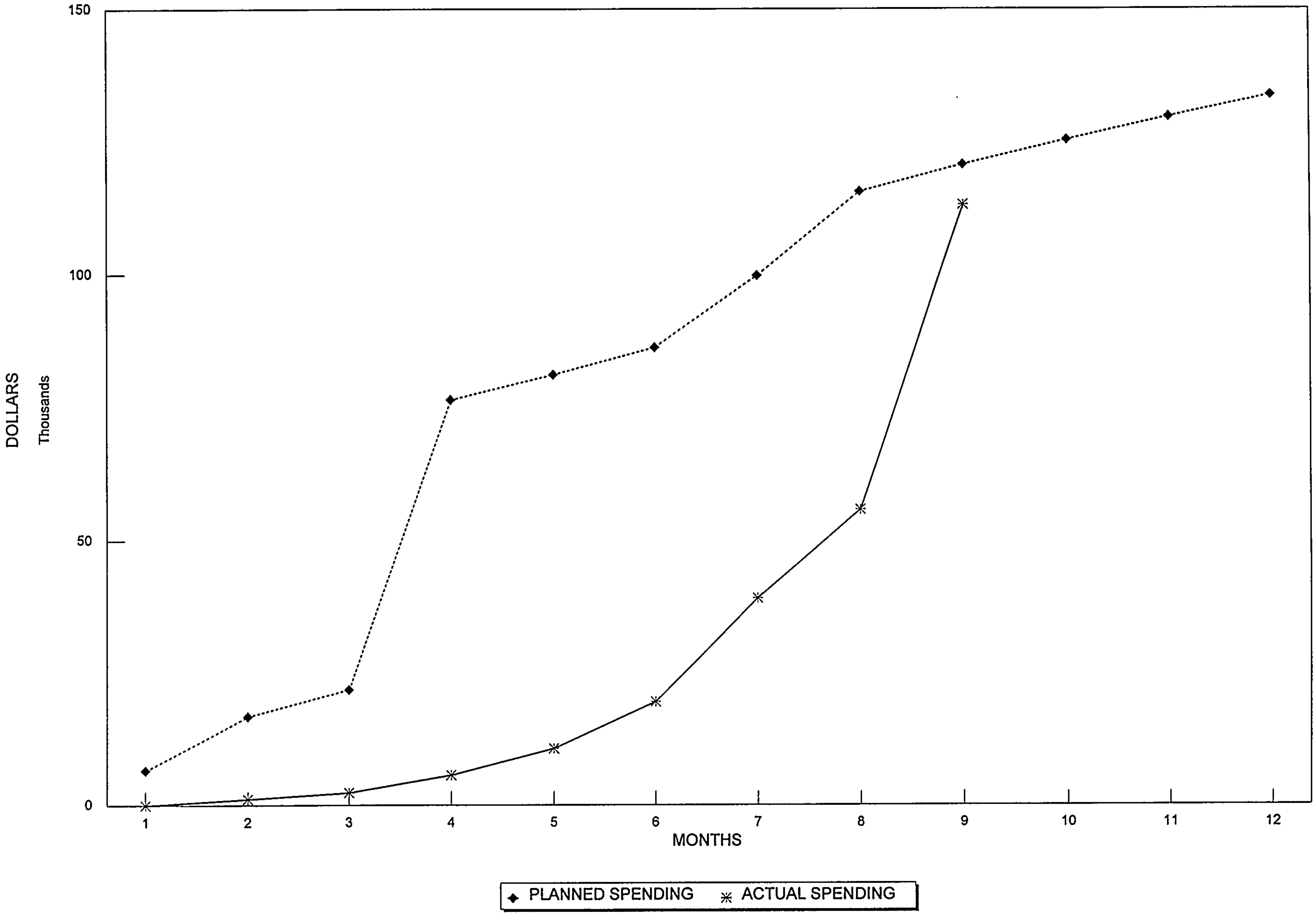
PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Principal B. Reed	planned	0	0	0	0	0	0	3,580	6,100	0	0	0	0	$9,680			
	actual	0	0	0	0	0	0	6,100	3,050	0					$9,150	0	530
Management T. Beatty	planned	305	305	305	305	305	305	305	305	305	305	305	305	$3,655			
	actual	0	0	0	305	1,220	305	311	311	311					$2,763	933	(41)
BENEFITS 29% of Full Time Salaries	planned	88	88	88	88	88	88	1,127	1,857	88	88	88	88	$3,867			
	actual	0	0	0	88	354	88	1,859	975	90	0	0	0		$3,455	271	142
STUDENTS Salary	planned	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200	1,200	$14,400			
	actual	0	0	0	0	0	0	4,000	3,875	3,150					$11,025	12,150	(8,775)
Non Salary	planned	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	$12,000			
	actual	0	788	698	413	1,550	5,710	1,020	260	320					$10,758	0	1,243
SUPPLIES	planned	2,000	1,000	700	700	700	700	700	700	700	700	400	0	$9,000			
	actual	0	0	165	810	398	56	339	840	1,271					$3,878	2,007	3,115
TRAVEL	planned	0	0	0	0	0	0	1,500	0	0	0	0	0	$1,500			
	actual	0	0	0	0	0	0	0	2,383	0					$2,383	0	(883)
OTHER	planned	0	0	250	0	0	250	0	0	250	0	0	250	$1,000			
	actual	0	0	0	0	0	0	48	0	0					$48	0	952
EQUIPMENT * > $1,000	planned	0	5,000	0	50,000	0	0	0	0	0	0	0	0	$55,000			
	actual	0	0	0	953	0	0	0	0	50,000					$50,953	0	4,047
INDIRECT COSTS 43% of Direct Costs except *	planned	1,975	1,545	1,523	1,416	1,416	1,623	4,047	4,800	1,623	1,416	1,287	1,222	$23,694			
	actual	0	339	371	695	1,514	2,649	5,881	5,028	2,211	0	0	0		$18,688	6,605	(1,599)
PLANNED TOTALS: PER MONTH		6,568	10,138	5,066	64,709	4,709	6,066	13,458	15,961	5,066	4,709	4,280	4,065	$133,795			
ACTUAL TOTALS: PER MONTH		0	1,126	1,233	3,264	5,035	8,808	19,558	16,722	57,353	0	0	0		$113,100	21,965	(1,270)
CUMULATIVE																	
PLANNED TOTALS		6,568	16,706	21,772	76,481	81,189	86,256	99,714	115,675	120,741	125,450	129,730	133,795	$133,795			
ACTUAL TOTALS		0	1,126	2,360	5,623	10,659	19,467	39,025	55,747	113,100	113,100	113,100	113,100		$113,100		(1,270)
Budget Variance		$6,568	$15,579	$19,412	$70,858	$70,531	$66,789	$60,689	$59,928	$7,642	$12,350	$16,630	$20,695				

Note: Negative balance will be cleared up next quarter

Sub-Task 2.3: Centrifugal Membranes to Treat Haz./Radio Active Wastes

PRINCIPAL INVESTIGATOR: B. Reed



PROJECT: Environmental Pollution Control Devices Based on Novel Forms of Carbon

METC TASK NO.: 2.4

WVU ACCOUNT NO.: 8527-089-2405

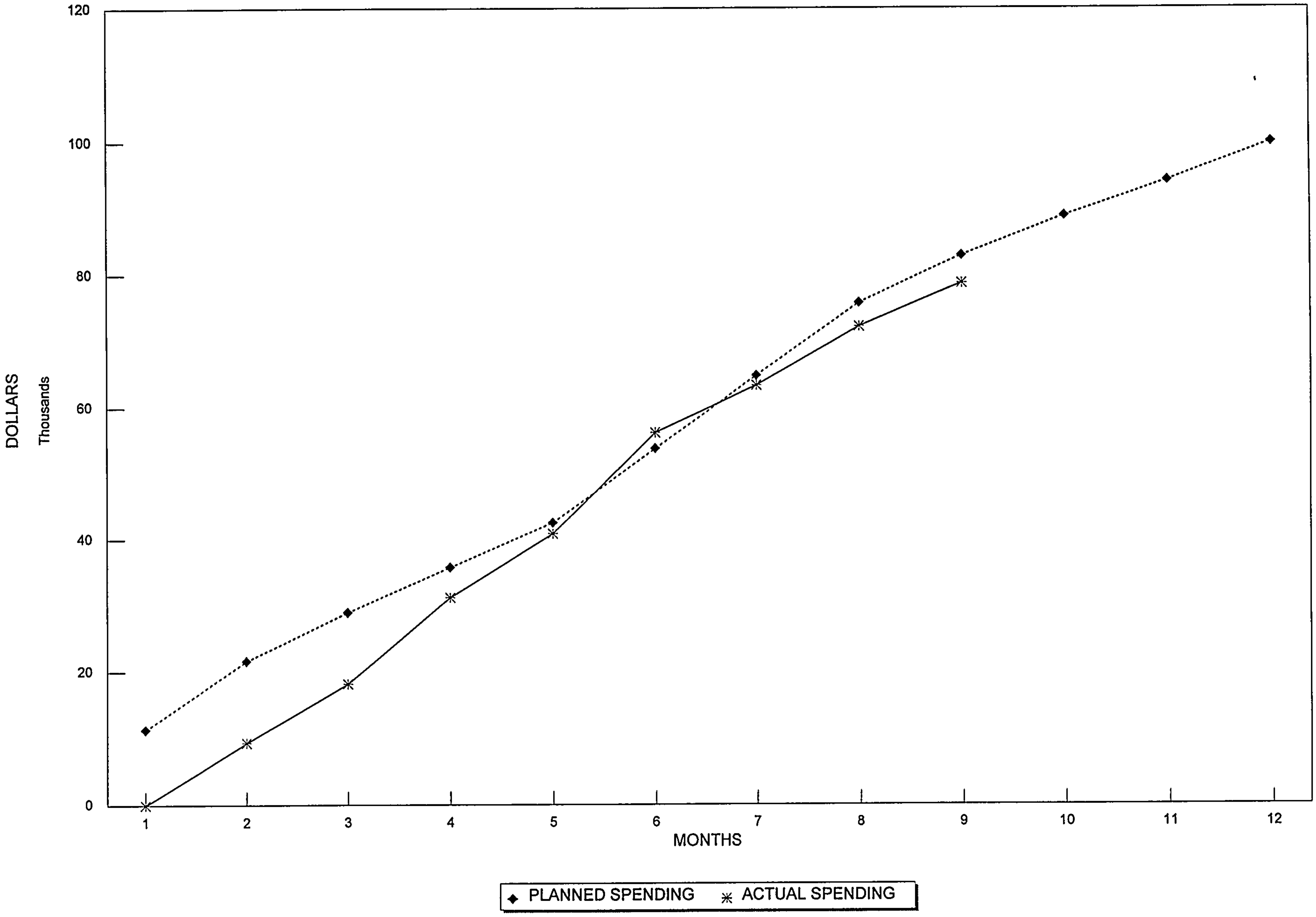
PRINCIPAL INVESTIGATOR (S): J. Zondlo, A. Stiller, A. Brennsteiner

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
A. Brennsteiner & P. Stansberry	planned	2,709	2,709	2,709	2,709	2,709	2,709	2,709	2,709	2,709	2,709	2,709	2,709	$32,510			
	actual	0	4,750	2,375	2,375	2,375	2,375	2,500	4,000	2,500					$23,250	7,500	1,760
J. Zondolo & A. Stiller	planned	0	0	0	0	0	2,267	2,267	2,267	0	0	0	0	$6,800			
	actual	0	0	0	0	1,725	5,075	0	0	0					$6,800	0	(0)
BENEFITS 29% of Full Time Salaries	planned	786	786	786	786	786	1,443	1,443	1,443	786	786	786	786	$11,400			
	actual	0	1,378	689	689	1,189	2,161	725	1,160	725	0	0	0		$8,715	2,175	510
GRADUATE ASSISTANT	planned	0	0	0	1,000	1,000	1,000	1,000	1,000	1,000	450	0	0	$6,450			
	actual	0	0	0	1,000	1,000	1,000	1,050	1,050	1,050					$6,150	3,150	(2,850)
SUPPLIES	planned	242	242	242	242	242	242	242	242	242	242	242	242	$2,900			
	actual	0	0	282	123	512	0	119	0	260					$1,296	203	1,400
TRAVEL	planned	0	0	1,100	0	0	0	0	0	0	0	0	0	$1,100			
	actual	0	0	0	1,076	0	0	337	0	0					$1,413	0	(313)
OTHER *off campus rent	planned	0	0	250	0	0	250	0	0	250	0	0	250	$1,000			
	actual	0	0	0	0	0	162	339	0	0					$502	0	498
EQUIPMENT * > $1,000	planned	6,000	5,000	0	0	0	0	0	0	0	0	0	0	$11,000			
	actual	0	632	4,019	5,411	0	0	0	0	0					$10,062	0	938
INDIRECT COSTS 43% of Direct Costs except *	planned	1,607	1,607	2,187	2,037	2,037	3,402	3,294	3,294	2,144	1,800	1,607	1,714	$26,729			
	actual	0	2,635	1,439	2,263	2,924	4,563	2,035	2,670	1,950	0	0	0		$20,478	5,602	648
PLANNED TOTALS: PER MONTH		11,343	10,343	7,274	6,773	6,773	11,312	10,955	10,955	7,131	5,987	5,343	5,701	$99,889			
ACTUAL TOTALS: PER MONTH		0	9,395	8,804	12,936	9,725	15,335	7,106	8,880	6,485	0	0	0		$78,666	18,631	2,592
CUMULATIVE																	
PLANNED TOTALS		11,343	21,686	28,960	35,733	42,507	53,819	64,773	75,728	82,859	88,845	94,189	99,889	$99,889			
ACTUAL TOTALS		0	9,395	18,199	31,135	40,860	56,196	63,301	72,181	78,666	78,666	78,666	78,666		$78,666		2,592
Budget Variance		$11,343	$12,292	$10,762	$4,598	$1,647	($2,377)	$1,472	$3,546	$4,192	$10,179	$15,522	$21,223				

Sub-Task 2.4 - Pollution Control Devices Based on Novel Forms of Carbon

PRINCIPAL INVESTIGATOR (S): A. Stiller, J. Zondolo & A. Brennsteiner



PROJE(Development of Instrumental Methods for Characterization a
Analysis of Nuclear Wastes & Environmental Contaminants

METC TASK NO.: 2.5
WVU ACCOUNT NO.: 8527-047-0702

PRINCIPAL INVESTIGATOR (S): V. Remcho - Dept. of Chemistry, WVU
N. Ballou - Battelle-Pacific Northwest National Laboratories

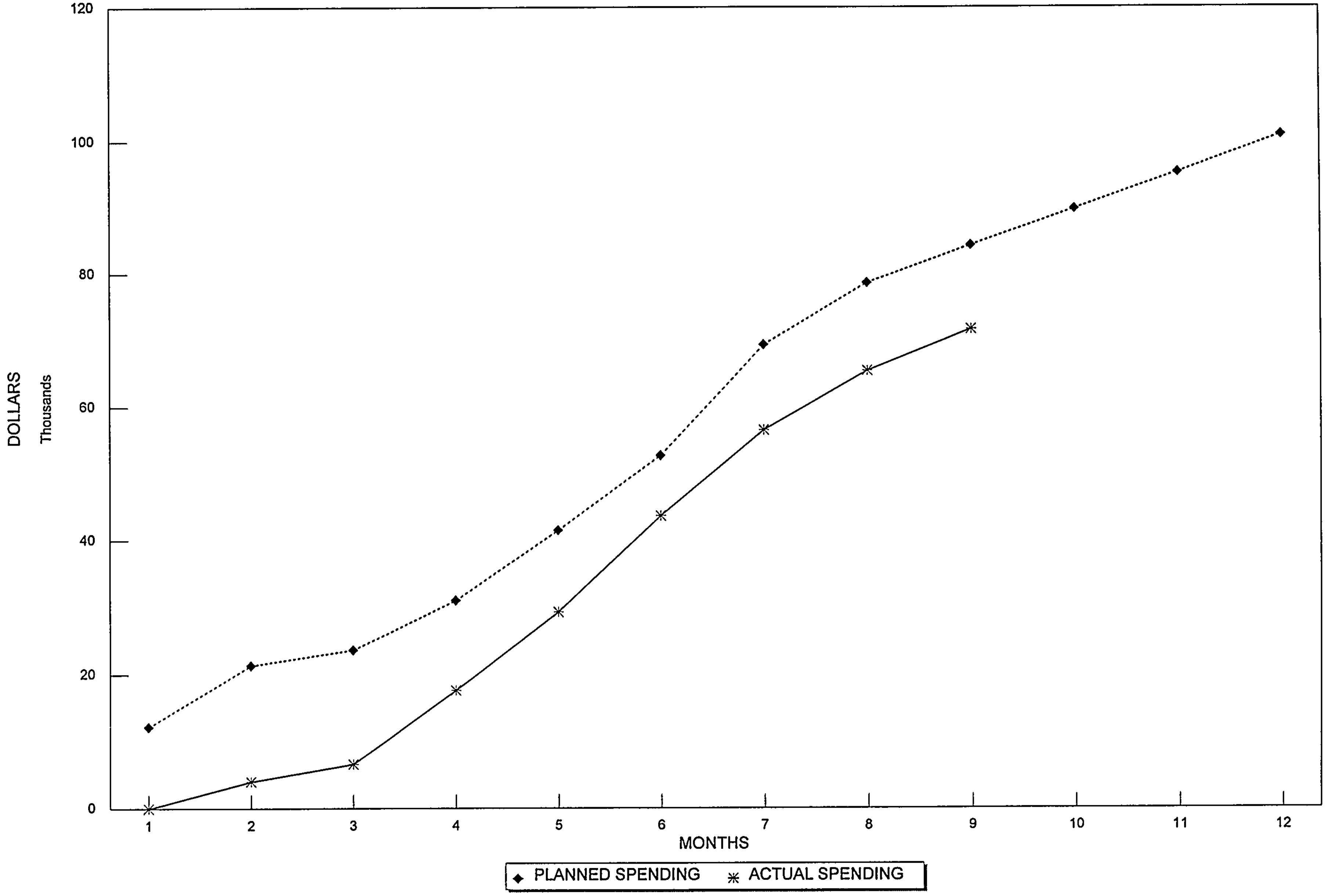
PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
V. Remcho	planned	0	0	0	0	2,067	4,134	4,134	2,067	0	0	0	0	$12,402			
	actual	0	0	0	0	2067	4134	4134	2067	0					$12,402	0	0
Post Doc H. Knobel	planned	1,000	1,000	1,000	1,000	1,000	1,000	1,000	0	0	0	0	0	$7,000			
	actual	0	2000	1000	1000	1000	1000	1000	0	0					$7,000	0	0
BENEFITS 29% of Full Time Salaries	planned	290	290	290	290	889	1,489	1,489	599	0	0	0	0	$5,627			
	actual	0	580	290	290	889	1,489	1,489	599	0	0	0	0		$5,627	0	0
STUDENTS Graduate	planned	0	0	0	0	500	1,000	1,000	1,000	1,000	1,000	1,000	1,000	$7,500			
	actual	0	0	0	0	500	1,000	1,000	1,000	1,000					$4,500	3,000	0
SUPPLIES	planned	230	230	230	230	230	230	230	230	230	230	230	230	$2,760			
	actual	0	231	535	1,133	109	27	1,085	55	1,626					$4,800	0	(2,040)
TRAVEL	planned	0	0	0	1,000	0	0	1,200	0	0	0	0	0	$2,200			
	actual	0	0	0	0	1,232	941	0	766	0					$2,939	0	(739)
OTHER	planned	0	0	125	0	0	125	0	0	125	0	0	125	$500			
	actual	0	0	0	289	271	0	277	161	301					$1,300	0	(800)
EQUIPMENT * > $1,000	planned	10,000	7,000	0	0	0	(3,990)	0	0	0	0	0	0	$13,010			
	actual	0	0	0	7,118	3,000	0	0	0	0					$10,118	0	2,892
SUB-CONTR. < $25,000	planned	0	0	0	2,611	2,611	2,611	2,611	2,611	2,611	2,611	2,611	2,611	$23,500			
	actual	0	0	0	0	0	1,425	0	1,583	1,422					$4,430	19,070	0
INDIRECT COSTS 43% of Direct Costs except *	planned	654	654	707	2,206	3,138	4,553	5,016	2,798	1,705	1,652	1,652	1,705	$26,440			
	actual	0	1,209	785	1,166	2,609	4,307	3,864	2,679	1,870	0	0	0		$18,489	9,490	(1,539)
PLANNED TOTALS: PER MONTH		12,174	9,174	2,352	7,337	10,435	11,153	16,679	9,306	5,672	5,493	5,493	5,672	$100,939			
ACTUAL TOTALS: PER MONTH		0	4,019	2,609	10,996	11,678	14,323	12,850	8,910	6,219	0	0	0		$71,604	31,560	(2,226)
CUMULATIVE																	
PLANNED TOTALS		12,174	21,347	23,700	31,037	41,473	52,625	69,305	78,610	84,282	89,775	95,267	100,939	$100,939			
ACTUAL TOTALS		0	4,019	6,628	17,625	29,303	43,626	56,475	65,386	71,604	71,604	71,604	71,604		$71,604		(2,226)
Budget Variance		$12,174	$17,328	$17,071	$13,412	$12,170	$9,000	$12,829	$13,225	$12,678	$18,170	$23,663	$29,335				

NOTE 1: All Sub-Contractor Expenses are time @ a loaded rate of $117.50/hour. No detail follows.
NOTE 2: Negative balance will be cleared up next quarter

Sub-Task 2.5: Instrumental Methods for Characterization & Analysis

PRINCIPAL INVESTIGATOR (S): V. Remcho & N. Ballou



PROJECT: Production and Evaluation of Biosorbents and Cleaning Solutions for use in Decontamination and Decommissioning

METC TASK NO.: 2.6A

** Sub-Contractor Detail & Summary **

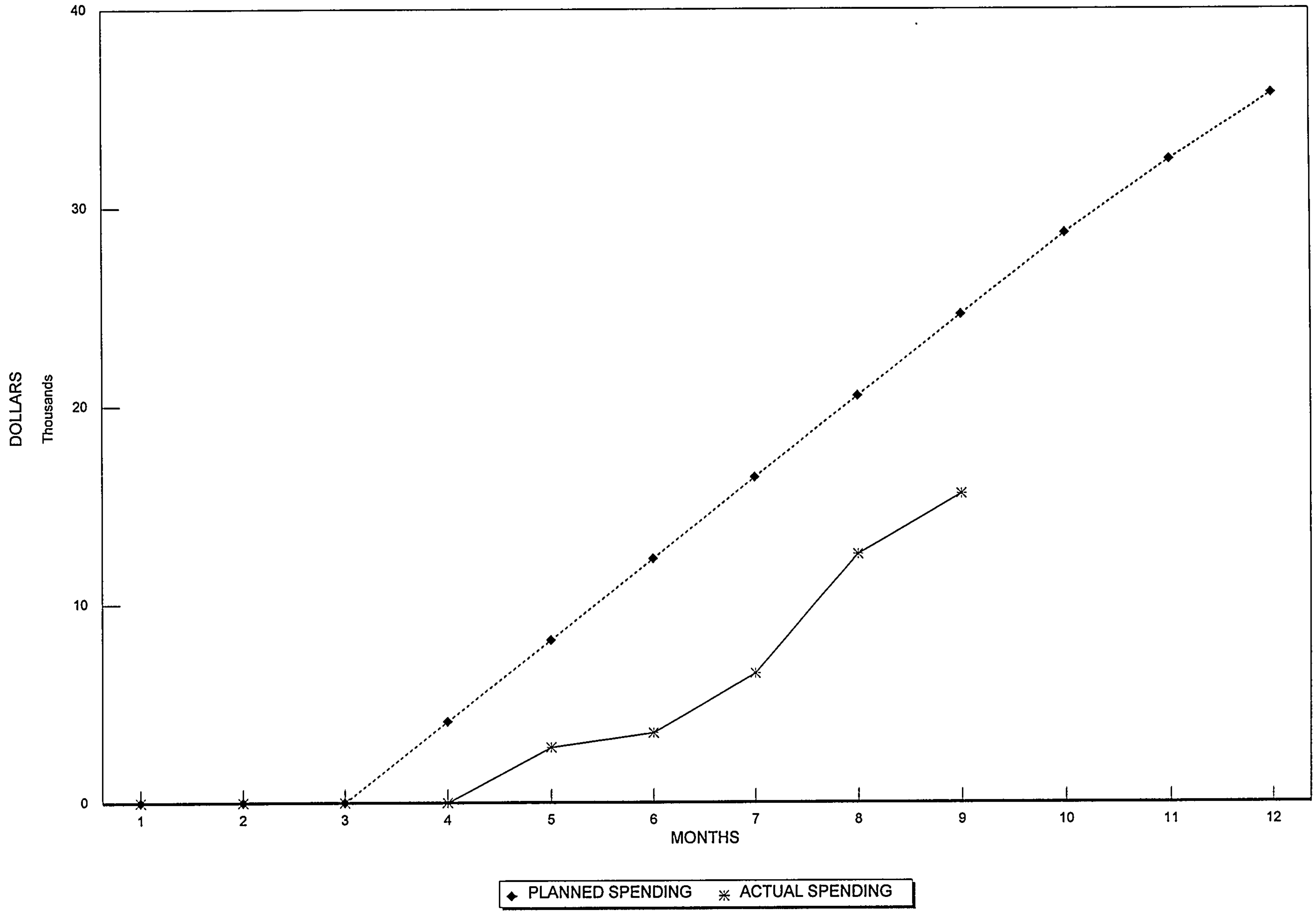
WVU ACCOUNT NO.: 8527-090-3254

PRINCIPAL INVESTIGATOR: J. Kilbane
Institute of Gas Technology

PERIOD ENDING: 9/30/96

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
Direct Labor	planned	0	0	0	1,049	1,049	1,049	1,049	1,049	1,049	1,049	1,049	1,049	$9,445			
	actual	0	0	0	0	777	206	837	1,685	852					$4,357	5,088	0
Overhead on Direct Labor	planned	0	0	0	1,448	1,448	1,448	1,448	1,448	1,448	1,448	1,448	1,448	$13,035			
	actual	0	0	0	0	1,073	284	1,155	2,325	1,176	0	0	0		$6,014	7,021	0
Materals & Services	planned	0	0	0	234	234	234	234	234	234	234	234	234	$2,110			
	actual	0	0	0	0	0	0	0	0	0					$0	2,110	0
TOTAL DIRECT Sub Contract	planned	0	0	0	2,732	2,732	2,732	2,732	2,732	2,732	2,732	2,732	2,732	$24,590			
	actual	0	0	0	0	1,850	490	1,993	4,010	2,028	0	0	0		$10,371	14,219	(0)
General and Administrative	planned	0	0	0	601	601	601	601	601	601	601	601	601	$5,410			
	actual	0	0	0	0	407	108	438	882	446	0	0	0		$2,282	3,128	(0)
TOTAL Sub Contract	planned	0	0	0	3,333	3,333	3,333	3,333	3,333	3,333	3,333	3,333	3,333	$30,000			
	actual	0	0	0	0	2,257	598	2,431	4,893	2,474	0	0	0		$12,653	17,347	(0)
INDIRECT COSTS 23% of Sub Cont < $25,000	planned	0	0	0	767	767	767	767	767	767	767	384	0	$5,750			
	actual	0	0	0	0	519	138	559	1,125	569	0	0	0		$2,910	2,840	0
PLANNED TOTALS: PER MONTH		0	0	0	4,100	4,100	4,100	4,100	4,100	4,100	4,100	3,717	3,333	$35,750			
ACTUAL TOTALS: PER MONTH		0	0	0	0	2,776	736	2,990	6,018	3,043	0	0	0		$15,563	20,187	(0)
CUMULATIVE																	
PLANNED TOTALS		0	0	0	4,100	8,200	12,300	16,400	20,500	24,600	28,700	32,417	35,750	$35,750			
ACTUAL TOTALS		0	0	0	0	2,776	3,511	6,502	12,520	15,563	15,563	15,563	15,563		$15,563		0
Budget Variance		$0	$0	$0	$4,100	$5,424	$8,788	$9,898	$7,980	$9,037	$13,137	$16,854	$20,187				

Sub-Task 2.6a: Production & Evaluation of Biosorbents and Cleaning Solutions for use in D&D - PRINCIPAL INVESTIGATOR: J. Kilbane, Institute of Gas Technology



PROJECT: Use of Spintek Centrifugal Membrane Technology and Sorbents/Cleaning Solutions in the Decontamination and Decommissioning of DOE Facilities

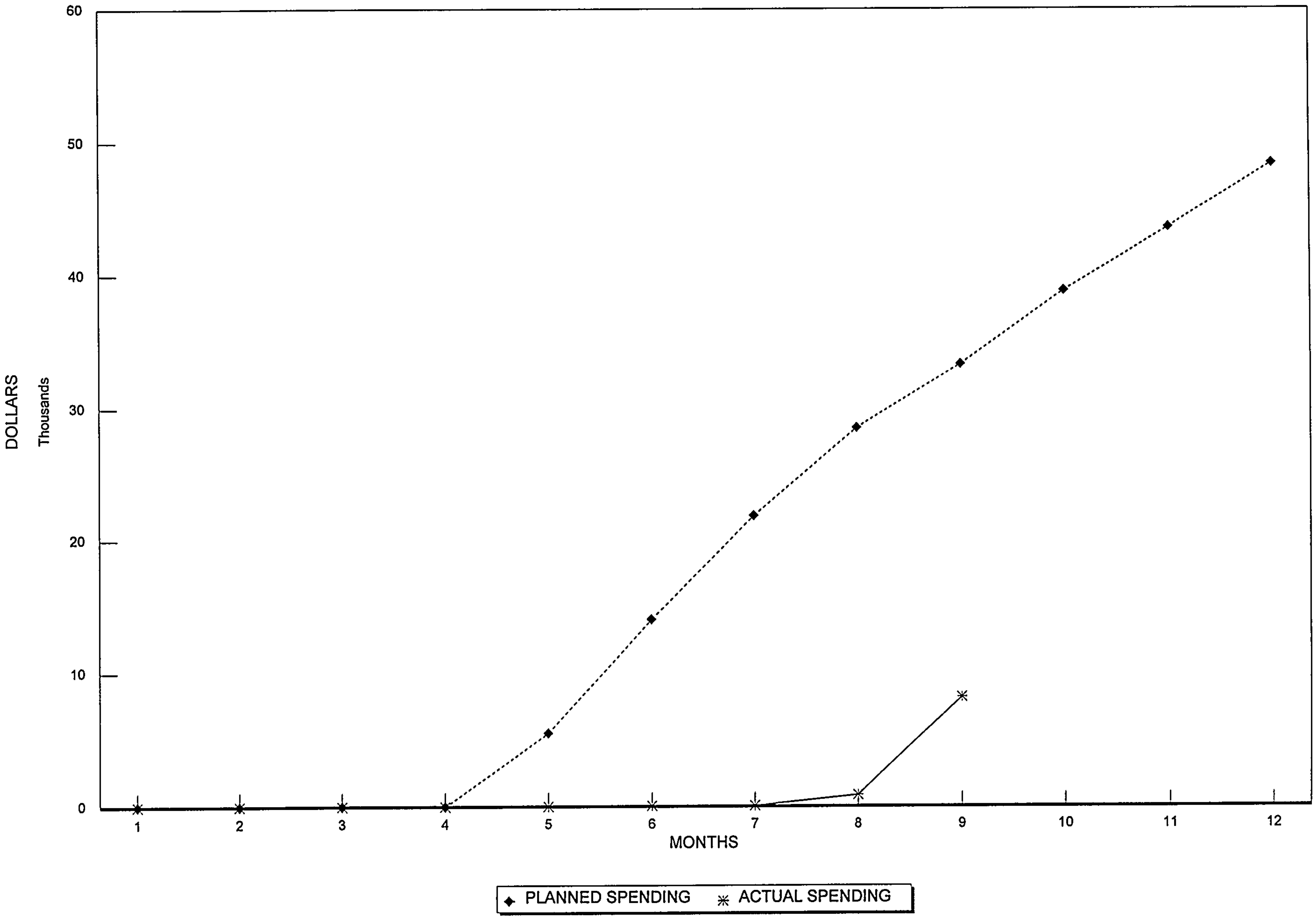
METC TASK NO.: 2.6B
WVU ACCOUNT NO.: 8527-090-3253

PRINCIPAL INVESTIGATOR (S): B. Reed and W. Lin

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
B. Reed	planned	0	0	0	0	1,017	2,034	2,034	1,017	0	0	0	0	$6,101	$0	0	6,101
	actual					0	0	0	0	0							
W. Lin	planned	0	0	0	0	688	688	688	688	688	688	688	688	$5,501	$0	0	5,501
	actual					0	0	0	0	0							
BENEFITS 29% of Full Time Salaries	planned	0	0	0	0	494	789	789	494	199	199	199	199	$3,364	$0	0	3,364
	actual	0	0	0	0	0	0	0	0	0	0	0	0				
STUDENTS Salary	planned	0	0	0	0	0	1,709	1,709	1,709	1,709	1,709	1,709	1,709	$11,960	$4,765	10,805	(3,610)
	actual					0	0	0	425	4,340							
Non-Salary	planned	0	0	0	0	0	0	0	0	500	500	500	500	$2,000	$0	0	2,000
	actual					0	0	0	0	0							
SUPPLIES	planned	0	0	0	0	225	225	225	225	225	225	225	225	$1,800	$615	1,867	(682)
	actual					0	33	0	130	451							
TRAVEL	planned	0	0	0	0	0	500	0	500	0	500	0	0	$1,500	$337	0	1,163
	actual					0	0	0	0	337							
OTHER	planned	0	0	0	0	25	25	25	25	25	25	25	25	$200	$0	0	200
	actual					0	0	0	0	0							
EQUIPMENT * > $1,000	planned	0	0	0	0	2,000	0	0	0	0	0	0	0	$2,000	$0	0	2,000
	actual					0	0	0	0	0							
INDIRECT COSTS 43% of Direct Costs except *	planned	0	0	0	0	1,053	2,567	2,352	2,003	1,439	1,654	1,439	1,439	$13,943	$2,458	5,449	6,036
	actual	0	0	0	0	0	14	0	239	2,205	0	0	0				
PLANNED TOTALS: PER MONTH		0	0	0	0	5,502	8,536	7,821	6,660	4,784	5,499	4,784	4,784	$48,369			
ACTUAL TOTALS: PER MONTH		0	0	0	0	0	48	0	794	7,334	0	0	0		$8,175	18,121	22,073
CUMULATIVE																	
PLANNED TOTALS		0	0	0	0	5,502	14,037	21,858	28,518	33,302	38,801	43,585	48,369	$48,369			
ACTUAL TOTALS		0	0	0	0	0	48	48	842	8,175	8,175	8,175	8,175		$8,175		22,073
Budget Variance		$0	$0	$0	$0	$5,502	$13,989	$21,810	$27,676	$25,126	$30,626	$35,410	$40,194				

Sub-Task 2.6B: Use of Spintek Centrifugal Membranes
and Sorbents/cleaning solutions in D&D work - PRINCIPAL INVESTIGATOR (S): B. Reed & W. Lin



TASK 3.0

Small Business Support Program

PROJECT: West Virginia High Tech Consortium (WVHTC) Foundation
Environmental Technology Support Program

METC TASK NO.: 3.1
SUMMARY

WVU ACCOUNT NO.: 8527-090-3248

PRINCIPAL INVESTIGATOR (S): E. Cook & J. Berkow

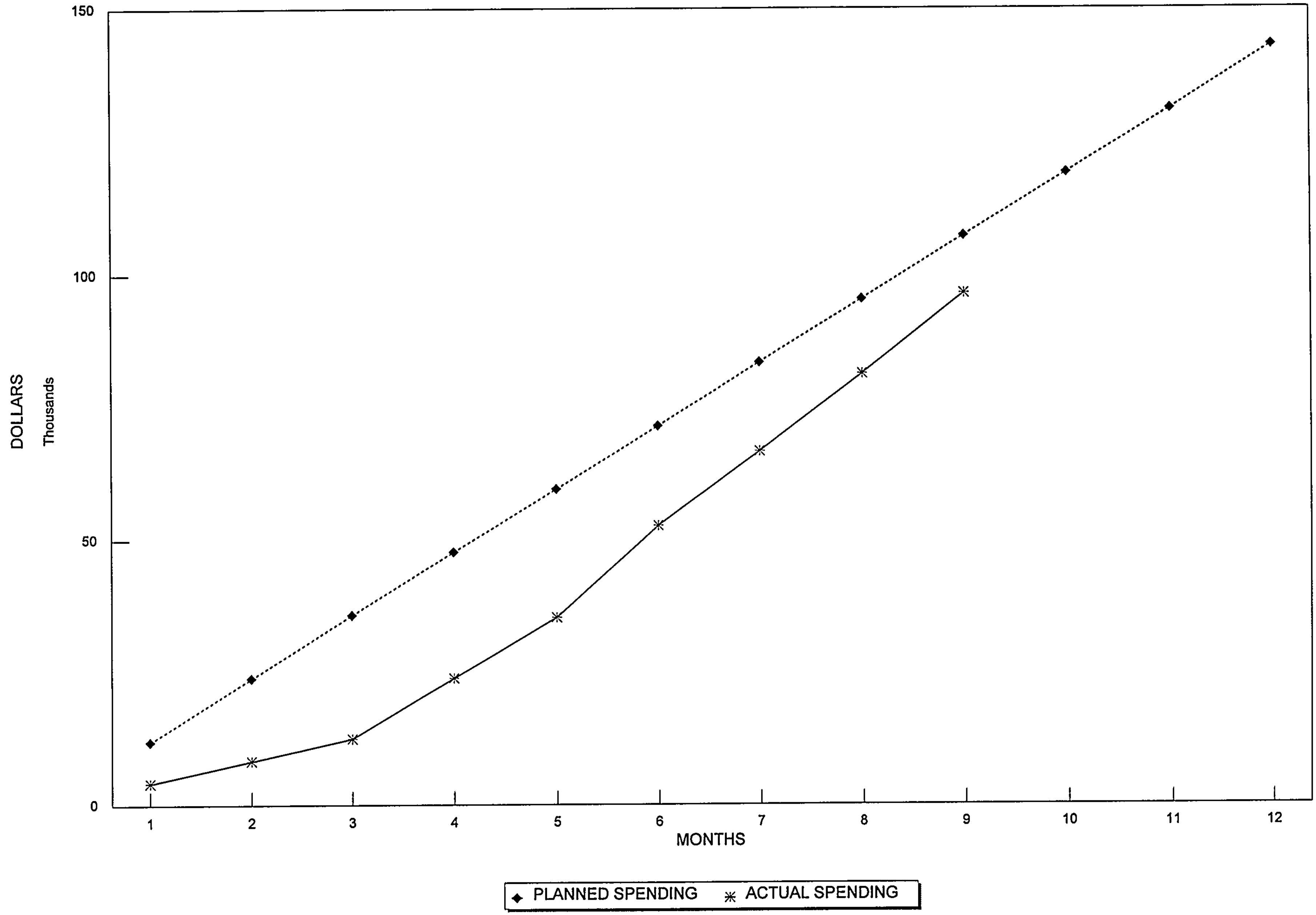
PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
E. Cook	planned	475	475	475	475	475	475	475	475	475	475	475	475	$5,700			
	actual	0	0	0	0	0	1,425	0	291	581					$2,297	1,743	1,661
T. Beatty	planned	609	609	609	609	609	609	609	609	609	609	609	609	$7,309			
	actual	0	0	0	0	0	1,827	934	934	934					$4,629	2,802	(122)
BENEFITS 29% of Full Time Salaries	planned	314	314	314	314	314	314	314	314	314	314	314	314	$3,773			
	actual	0	0	0	0	0	943	271	355	439	0	0	0		$2,008	1,318	446
SUB-CONTR. > $25,000	planned	9,917	9,917	9,917	9,917	9,917	9,917	9,917	9,917	9,917	9,917	9,917	9,917	$119,000			
	actual	4,145	4,145	4,145	11,407	11,407	11,407	12,315	12,315	12,315					$83,601	35,399	0
INDIRECT COSTS 43% of Direct Costs except *	planned	601	601	601	601	601	601	601	601	601	601	601	601	$7,216			
	actual	0	0	0	0	0	1,804	518	679	840	0	0	0		$3,842	2,521	854
PLANNED TOTALS: PER MONTH		11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	11,917	$142,998			
ACTUAL TOTALS: PER MONTH		4,145	4,145	4,145	11,407	11,407	17,406	14,038	14,574	15,109	0	0	0		$96,376	43,783	2,839
CUMULATIVE																	
PLANNED TOTALS		11,917	23,833	35,750	47,666	59,583	71,499	83,416	95,332	107,249	119,165	131,082	142,998	$142,998			
ACTUAL TOTALS		4,145	8,290	12,435	23,842	35,250	52,656	66,694	81,267	96,376	96,376	96,376	96,376		$96,376		2,839
Budget Variance		$7,772	$15,543	$23,315	$23,824	$24,333	$18,843	$16,722	$14,065	$10,872	$22,789	$34,705	$46,622				

*Sub Contractor Detail Follows

Sub-Task 3.1: WVHTC Foundation - Technology Support Program

PRINCIPAL INVESTIGATOR (S): E. Cook & J. Berkow <<<SUMMARY>>>



PROJECT: West Virginia High Tech Consortium (WVHTC) Foundation
Environmental Technology Support Program

TASK NO. 3.1
Sub-Contractor Detail

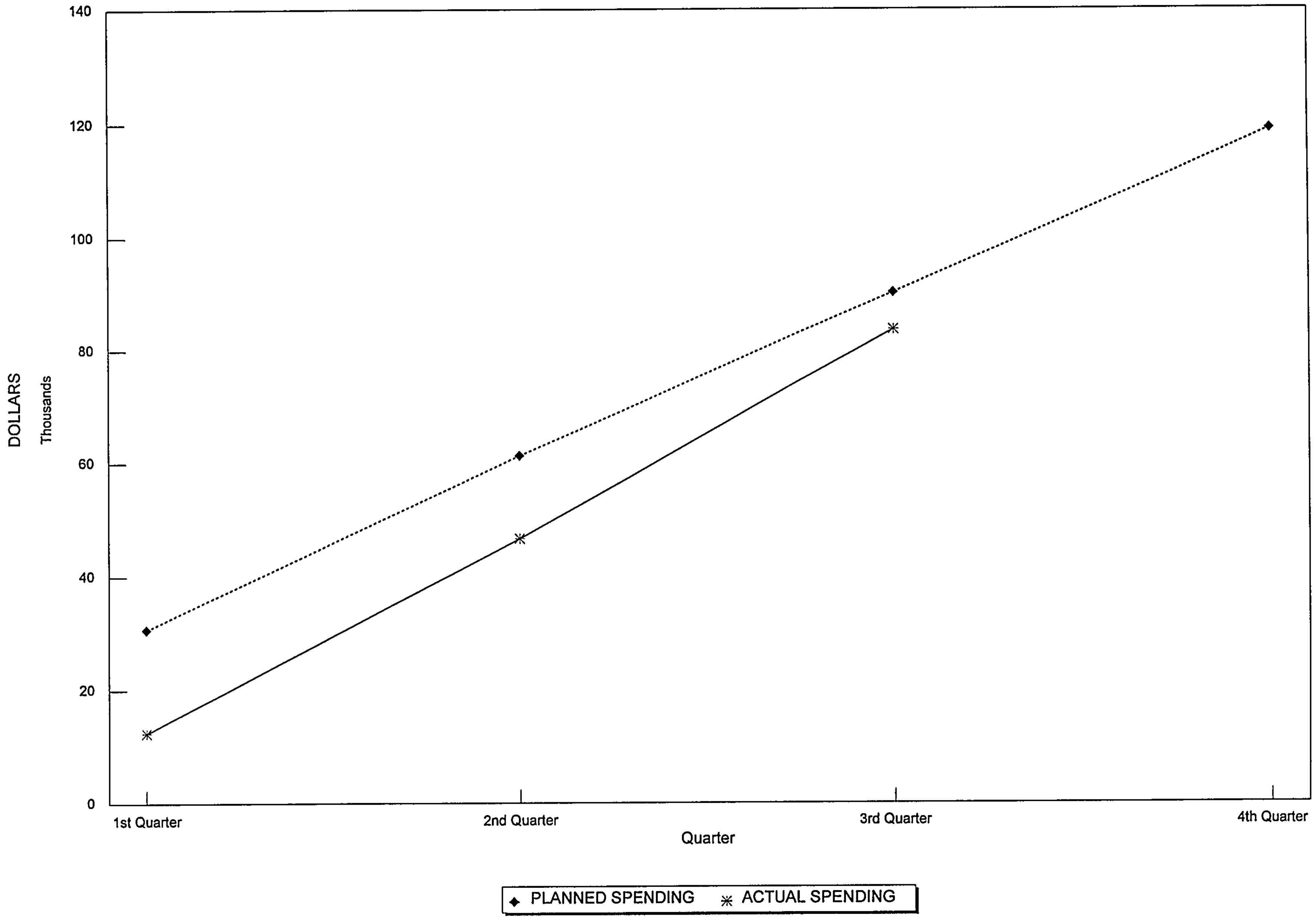
PRINCIPAL INVESTIGATOR: Jan Berkow

Period Ending: 9/30/96

DESCRIPTION	Planned Actual	1st Quarter Jan - Mar	2nd Quarter April - June	3rd Quarter July - Sept	4th Quarter Oct - Dec	PLANNED TOTALS	ACTUAL TOTALS	Amount Remaining
SALARIES								
Direct Labor	planned	13,025	13,025	13,025	13,025	$52,100		
	actual	5,204	12,123	10,749			$28,076	24,024
Overhead	planned	10,529	10,529	10,529	10,529	$42,118		
	actual	2,985	11,022	8,689			$22,696	19,422
Indirect Labor	planned	0	0	0	0	$0		
	actual	0	0	5,730			$5,730	(5,730)
Equipment	planned	1,699	1,699	0	0	$3,397		
	actual	343	2,754	50			$3,147	250
Promotional Activities Technical/Facilities Support	planned	2,033	2,033	2,033	2,033	$8,131		
	actual	3,190	2,954	6,281			$12,425	(4,294)
General Expense Supplies & Misc.	planned	574	574	574	574	$2,295		
	actual	10	1,010	104			$1,124	1,171
Travel	planned	703	703	703	703	$2,812		
	actual	27	1,768	2,754			$4,549	(1,737)
SUB-TOTAL	planned	28,562	28,562	26,864	26,864	$110,853		
	actual	11,759	31,631	34,357	0		$77,747	33,106
General & Administrative	planned	2,099	2,099	1,974	1,974	$8,148		
	actual	676	2,591	2,587	0		$5,854	2,294
PLANNED TOTALS: PER MONTH		30,662	30,662	28,838	28,838	$119,000		
ACTUAL TOTALS: PER MONTH		12,435	34,222	36,944	0		$83,601	35,399
CUMULATIVE								
PLANNED TOTALS		30,662	61,323	90,162	119,000	$119,000		
ACTUAL TOTALS		12,435	46,657	83,601	83,601		$83,601	35,399
Budget Variance		$18,227	$14,666	$6,561	$35,399			

Sub-Task 3.1: WVHTC Foundation, Support Program

PRINCIPAL INVESTIGATOR: Jan Berkow <<<SUB-CONTRACTOR DETAIL>>>



PROJECT: Small Business Opportunities

PRINCIPAL INVESTIGATOR: E. Cook

METC TASK NO.: 3.2

WVU ACCOUNT NO.: 8527-090-3234

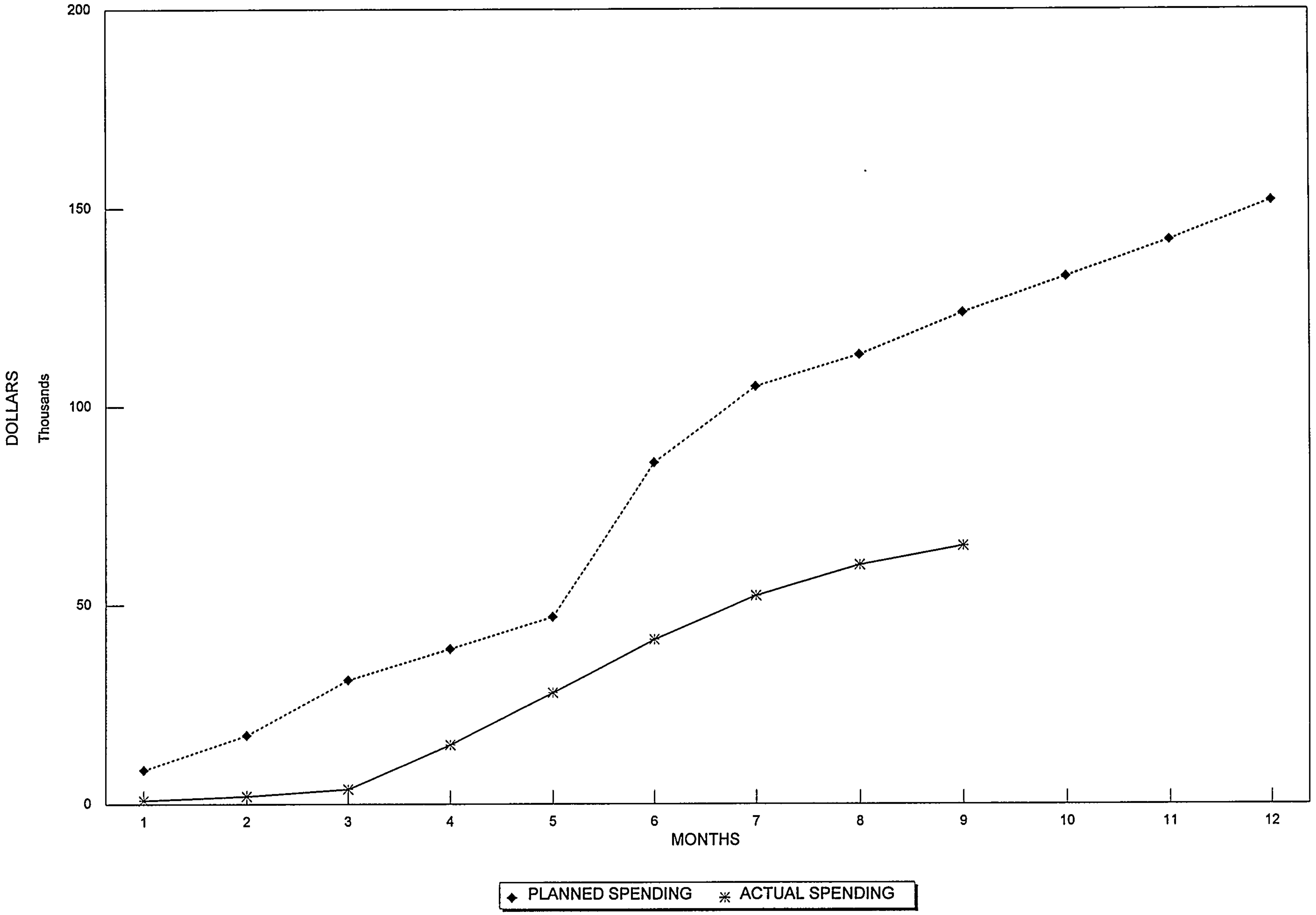
PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
Director	planned	2,375	2,375	2,375	2,375	2,375	2,375	2,375	2,375	2,375	2,375	2,375	2,375	$28,501			
	actual	0	0	0	4,908	4,908	4,908	4,750	3,198	1,646					$24,319	4,938	(756)
Clerical	planned	750	750	750	750	750	750	750	750	750	750	750	750	$8,996			
	actual	476	476	476	1,076	1,076	1,076	276	276	276					$5,484	828	2,684
BENEFITS																	
29% of Full Time Salaries	planned	906	906	906	906	906	906	906	906	906	906	906	906	$10,874			
	actual	138	138	138	1,735	1,735	1,735	1,458	1,007	557	0	0	0		$8,643	1,672	559
GRADUATE ASSISTANT	planned	0	0	1,560	0	0	1,560	0	0	1,560	905	905	1,560	$8,050			
	actual	0	0	0	0	425	850	850	850	850					$3,825	2,550	1,675
SUPPLIES	planned	1,000	1,000	1,000	500	500	500	500	500	500	500	500	0	$7,000			
	actual	51	108	656	0	655	210	0	31	74					$1,784	1,576	3,640
TRAVEL	planned	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	1,000	$12,000			
	actual	0	0	0	0	0	0	301	77	0					$379	941	10,680
OTHER	planned	0	0	375	0	0	375	0	0	375	0	0	375	$1,500			
	actual	0	0	0	0	361	299	33	0	0					$693	0	807
*SUB-CONTR. < $25,000	planned	0	0	0	0	0	18,007	0	0	0	0	0	0	$18,007			
	actual	0	0	0	0	0	0	0	0	0					$0	0	18,007
* > $25,000	planned	0	0	0	0	0	0	11,135	0	0	0	0	0	$11,135			
	actual	0	0	0	0	0	0	0	0	0					$0	0	11,135
EQUIPMENT > $1,000	planned	0	0	2,500	0	0	2,500	0	0	0	0	0	0	$5,000			
	actual	0	0	0	0	0	402	0	0	0					$402	858	3,740
INDIRECT COSTS																	
43% of Direct Costs except *	planned	2,593	2,593	3,425	2,378	2,378	10,953	2,378	2,378	3,210	2,767	2,767	2,995	$40,819			
	actual	286	310	546	3,320	3,939	3,904	3,297	2,339	1,463	0	0	0		$19,405	5,377	16,037
PLANNED TOTALS: PER MONTH		8,624	8,624	13,891	7,909	7,909	38,926	19,044	7,909	10,676	9,203	9,203	9,961	$151,882			
ACTUAL TOTALS: PER MONTH		950	1,033	1,816	11,039	13,100	13,385	10,965	7,778	4,867	0	0	0		$64,933	18,741	68,208
CUMULATIVE																	
PLANNED TOTALS		8,624	17,249	31,140	39,049	46,958	85,885	104,929	112,838	123,514	132,718	141,921	151,882	$151,882			
ACTUAL TOTALS		950	1,983	3,799	14,838	27,938	41,323	52,288	60,066	64,933	64,933	64,933	64,933		$64,933		68,208
Budget Variance		$7,674	$15,266	$27,341	$24,211	$19,020	$44,562	$52,641	$52,772	$58,581	$67,785	$76,988	$86,949				

*Note: Budget reduced this quarter in support of Task 3.3

Sub-Task 3.2: Small Business Opportunities

PRINCIPAL INVESTIGATOR: E. Cook



PROJECT: Approach for Assessing Potential Voluntary Environmental Protection Kanawha River Area *** Continuation from 1995***

METC TASK NO.: Old 3.4

WVU ACCOUNT NO.: 8527-224-1422

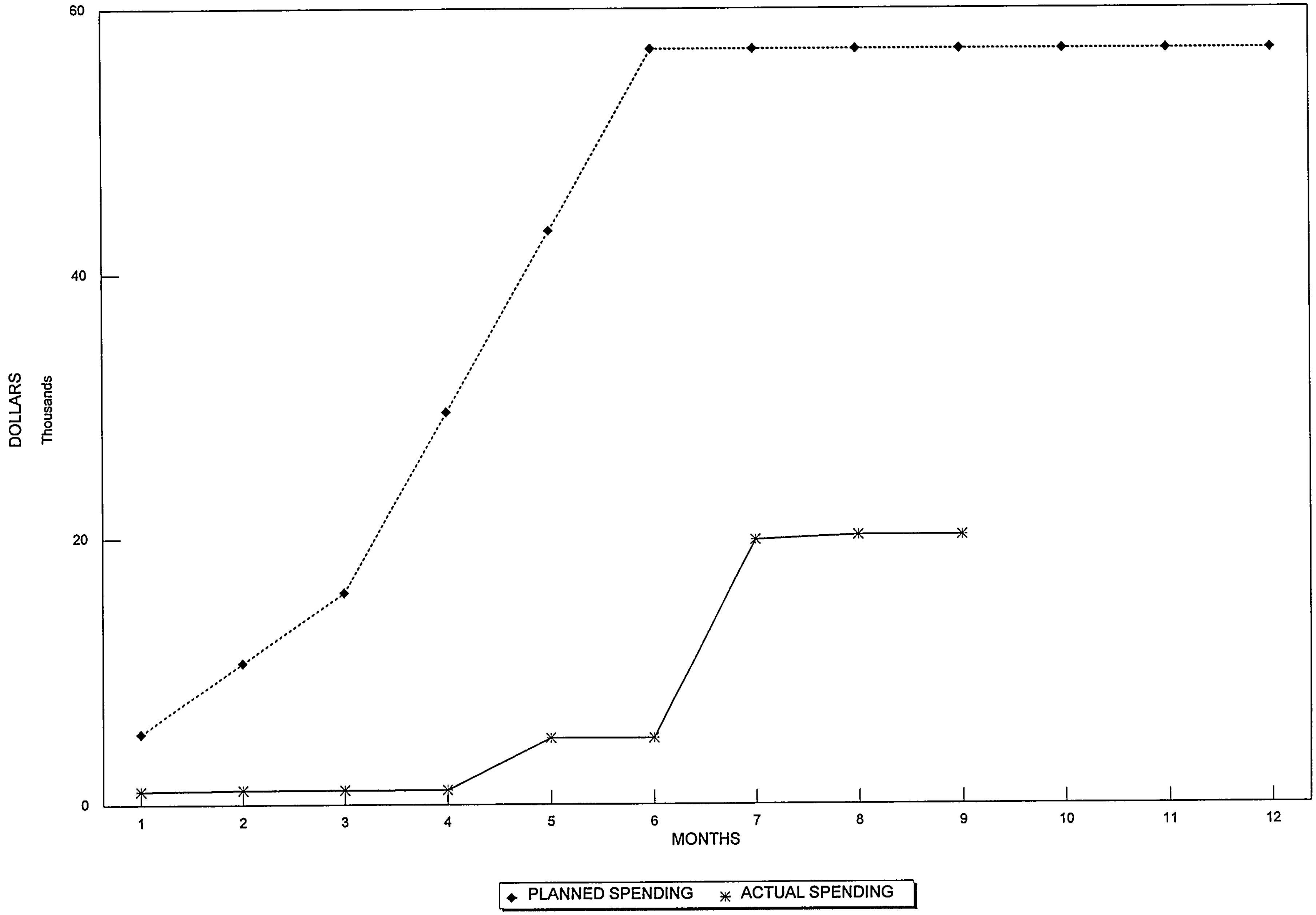
PRINCIPAL INVESTIGATOR: Ray Lovett

PERIOD ENDING: September 30, 1996

DESCRIPTION	Planned Actual	1 JAN	2 FEB	3 MARCH	4 APRIL	5 MAY	6 JUNE	7 JULY	8 AUG	9 SEPT	10 OCT	11 NOV	12 DEC	PLANNED TOTALS	ACTUAL TOTALS	Obligated Encumbrances	Amount Remaining
SALARIES																	
R. Lovett	planned	0	0	0	1,047	1,047	1,047	0	0	0	0	0	0	$3,142			
	actual	0	0	0	0	0	0	0	0						$0	0	3,142
BENEFITS 29% of Full Time Salaries	planned	0	0	0	304	304	304	0	0	0	0	0	0	$911			
	actual	0	0	0	0	0	0	0	0	0	0	0	0		$0	0	911
SUPPLIES	planned	28	28	28	28	28	28	0	0	0	0	0	0	$165			
	actual	0	0	0	0	0	0	67	0						$67	0	98
TRAVEL	planned	667	667	667	667	667	667	0	0	0	0	0	0	$4,000			
	actual	0	71	0	0	0	0	0	89						$160	0	3,840
OTHER	planned	0	0	0	0	0	0	0	0	0	0	0	0	$0			
	actual	0	0	0	0	0	0	0	0						$0	0	0
SUB-CONTR. < $25,000	planned	3,766	3,766	3,766	3,766	3,766	3,766	0	0	0	0	0	0	$22,595			
	actual	846	0	0	0	3,250	0	7,612	0						$11,708	10,887	0
SUB-CONTR. > $25,000	planned	0	0	0	6,760	6,760	6,760	0	0	0	0	0	0	$20,280			
	actual	0	0	0	0	0	0	5,780	230						$6,010	14,270	0
INDIRECT COSTS 19% of Direct Costs except *	planned	847	847	847	1,104	1,104	1,104	0	0	0	0	0	0	$5,855			
	actual	161	13	0	0	618	0	1,459	17	0	0	0	0		$2,268	2,068	1,518
PLANNED TOTALS: PER MONTH		5,307	5,307	5,307	13,675	13,675	13,675	0	0	0	0	0	0	$56,948			
ACTUAL TOTALS: PER MONTH		1,007	84	0	0	3,868	0	14,918	336	0	0	0	0		$20,213	27,225	9,510
CUMULATIVE																	
PLANNED TOTALS		5,307	10,615	15,922	29,597	43,273	56,948	56,948	56,948	56,948	56,948	56,948	56,948	$56,948			
ACTUAL TOTALS		1,007	1,091	1,091	1,091	4,959	4,959	19,877	20,213	20,213	20,213	20,213	20,213		$20,213		9,510
Budget Variance		$4,301	$9,524	$14,831	$28,506	$38,314	$51,989	$37,071	$36,735	$36,735	$36,735	$36,735	$36,735				

Sub-Task *OLD 3.4* - Kanawha River Area Environmental Protection

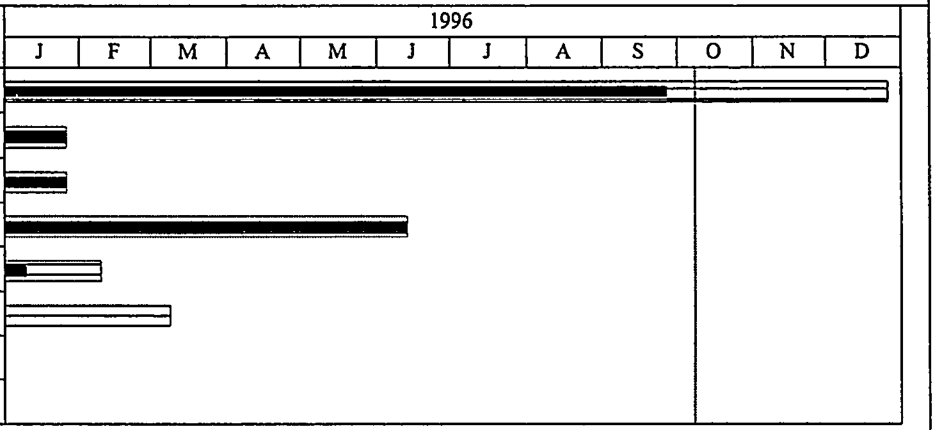
PRINCIPAL INVESTIGATOR: Ray Lovett - Note: Continuation from 1995, only 1996 work shown



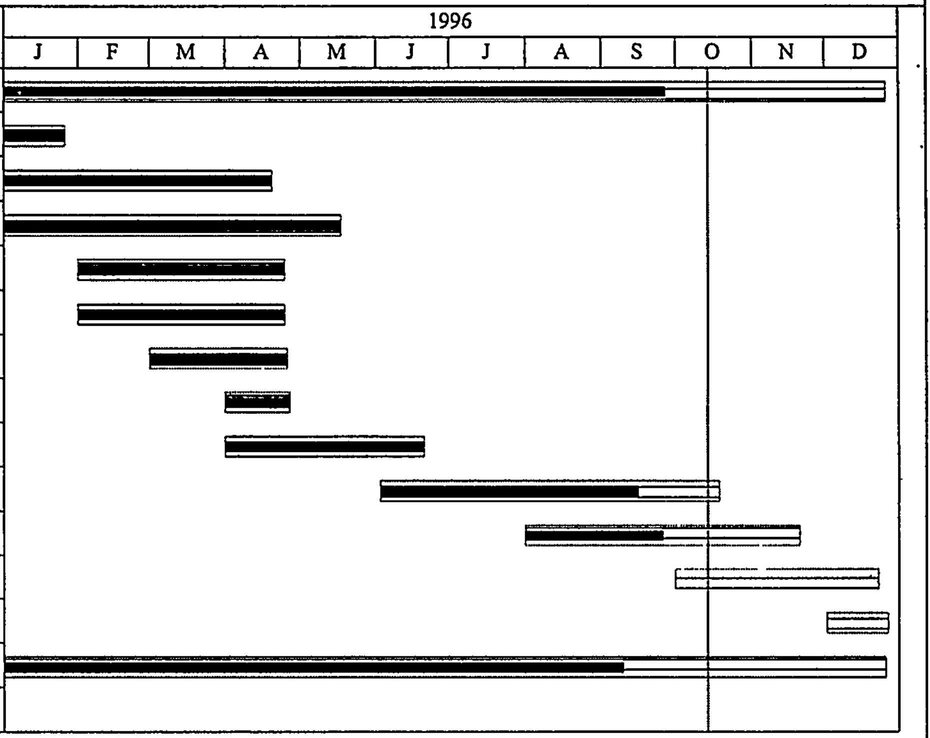
TASK 1.0

Contaminant Plume Containment & Remediation

ID	Task Name	Dur	Start	Finish	% Comp
1	**Task 1-1 Refinement of Combustion & Melting System**	52w	1/1/96	12/26/96	75%
2	NEPA Information	4w	1/1/96	1/26/96	100%
3	Conceptual Design Criteria	4w	1/1/96	1/26/96	100%
4	Recuperator Base Analysis	24w	1/1/96	6/13/96	100%
5	Recuperator Analysis	6w	1/1/96	2/9/96	25%
6	Analysis of Seperator Reservior	10w	1/1/96	3/8/96	0%
7	Final Report and Assessment	4w	12/1/97	12/26/97	0%
8					

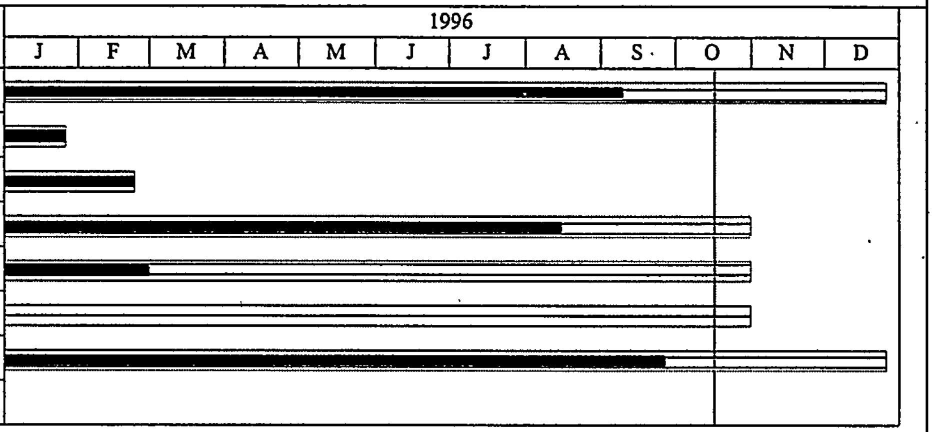


ID	Task Name	Dur	Start	Finish	% Comp
9	**Task 1-2 Drain Enhanced Soil Flushing**	52w	1/1/96	12/26/96	75%
10	NEPA Information	4w	1/1/96	1/26/96	100%
11	Pilot Scale Experiment Design	16w	1/1/96	4/19/96	100%
12	Field Site Selection	20w	1/1/96	5/17/96	100%
13	Design & Drafting	12w	2/1/96	4/24/96	100%
14	Permitting	12w	2/1/96	4/24/96	100%
15	Procure Equip & Supplies	8w	3/1/96	4/25/96	100%
16	Field Mobilization & Layout	4w	4/1/96	4/26/96	100%
17	Field Construction	12w	4/1/96	6/20/96	100%
18	Testing Phase	20w	6/3/96	10/18/96	75%
19	Lab Analysis	16w	8/1/96	11/20/96	50%
20	Demobilization	12w	10/1/96	12/23/96	0%
21	Site Closure/Restoration	4w	12/2/96	12/27/96	0%
22	Data Reduction/Report Development	52w	1/1/96	12/26/96	70%
23					

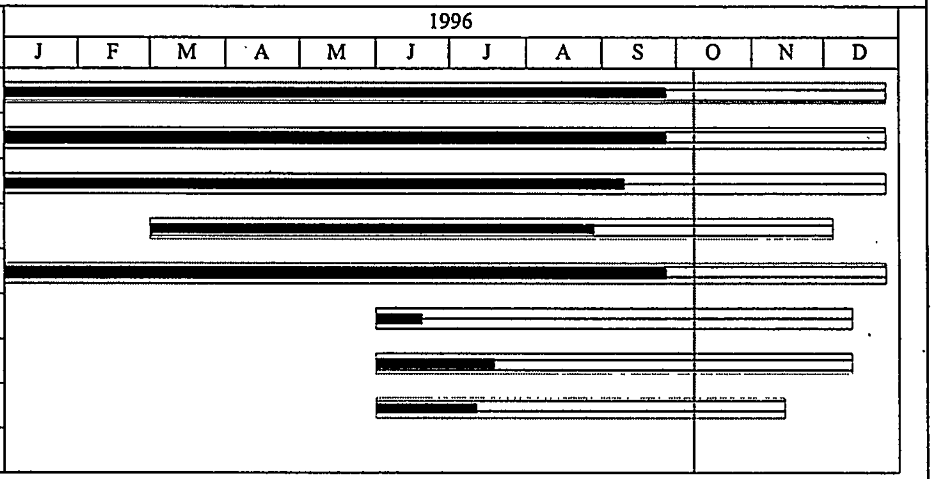


ID	Task Name	Dur	Start	Finish	% Comp
24	**Task 1-3 Evaluation of Acrylate Grout Barriers**	52w	1/1/96	12/26/96	70%
25	NEPA Information	4w	1/1/96	1/26/96	100%
26	Grout Development & Testing	46w	1/1/96	11/14/96	80%
27	Viscosity Tests	16w	1/1/96	4/19/96	100%
28	Setting Time Tests	16w	2/1/96	5/22/96	100%
29	Immersion Tests	24w	2/1/96	7/16/96	100%
30	Durability Tests	12w	6/1/96	8/22/96	100%
31	Permeability Tests	32w	3/1/96	10/9/96	80%
32	Shear Strength	20w	7/1/96	11/15/96	50%
33	Compressibility Characteristics	20w	8/1/96	12/18/96	25%
34	Grout Performance Testing	36w	4/1/96	12/5/96	60%
35	Data Reduction/Report Development	52w	1/1/96	12/26/96	70%
36					

ID	Task Name	Dur	Start	Finish	% Comp
37	**Task 1-4 Desication Barriers**	52w	1/1/96	12/26/96	70%
38	NEPA Information	4w	1/1/96	1/26/96	100%
39	Testing Protocol Development	8w	1/1/96	2/23/96	100%
40	Sand Pack Characterization	44w	1/1/96	10/31/96	75%
41	Adiabatic Cooling	44w	1/1/96	10/31/96	20%
42	Liquid Storage and Migration	44w	1/1/96	10/31/96	0%
43	Data Reduction/Report Development	52w	1/1/96	12/26/96	75%
44					



ID	Task Name	Dur	Start	Finish	% Comp
45	**Task 1-5 Technical Support for Barrier Program**	52w	1/1/96	12/26/96	75%
46	Planning & Technology Transfer	52w	1/1/96	12/26/96	75%
47	Circulating Air Barrier Bench Scale	52w	1/1/96	12/26/96	70%
48	Circulating Air Barrier Large Scale	40w	3/1/96	12/4/96	65%
49	Grout Barrier Bench Scale	52w	1/1/96	12/26/96	75%
50	Grout Barrier Large Scale	28w	6/1/96	12/12/96	10%
51	Freeze Barrier Bench Scale	28w	6/1/96	12/12/96	25%
52	Dual Barrier System Bench Scale	24w	6/1/96	11/14/96	25%
53					



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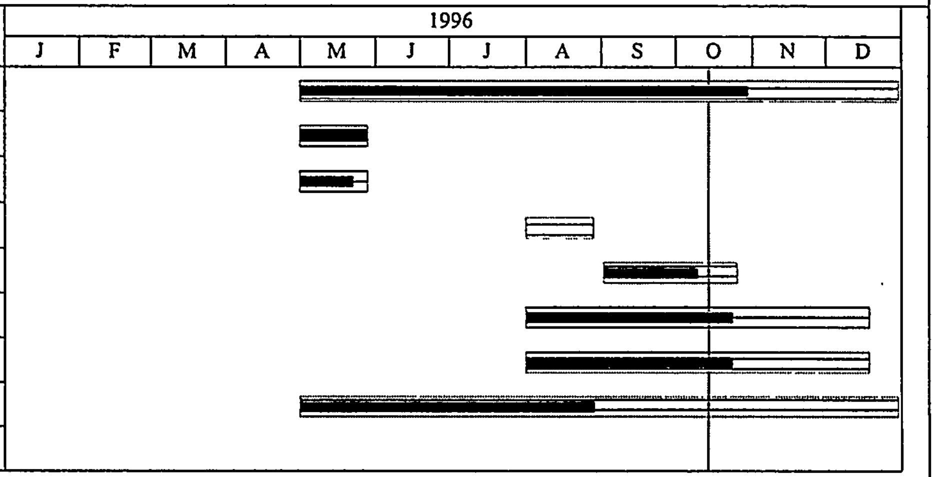
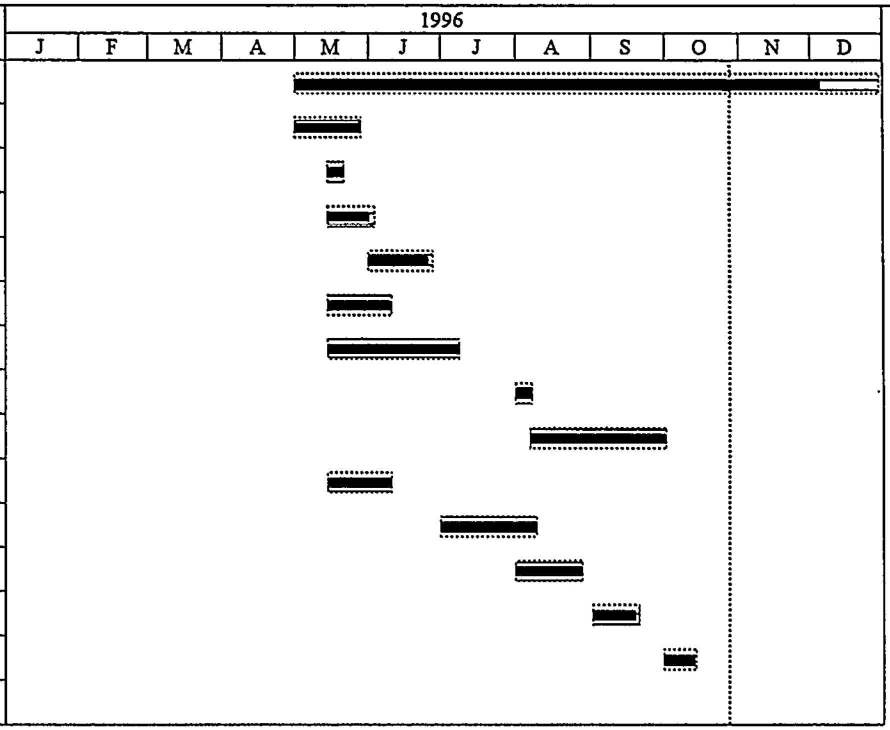
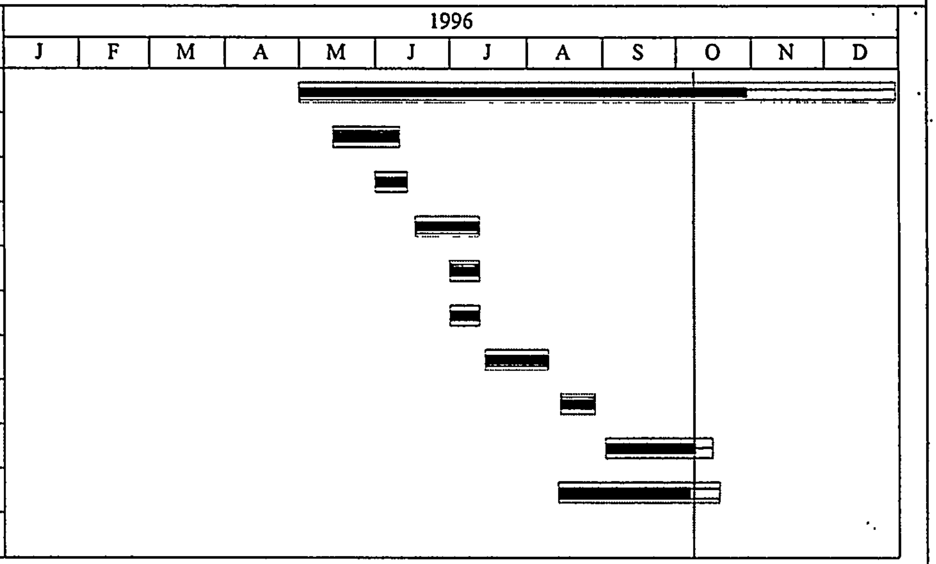
54	**Task 1-6 In Situ Bioremediation of Chlorinated Solvents**	35w	5/1/96	12/30/96	75%
55	NEPA Information	4w	5/1/96	5/28/96	100%
56	Review Existing Site Data	4w	5/1/96	5/28/96	80%
57	Procure Samples	4w	8/1/96	8/28/96	0%
58	Charcterize Samples	8w	9/2/96	10/25/96	70%
59	Evaluate Bio-Polishing/Columns	20w	8/1/96	12/18/96	60%
60	Evaluate Bio-Polishing/Micro Columns	20w	8/1/96	12/18/96	60%
61	Data Reduction/Report Development	35w	5/1/96	12/30/96	50%
62					



ID	Task Name	Dur	Start	Finish	% Comp
63	**Task 1-7A Decision Support System**	35w	5/1/96	12/30/96	90%
64	NEPA Information	4w	5/1/96	5/28/96	100%
65	Subsurface Contaminant Data Validation	1w	5/15/96	5/21/96	100%
66	Data Assembly	3w	5/15/96	6/3/96	90%
67	Data Entry	4w	6/1/96	6/27/96	90%
68	Existing Data Base Application	4w	5/15/96	6/10/96	100%
69	Data Base Development	8w	5/15/96	7/8/96	100%
70	Data Base Demonstration	1w	8/1/96	8/7/96	100%
71	Final Prototype Development	8w	8/7/96	10/1/96	100%
72	Decision Support System (DSS) Develpment	4w	5/15/96	6/10/96	100%
73	DSS Prototype Development/Demonstration	6w	7/1/96	8/9/96	100%
74	DSS Prototype Modifications	4w	8/1/96	8/28/96	100%
75	DSS Full Data Base Extension	3w	9/2/96	9/20/96	90%
76	DSS /Data Base Test/Demonstration	2w	10/1/96	10/14/96	90%
77					



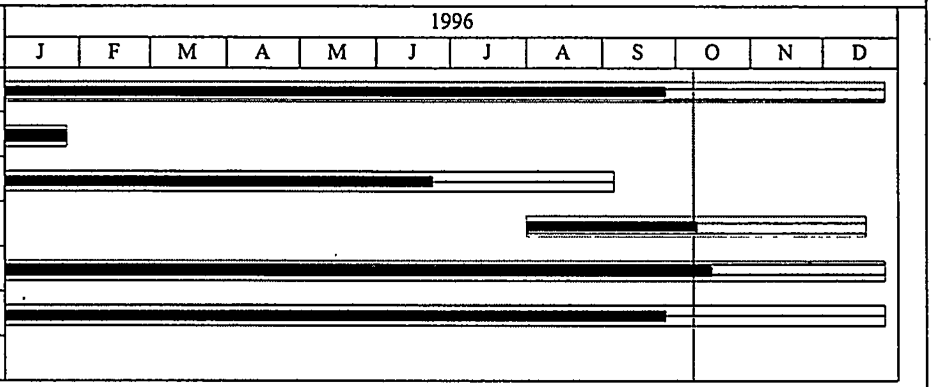
ID	Task Name	Dur	Start	Finish	% Comp
78	**Task 1-7B Prototype Data Base & Decision Support System**	35w	5/1/96	12/30/96	75%
79	Review DSS Methodology	4w	5/15/96	6/10/96	100%
80	Determination of Work Plan	2w	6/1/96	6/13/96	100%
81	Review EM Nomenclature	4w	6/17/96	7/12/96	100%
82	Build Glossary of Terms	2w	7/1/96	7/12/96	100%
83	Help System Design	2w	7/1/96	7/12/96	100%
84	Topic Data Entry	4w	7/15/96	8/9/96	100%
85	Add Special Features	2w	8/15/96	8/28/96	100%
86	Review/Debug	2w	9/2/96	10/15/96	50%
87	AHP Doccumentation	8w	8/14/96	10/18/96	75%
88					



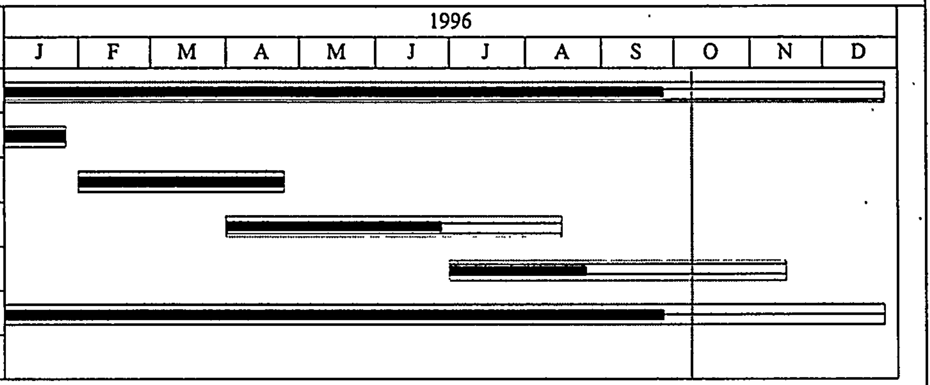
TASK 2.0

Cross Cutting Innovative Technologies

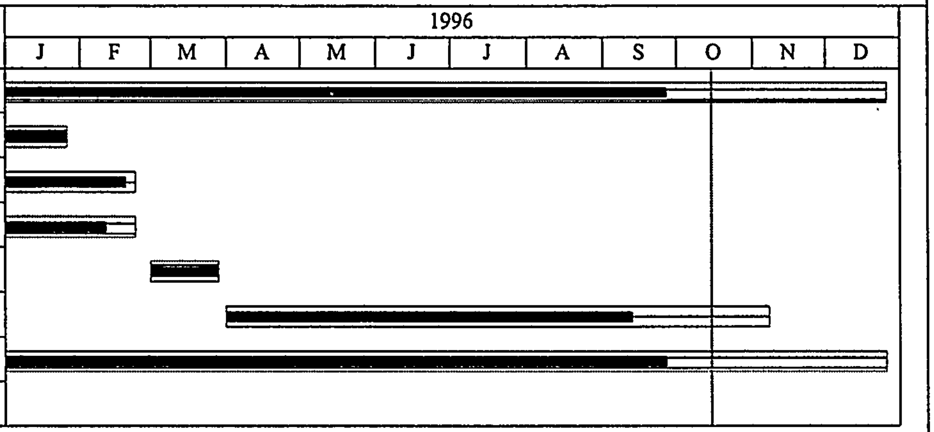
ID	Task Name	Dur	Start	Finish	% Comp
89	**Task 2-1 GIS - Based Infrastructure**	52w	1/1/96	12/26/96	75%
90	NEPA Information	4w	1/1/96	1/26/96	100%
91	GIS Application	36w	1/1/96	9/5/96	70%
92	Assessment for D&D Use	20w	8/1/96	12/18/96	50%
93	Tutorial Development	52w	1/1/96	12/26/96	80%
94	Data Reduction/Report Development	52w	1/1/96	12/26/96	75%
95					



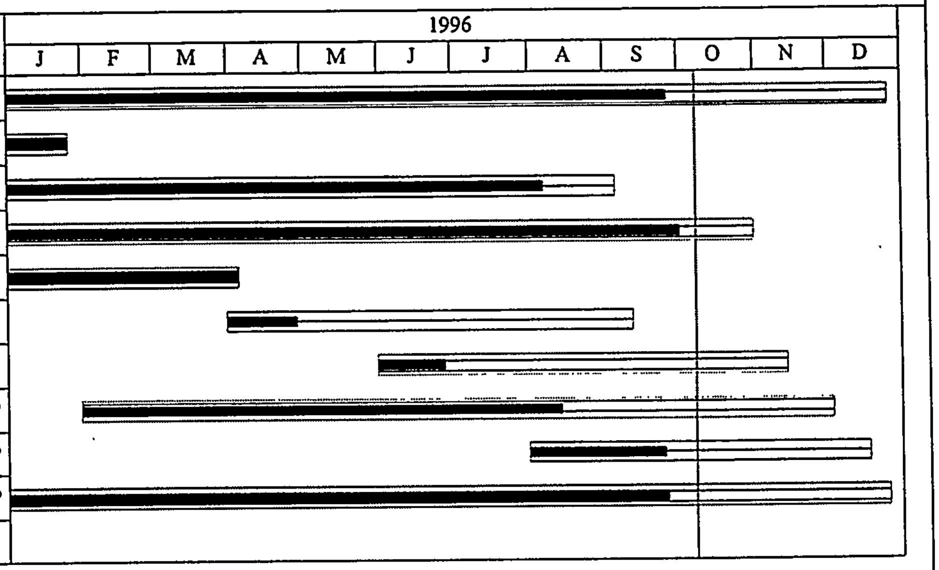
ID	Task Name	Dur	Start	Finish	% Comp
96	**Task 2-2 Treatment of Mixed Wastes via Steam Reforming**	52w	1/1/96	12/26/96	75%
97	NEPA Information	4w	1/1/96	1/26/96	100%
98	Calibrate GC	12w	2/1/96	4/24/96	100%
99	Destruction Efficiency Evaluation	20w	4/1/96	8/15/96	65%
100	Catalyst Evaluation	20w	7/1/96	11/15/96	40%
101	Data Reduction/Report Development	52w	1/1/96	12/26/96	75%
102					



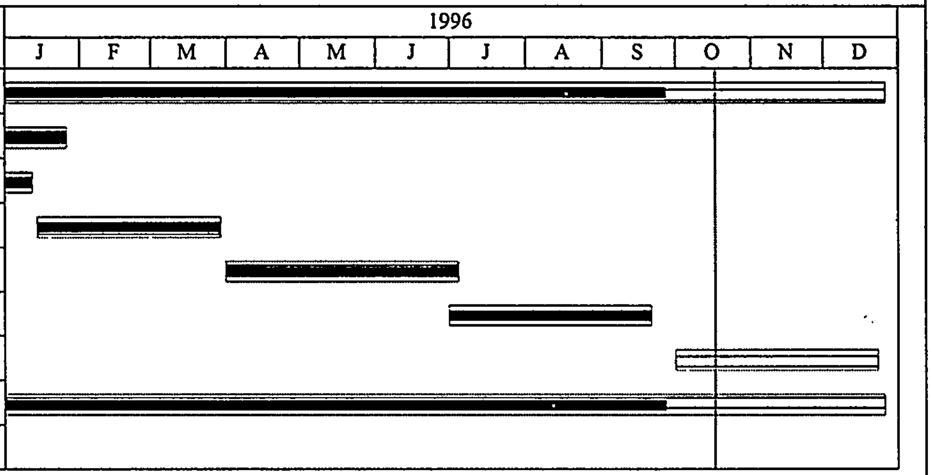
ID	Task Name	Dur	Start	Finish	% Comp
103	**Task 2-3 Centrifugal Membrane Technology**	52w	1/1/96	12/26/96	75%
104	NEPA Information	4w	1/1/96	1/26/96	100%
105	Literature Review	8w	1/1/96	2/23/96	90%
106	Waste & Membrane ID & Acq.	8w	1/1/96	2/23/96	75%
107	Technology Modification Training	4w	3/1/96	3/28/96	100%
108	Testing & Evaluation	32w	4/1/96	11/7/96	75%
109	Data Reduction/Report Development	52w	1/1/96	12/26/96	75%
110					



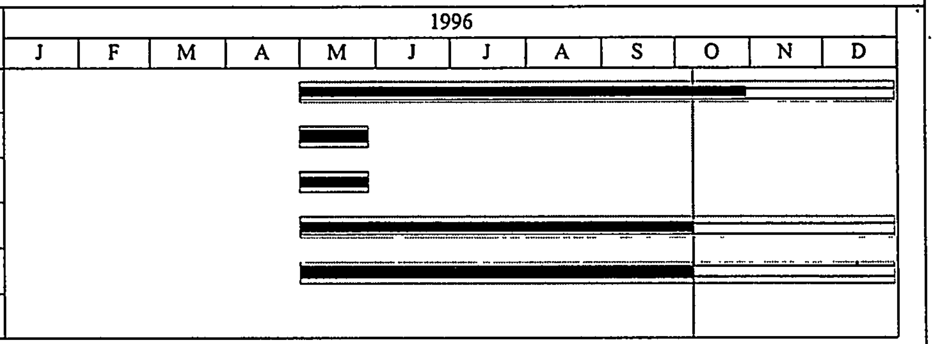
ID	Task Name	Dur	Start	Finish	% Comp
111	**Task 2-4 Environmental Pollution Control Devices**	52w	1/1/96	12/26/96	75%
112	NEPA Information	4w	1/1/96	1/26/96	100%
113	Small Cell Carbon Tests	36w	1/1/96	9/5/96	88%
114	Access Metal & Radionuclide Removal	44w	1/1/96	10/31/96	90%
115	Construct Prototype	14w	1/1/96	4/5/96	100%
116	Parametric Studies	24w	4/1/96	9/12/96	17%
117	Derive Correlations	24w	6/1/96	11/14/96	17%
118	Test Recovery Schemes	44w	2/1/96	12/3/96	64%
119	Apply Prototype	20w	8/1/96	12/18/96	40%
120	Data Reduction/Report Preparation	52w	1/1/96	12/26/96	75%
121					



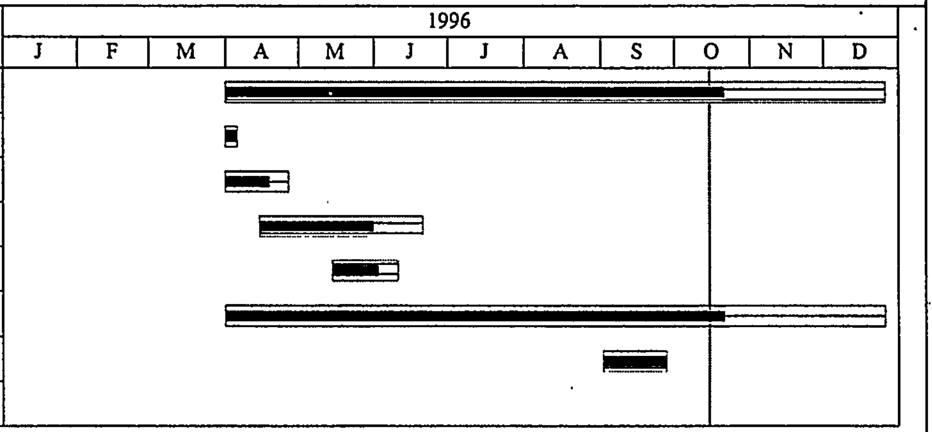
ID	Task Name	Dur	Start	Finish	% Comp
122	**Task 2-5 Development of Instrumental Methods**	52w	1/1/96	12/26/96	75%
123	NEPA Information	4w	1/1/96	1/26/96	100%
124	Literature Review	2w	1/1/96	1/12/96	100%
125	Anaylisis & Speciation of Iodine	11w	1/15/96	3/29/96	100%
126	Analysis & Speciation of Re&Tc	14w	4/1/96	7/4/96	100%
127	Analysis & Speciation of Iodine	12w	7/1/96	9/20/96	100%
128	Analysis & Speciation of Re & Tc	12w	10/1/96	12/23/96	0%
129	Data Reduction/Report Development	52w	1/1/96	12/26/96	75%
130					



ID	Task Name	Dur	Start	Finish	% Comp
131	**Task 2-6A Biosorbents & Cleaning Solutions**	35w	5/1/96	12/30/96	75%
132	NEPA Information	4w	5/1/96	5/28/96	100%
133	Development of Waste Streams	4w	5/1/96	5/28/96	100%
134	Selection & Evaluation of Solutions	35w	5/1/96	12/30/96	66%
135	Data Reduction /Report Development	35w	5/1/96	12/30/96	66%
136					



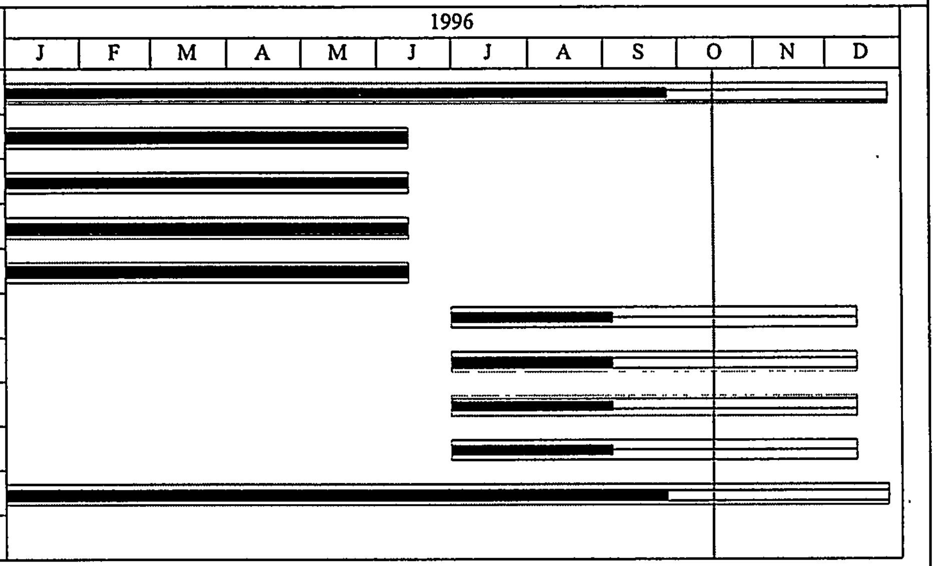
ID	Task Name	Dur	Start	Finish	% Comp
137	**Task 2-6 B SpinTek Technology**	39w	4/1/96	12/26/96	75%
138	NEPA Information	1w	4/1/96	4/5/96	100%
139	Synthetic Waste Development	4w	4/1/96	4/26/96	70%
140	Selection/Evaluation of Cleaning Solns.	10w	4/15/96	6/20/96	70%
141	Memebrane Selection	4w	5/15/96	6/10/96	70%
142	Data Reduction /Report Development	39w	4/1/96	12/26/96	75%
143	Development of Joint Proposals	4w	9/2/96	9/27/96	100%
144					



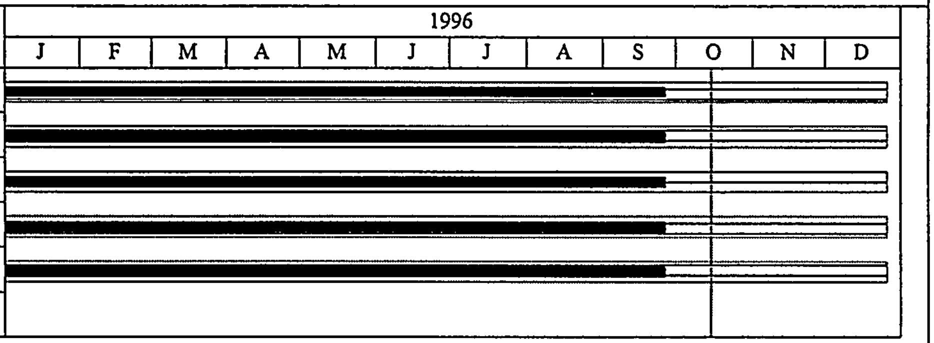
TASK 3.0

Small Business Support Program

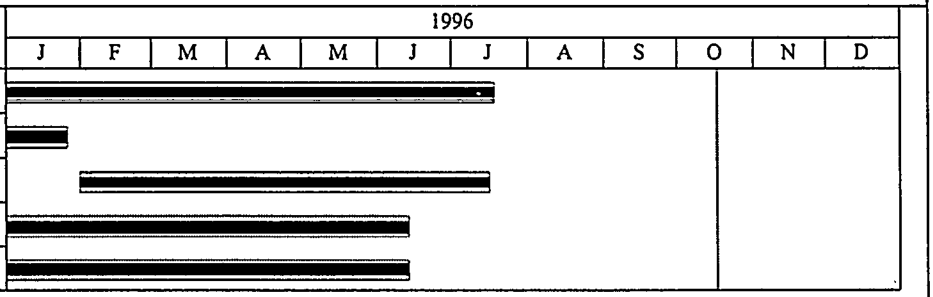
ID	Task Name	Dur	Start	Finish	% Comp
145	**Task 3-1 WVHTC**	52w	1/1/96	12/26/96	75%
146	Phase II Implementation	24w	1/1/96	6/13/96	100%
147	Start-up Activities	24w	1/1/96	6/13/96	100%
148	Infrastructure Development	24w	1/1/96	6/13/96	100%
149	Operating Policies	24w	1/1/96	6/13/96	100%
150	Phase III Continued ETI Development	24w	7/1/96	12/13/96	40%
151	Infrastructure Development	24w	7/1/96	12/13/96	40%
152	Operating Policies	24w	7/1/96	12/13/96	40%
153	Tennant Company Incubation	24w	7/1/96	12/13/96	40%
154	Report Development	52w	1/1/96	12/26/96	75%
155					



ID	Task Name	Dur	Start	Finish	% Comp
156	**Task 3-2 Small Business Interaction/Opportuni**	52w	1/1/96	12/26/96	75%
157	Develop Small Business Contacts	52w	1/1/96	12/26/96	75%
158	Team Business & University Researchers	52w	1/1/96	12/26/96	75%
159	Develop New Subtasks & Outreach	52w	1/1/96	12/26/96	75%
160	Report Development	52w	1/1/96	12/26/96	75%
161					



ID	Task Name	Dur	Start	Finish	% Comp
165	**Task 3-4 Kanawha Valley Program**	29w	1/1/96	7/18/96	100%
166	Initial Contact of Parties	4w	1/1/96	1/26/96	100%
167	Conduct Meetings	24w	2/1/96	7/16/96	100%
168	Meet w/ US EPA & WV DEP	24w	1/1/96	6/13/96	100%
169	Group Meeting	24w	1/1/96	6/13/96	100%



ANALYSIS OF THE VORTEC CYCLONE MELTING SYSTEM (CMS^{TM}) FOR REMEDIATION OF PCB CONTAMINATED SOILS USING CFD

METC Task No. 1.1

Quarterly Technical Progress Report
Reporting Period: July 1 - September 30, 1996

Work Performed Under Contract
No.: DE-FE21-92MC29467

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

by

Ismail Celik, Ph.D.

Department of Mechanical and Aerospace Engineering
West Virginia University
Morgantown, WV 26506-6106

DISCLAIMER

ABSTRACT

In this report, the results of flow and heat transfer analysis for selected retrofits of a cyclone melting system (CMS) are presented. The analysis is based on computational fluid dynamics (CFD) simulations. The assumptions, approximations, and the implications of the results with respect to the gas residence time, flow patterns, and temperature distribution, and conversion and destruction rates for selected chemical species are discussed. Problem specific mathematical models are presented. Recommendations are made for improved flow conditions that will lead to longer residence times, and higher conversion and destruction efficiency. Specific application target is the CMS^{TM} of Vortec Corporation. The results are summarized in the executive summary section as they become available for each quarterly reporting period.

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EXECUTIVE SUMMARY (current period)

Work continued in this period on the analysis of the recuperator base (residence chamber) and the recuperator itself. A complete run including all species and a global kinetic reaction of dichlorometane was performed. The temperature distribution and species concentrations inside the residence chamber have been calculate. An approximate kinetic model has been constructed to calculate the concentration of a specified hydrocarbon or a PCB compound as a function of time assuming that the concentration of oxygen is constant. Also equilibrium calculations are performed to determine the species concentrations at the exit of the residence chamber. All these results are compared with each other for consistency. The calculations with the FLUENT code confirmed a destruction time of 0.5 seconds for 99.9999% conversion of dichloromethane which was selected to represent all other PCB compounds in the flue gas. This selection was based on the kinetic model calculations which indicated the longest destruction time for dichloromethane among a dozen hydrocarbon and PCB compounds considered. Equilibrium calculations showed also that the equilibrium concentration of dichloromethane is less than one PPM which confirms the kinetic model calculations as well as the FLUENT calculations.

A preliminary simulation was performed for the recuperator where the flue gas was represented as air. The radiation model was turned off to be included later to investigate the influence. The preliminary results indicate that the temperature of the cold air stream was raised from 298 K to 835 K with a corresponding decrease of about 100 K in the flue gas temperature.

In the next quarterly period the radiation model and the species transport equations will be included to complete the analysis of the recuperator. The analysis of the separator/reservoir will also begin in the next quarterly period.

1.0 INTRODUCTION

The low level radioactive waste and other hazardous waste materials such as Polychlorinated Biphenyls (PCB) has become an important risk factor in endangering human health and environment. Each year millions of tons of contaminated soil is identified and classified for clean up only in the United States. An efficient and safe way to reduce and in most cases eliminate this risk factor to health and environment is the so called vitrification and remediation process via thermochemical treatment at high temperatures. In this process the hazardous waste and the contaminated residuals are heated, where most organic contaminants are vaporized and oxidized, while the inorganic contaminants are immobilized within a glassy, vitrified matrix. One such system has been developed by Vortec Corporation (here and after referred as Vortec) which has been demonstrated to be flexible in processing a wide variety of contaminated soils, sludges, and other hazardous materials. The system consists of a counter rotating combustor (or melter), a separation unit where liquid phase is separated from the gas phase, a residence chamber to provide reaction time to complete the conversion processes, and a recuperator which provides further residence time in addition to providing hot air/gas to be used as a heating source for the combustor (see Figure 1.0). The design, refinement, and retrofits of such systems require calculation of optimum design parameters such as residence time, heat loss, pressure drop, mixing rates, and the flow field dependent conversion and destruction (C&D) rates.

In practice where detailed flow and temperature distributions are not available, usually a plug flow assumption is used to calculate the gas residence time from empirical relations(see e.g. Van Dell et al., 1994a, 1994b; Van Dell and Mahle, 1992; Duvall and Rubey, 1977). This is a crude assumption and it may lead to significant errors. For example Van Dell and Mahle's calculations show a two-three fold difference in residence time even in a controlled, bench-scale laminar flow reactor due to temperature differences within the same reactor. Such errors directly translate to the calculation of species concentrations, hence may lead to quite erroneous information for design of PCB destruction systems. Celik and Chattree (1988) developed a rigorous method for calculating mean gas and particle residence times in an entrained flow reactor. This method (which will also be used for the present analysis) takes into account the gas temperature and density changes locally in the reactor as well as the recirculation zones. Their results also showed significant differences between the particle and gas residence time due to inertia and gravitational effects.

Furthermore, the destruction rates of PCB's are usually modeled by a simple Arrhenius type rate expressions where the gas temperature is assumed to be constant. To obtain more accurate results the gas temperature needs to be varied locally within the reactor and the reaction rates should be generalized to account for reformation of the compounds. Thus the resulting rate expression should include both the coefficients for the rate of formation as well as for the rate of destruction. Such a model has been proposed by Van Dell et al. (1994a) and it is a good candidate to be used in the present analysis.

The computation and optimization of the above mentioned design parameters can be accomplished in a cost effective way using Computational Fluid Dynamics (CFD) analysis. With CFD codes the flow field, temperature distribution, and species concentrations can be calculated at many discrete spatial points inside the reactor as well as temporal (or transient) variations. These primitive flow variables, in turn, can be used, by appropriate post processing to calculate integral design parameters of interest. The calculated design parameters along with the detailed information and virtual visualization of flow patterns can increase our understanding of the physical processes involved, and hence reduce the risk and the cost of experimental testing and/or validation.

2.0 PURPOSE

The primary objective of the proposed research work is to provide via computer simulations, needed design information such as gas residence time, temperature distribution, species concentrations, and heat and pressure losses for refining the proposed design and/or validation of the assumptions made during the design of sub-component of a cyclone melting system (CMS) currently under development by the Vortec Corporation. The analysis is to focus on specific needs for remediation of PCB contaminated soils. A secondary objective is to develop and implement a global kinetic model for the formation and destruction of selected PCB's such as *o*-dichlorobenzenes in conjunction with a readily available computational fluid dynamics (CFD) code. This model will rely upon experimental information available in the literature (see. e.g. Van Dell et al., 1994a, 1994b; Sethuraman et al., 1992; Ritter and Bozzelli, 1990; Tsang, 1990;). If successful, the proposed analysis will allow important enhancements for processing capabilities, and it will further reduce the development risk associated with commercial implementation of the CMS^{TM} technology.

3.0 BACKGROUND

An extensive research and development (R&D) program is being implemented by the Vortec Corporation with considerable funding from the Department of Energy(DOE) to provide waste processing equipment and services for industrial and institutional waste generators, and to manufacture glass and ceramic products from these waste materials. In particular, Vortec is planning to modify their existing high temperature waste processing and recycling units and retrofit it with appropriate units so that the system can be used for efficient remediation of PCB (Polychlorinated Biphenyls) contaminated soils. This requires determination of optimum design parameters, such as residence times, heat loss, pressure drop, and size for each subcomponent of the system. The conceptual system consists of four interconnected sub-components, namely, a cyclone melting system (CMS™), separation reservoir, recuperator base (or residence chamber), and recuperator (see Figure 1.0). The gas residence time in each of these components is strongly dependent on the degree of turbulent mixing and the presence of recirculation zones. The time required for complete conversion of the PCB compounds is, in turn, depends on the residence time as well as the temperature distribution. The very complex nature of the particle laden flow within the above mentioned units requires the use of sophisticated computer simulations to obtain a detailed and accurate description of the flow field, temperature distribution, species concentrations as well as calculating the total heat loss and the overall pressure drop within each unit.

Although this study specifically targets the CMS™ of Vortec Corporation, the end results could also be used, with minor modifications, in assessment of other type of incinerators for waste material. More importantly, this analysis can be extended to such cases where severely toxic and radioactive materials can be tested with computer simulations which would otherwise be impossible.

4.0 METHODOLOGY

The difficulties mentioned above can be resolved effectively using a readily available and extensively tested computational fluid dynamics (CFD) code. In this way, the PCB tracking can be done dynamically during the computation of the flow field where the local variations in the gas properties are automatically taken into account. Heat transfer and flow analysis, and PCB tracking will be performed using a commercial CFD code to numerically simulate the processes in the above mentioned units with the objective of providing supplemental information to improve the conceptual designs towards the most efficient system. Once such a code is reconfigured to simulate a certain unit, the key parameters can then be easily changed, and results can be obtained within a matter of days to determine optimum operating conditions.

The present computations are performed using the commercial code FLUENT (FUG-V4.3, 1995) This is a general purpose, multidimensional CFD code suitable for simulating flows in complex geometries. However, application to turbulent reacting flows is not trivial. To achieve properly converged solutions, and to avoid misinterpretation of large amounts of numerical data, extreme caution and expertise both in physics of fluids and in computational fluid dynamics is necessary. Moreover, the chemistry sub-model for PCB tracking is not available in any of these codes. Such a model shall be developed and attached to the main code as a subroutine. It may be necessary to use the well known chemical kinetics code, CHEMKIN (Kee et al., 1980) and/or the general purpose chemical equilibrium code, CET (Gordon et al., 1984) in an interactive manner with the hydrodynamic data that will be obtained from the CFD code.

5.0 WORK PERFORMED THIS PERIOD

In this period, analysis on the residence chamber (or the recuperator base) was completed by including the chemical reactions into the model on FLUENT along with the mixture compositions provided by Vortec Corp. In the previously reported computations, air (at 1694 K) was used as the fluid in the residence chamber. Literature survey on PCB kinetic rates was completed and dichloromethane was selected as the representative PCB to be simulated. Equilibrium composition calculations were performed using NASA CET89 code (Gordon et al. 1989) for the residence chamber. The results are compared with the kinetic calculations.

The CFD code, FLUENT , was also used to model the recuperator. The recuperator is a parallel flow heat exchanger that uses the outgoing flue gas to preheat the incoming cold air. The recuperator was assumed to be axisymmetric. This is believed to be a good assumption, because it consists of a long tube within a tube without any change in the boundary conditions. The header assembly was assumed not to influence the heat transfer, because its role was to distribute the air evenly throughout the pipe. For the case simulated, the dimensions and material properties provided by the Vortec Corporation were used. As a first approximation, the flue gas was assumed to be standard air at 1636 K and radiation was not included. This temperature was used with the rationale that, there is an approximately 60 K temperature drop due to heat loss from the residence chamber. The mass flow rate of the flue gas was run at 1.0 kg/s and the mass flow rate of the incoming air was considered approximately .5 kg/s. The calculated outlet temperature for the residence chamber when FLUENT was used, is about 1640 K. (For future calculations, the mass flow rate of air will be varied from 0.1 kg/s to 1.0 kg/s. This will give a flexible range of operation for the recuperator.)

6.0 RESULTS AND DISCUSSION

6.1 Recuperator Base Calculations (including reactions) :

PCB Conversion Time Computations :
Air in the previous simulations was replaced by a hydrocarbon and the other flue gas species entering to the recuperator base. The mass fraction of the species at the inlet of the system are summarized in Table 1. Thermal conductivity and viscosity were computed as a mixture property. Specific heat capacity was set as a constant while defining the properties of this mixture in Fluent.

Table 1. Mass fraction of the exhaust gas entering to the recuperator base

Compound	Mass (kg/s)	Mass Fraction at the inlet
CO_2	1.916×10^{-1}	190.9×10^{-3}
H_2O	96.66×10^{-3}	99.39×10^{-3}
N_2	6.286×10^{-1}	680.7×10^{-3}
O_2	26.84×10^{-3}	26.7×10^{-3}
SO_2	0.00	0.00
C_6H_5Cl	53.33×10^{-9}	689.7×10^{-6}
Particulate Mass (PM)	14.11×10^{-3}	1.406×10^{-3}

A dichloromethane reaction was selected to simulate the effect of all PCB compounds. This reaction is given by,

$$CH_2Cl_2 + O_2 \Longrightarrow 2HCl + CO_2$$

The global reaction rate calculations have shown that it takes longer time to destroy dichloromethane compared to other PCB's. A mass fraction of 5.18×10^{-4} (5.26×10^{-4} kg/s) was prescribed for dichloromethane at the inlet. This figure is based on the 175 PPM PCB content of the flue gas, as given by Vortec Corp. (see Appendix C for the details of the mass fractions used in the simulation).

Figure 1.1 shows the enlarged view of the inlet of the recuperator base along with the contours of mass fraction of dichloromethane (CH_2Cl_2). Concentration level decreases significantly within half radius of the residence chamber from the inlet. This shows that the destruction in the residence chamber takes place in a very short time even for dichloromethane. Similarly Figure 1.2 shows the contours of mass fraction of HCl which increases rapidly from the inlet. The mass weighted average concentration of CH_2Cl_2 and HCl at the outlet and inlet are given in Table 2.

Table 2. Inlet and outlet concentration of important species

Species	Inlet Concentration	Outlet Concentration
CH_2Cl_2	5.18×10^{-4}	5.89×10^{-14}
HCl	0.0	4.45×10^{-4}

The comparison of conversion rates of the dichloromethane and the time required for the corresponding conversion are summarized in Table 3. The time values obtained from Fluent are based on the observations obtained by massless particle trajectories. The values listed under calculations column are obtained from a constant temperature kinetic model. The accuracy level of Fluent was not sufficient to obtain the time required for an 99.9999% conversion. However, the remaining Fluent based data shows a good agreement with the approximate kinetic model calculations.

Table 3. Conversion rate comparison for CH_2Cl_2

Time (sec)	Conversion rate of CH_2Cl_2	
	Fluent	Calculations*
0.025	57 %	50 %
0.05	80 %	75 %
0.11		99.9999 %

* Constant temperature kinetic model

Temperature Distribution in the Recuperator Base:

Figures 2.1-2.4 show the variation of radial temperature profiles at various theta values and several axial locations in the recuperator base. The corresponding locations for i and k grid points are given in Table 4.

Table 4. Corresponding locations for various i and k grid points

k	axial distance (m)
2	0.04
8	0.31
16	0.61
34	1.56
54	3.70

i	angle (°)
2	18
4	36
6	54
12	108
22	198
32	288

Note that k = 54 (x = 3.70 m) corresponds to the plane at the center of the inlet.

As seen from the profiles, temperature variation can be considered as axisymmetric. The large temperature drop seen in Fig. 2.1 is due to the wall segments at the base which are cooler than the flow.

The maximum temperature in the system is 1694.024 K which indicates a slight increase in the temperature compared to the inlet temperature of the species (i.e., 1694 K). The enthalpy of combustion calculations show that approximately 6 kJ of energy will be available as a result of the dichloromethane reaction with the given inlet concentrations. Considering the fact that there is a heat loss of 2440 W/m^2 through the walls of the residence chamber, one can justify the slight increase in the temperature.

6.2 Chemical Equilibrium Calculations:

Chemical Equilibrium has many practical applications. From I.C. engines to waste incinerators, equilibrium gives us a fundamental understanding of what chemical species are left after a chemical reaction that have taken place in a relatively short time. If a reaction takes a long time to occur, then Chemical Kinetics would be a better choice. For many fast reactions, Chemical Equilibrium gives acceptable results. The gas residence time for the Vortec Corp. recuperator base is about 2.0 sec which was shown to be much larger than the reaction times using kinetic model. Hence we expect that Chemical Equilibrium assumption should be valid.

For this project, dichloromethane was used as a model of the chlorinated hydrocarbons or PCB, because of its relatively slow Chemical Kinetics. Although heavier chlorinated hydrocarbons and PCB's may be present, their global (overall) kinetic reactions occur much faster than that of dichloromethane. This means that the intermediate products would react quickly. First air was used as the oxidizer in both the approximate model and the general chemical equilibrium calculations. Then the general equilibrium calculations were applied to the flue gas with the actual inlet composition. Further details on the conversion rates and times for selected hydrocarbons and PCB compounds is given in Appendix C.

The results of the actual equilibrium calculations are shown in Table 3 and Figures 3.1 and 3.2. The equilibirum calculations not only on PCB concentration, but also on concentrations of minor species like CO, NO, Cl and O (see Appendix C for more discussion). The most important results from the CET calculations is that more than 99.9999 % destruction is achievable, and less than 1 PPM of dichloromethane is present at equilibrium conditions. This condition holds true for both temperatures of 1694 K and 1600 K (see Appendix E, Table 3 and Figures 3.1 and 3.2).

NOTE: Page 8 is missing from this section

7.0 CONCLUSIONS (*for the current period of performance*)

The computer simulations of the three-dimensional, turbulent, chemically reacting flow inside the residence chamber showed that 75-80% conversion of the dichloromethane takes place in about 0.05 sec. The gas-residence time (based on air as the fluid) was previously calculated to be greater or equal to 2.0 seconds. Although residence time computations for the exhaust gas species and the combustion products are in progress, these initial figures indicate that conversion of dichlorobenzene will be completed within approximately 0.5 sec. The temperature variation in the recuperator base shows a nearly axisymmetric distribution through out majority of the system with a maximum drop of approximately 60K. Equilibrium calculations for the recuperator base shows traces of readicals like CO, NO, Cl, and O. These calculations also inidicate more than 99.9999% PCB conversion at equilibirum conditions.

The simulation of the recuperator using FLUENT as an axisymmetric case shows that the incoming air can be preheated to 834 K when there is no radiation present. Preliminary results show that with radiation included in the simulation, the incoming air can be preheated to 1285 K. Although more work needs to be done on the recuperator simulations, these results give insight to the actual temperature of the preheated air for a range of conditions.

8.0 WORK PLANNED FOR NEXT PERIOD

For future work, the P -1 Radiation model will be extensively applied to the recuperator. The recuperator will be simulated over a range of mass flow rates of the incoming air from 0.1 kg/s to 1.0 kg/s. This will give a flexible range of operation for the recuperator. Chemical species will then be added to include the radiation effects of the CO_2 and H_2O present in the flue gas. A Discrete Transfer Radiation Model (DTRM)will be substituted for the P -1 model and results will be compared. Finally, we will include solid and liquid phases in the recuperator to account for any condensation or solidification in the recuperator. The reservoir/separator analysis will also be completed in this period. There the focus will be on predicting the species concentrations at the outlet of the separator (i.e. the inlet of the recuperator base).

APPENDIX A

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APPENDIX B: FIGURES

VORTEC'S CMS SYSTEM

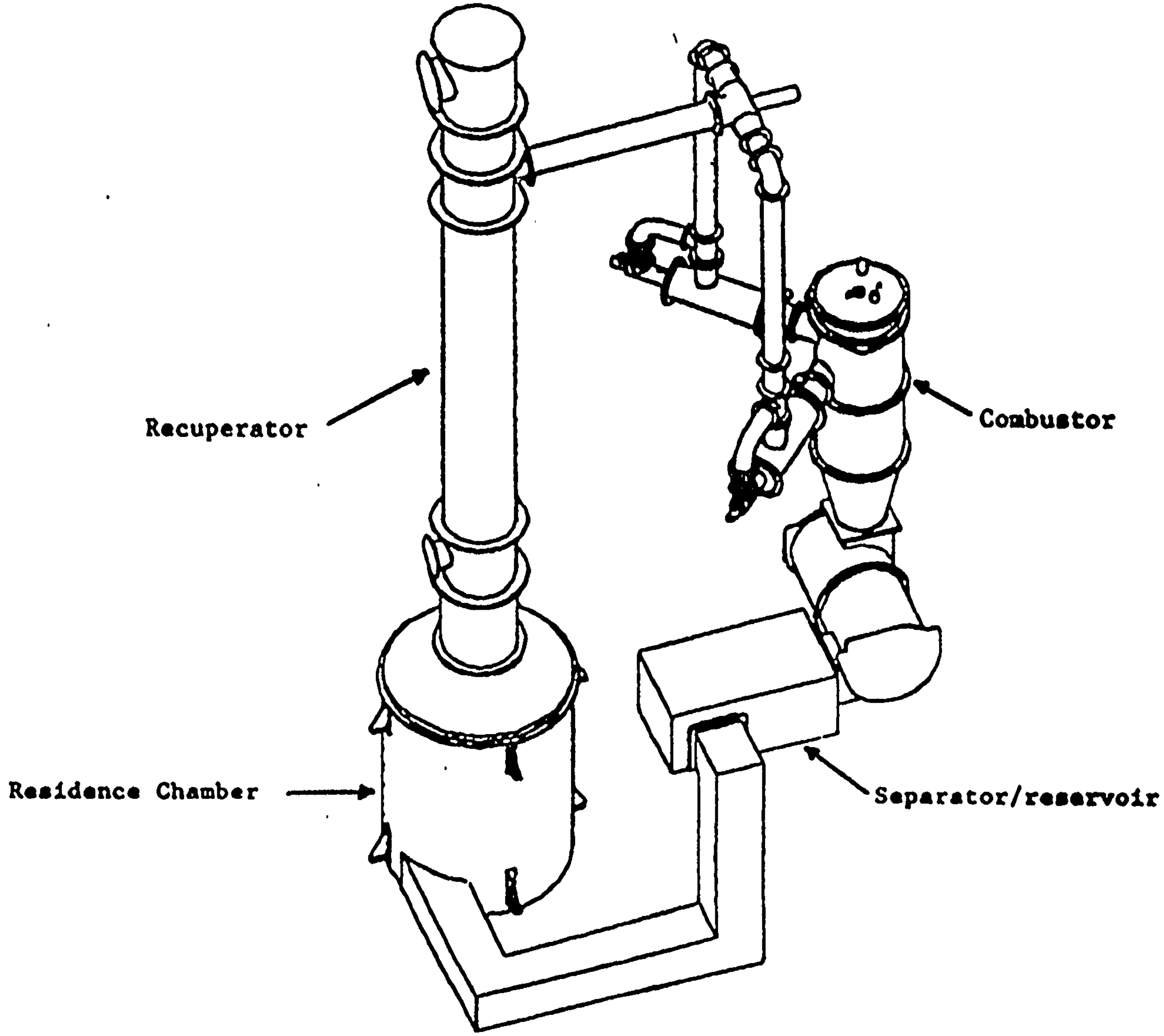
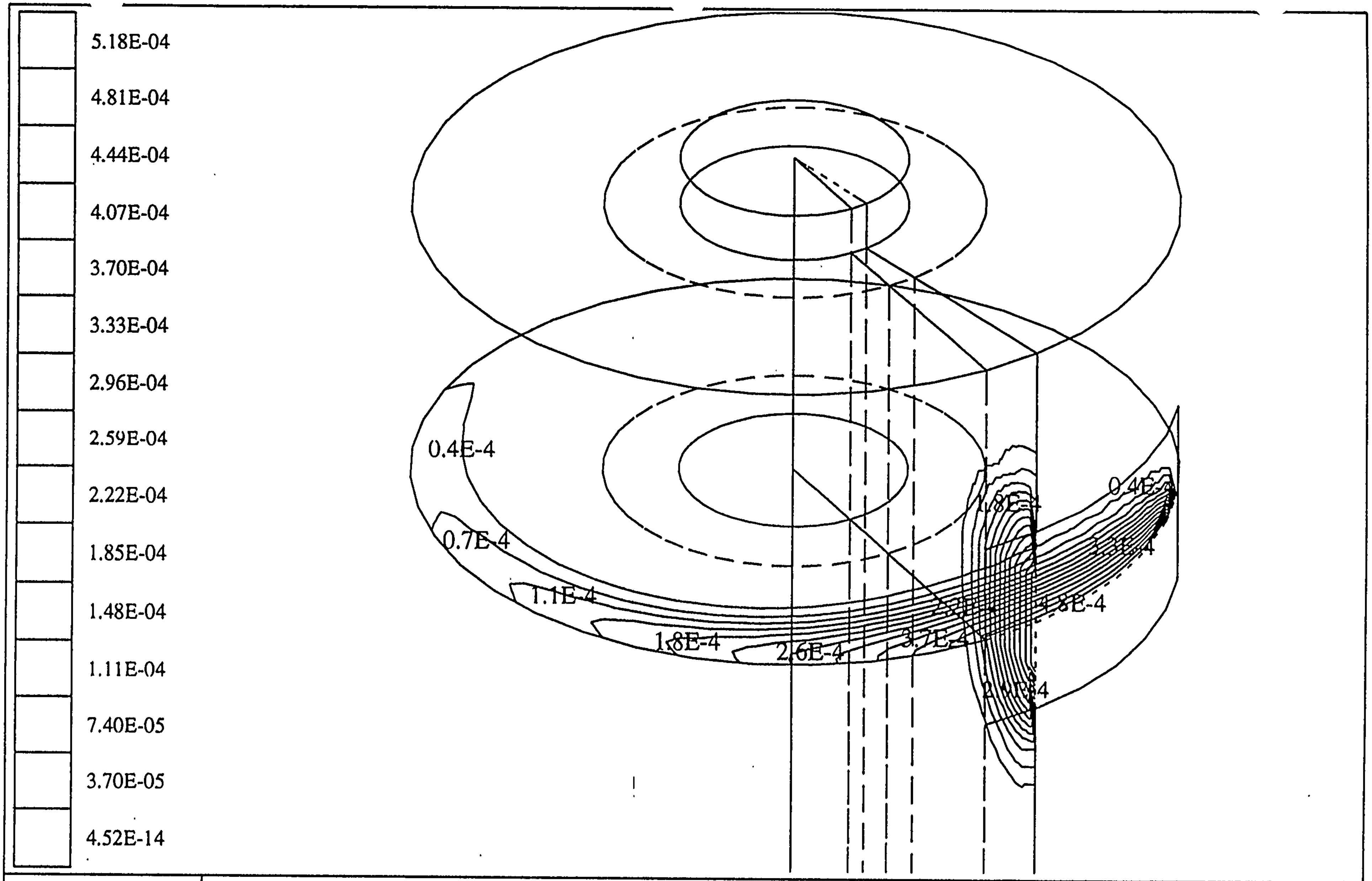
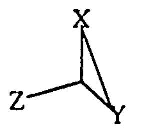


Figure 1. Vortec CMS$^{\text{TM}}$ System Sketch



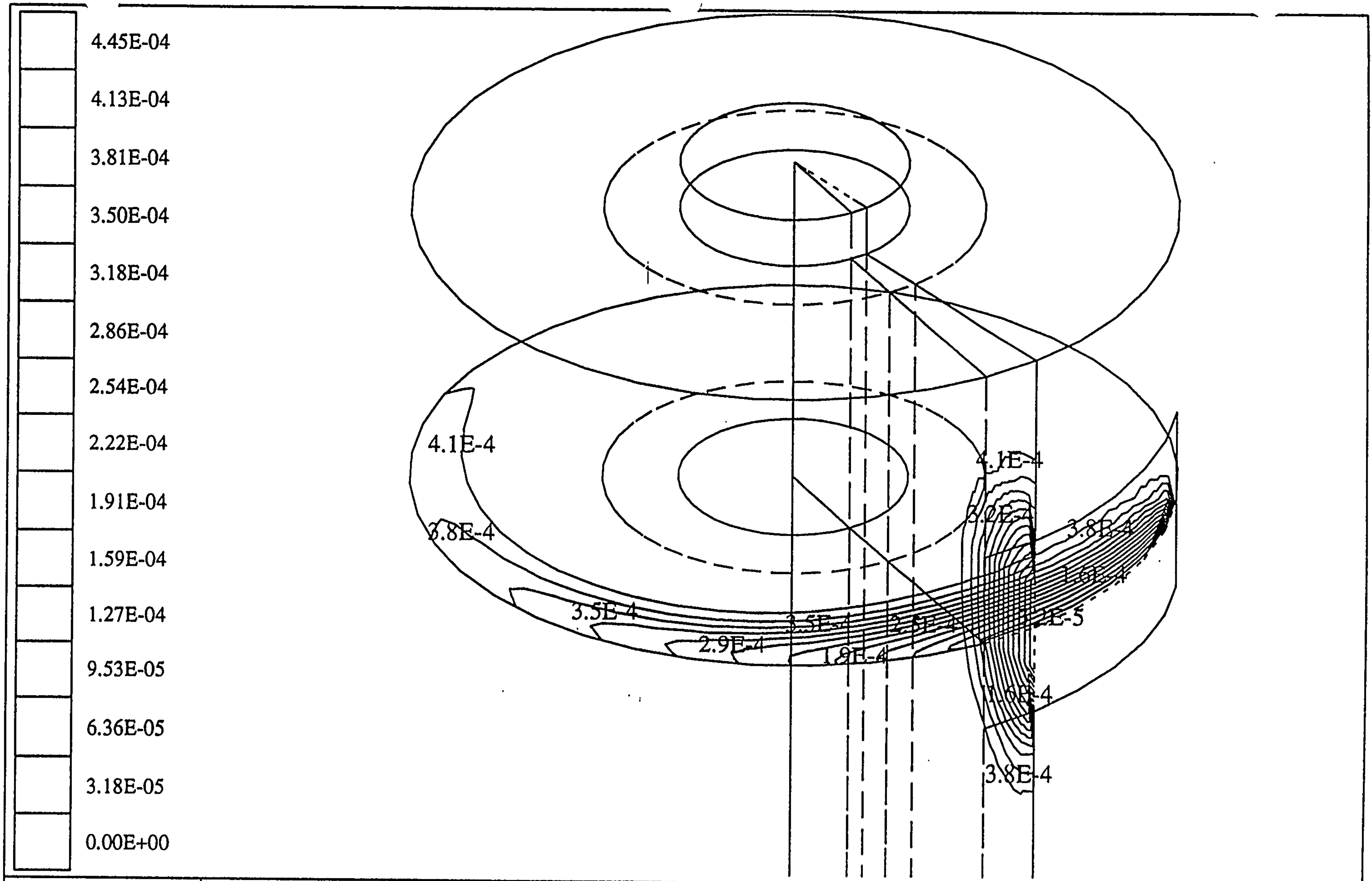


VORTEC RECUPERATOR BASE NEW 3D GEOMETRY

Fig. 1.1 CH_2Cl_2 Mass Fraction Contour Plot (Enlarged view of the inlet)

Lmax = 5.180E-04 Lmin = 4.524E-14

Oct 03 1996
Fluent 4.32
Fluent Inc.



VORTEC RECUPERATOR BASE NEW 3D GEOMETRY

Fig. 1.2 HCl Mass Fraction Contour Plot (Enlarged view of the inlet)

Lmax = 4.449E-04   Lmin = 0.000E+00

Oct 03 1996
Fluent 4.32
Fluent Inc.

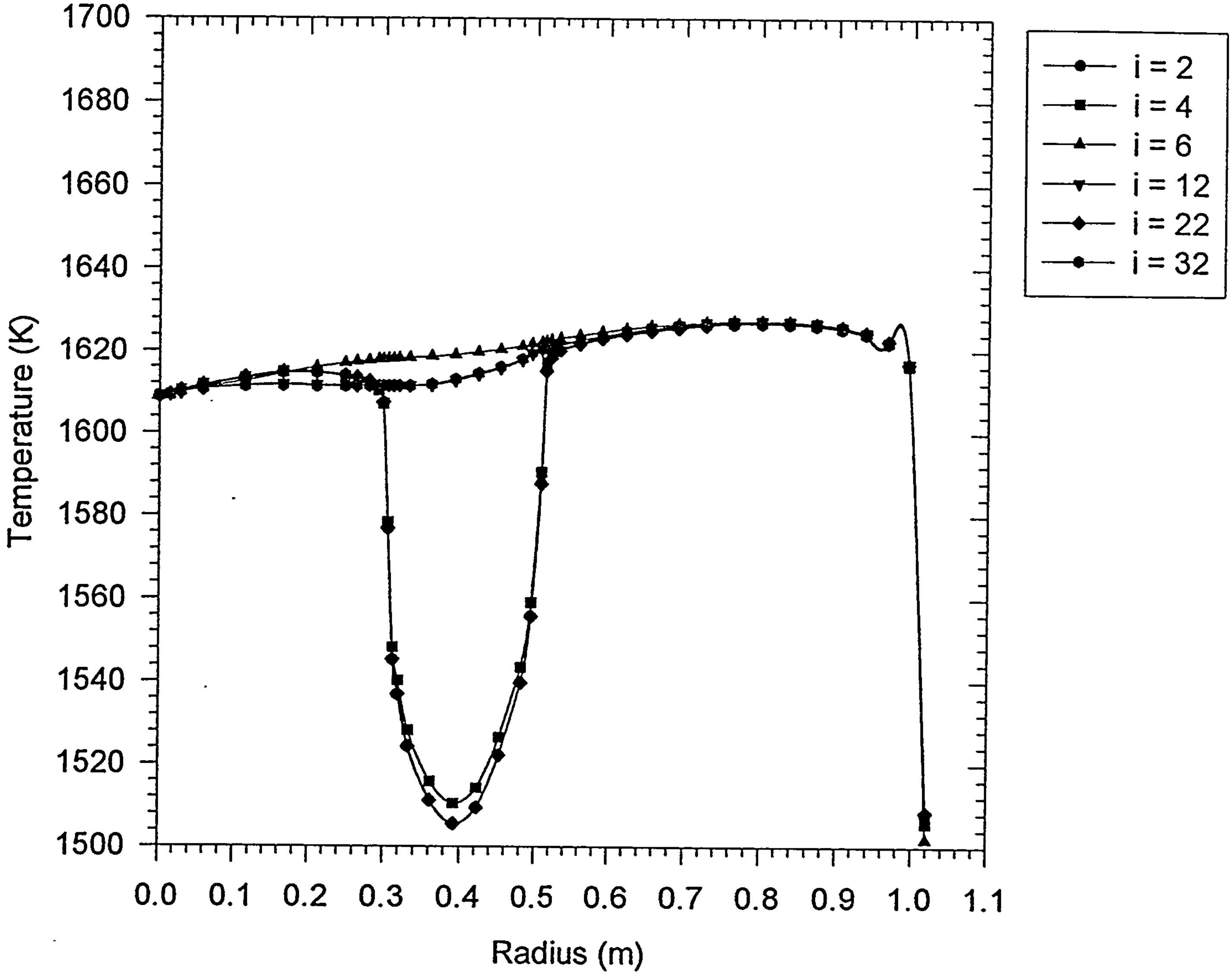


Figure 2.1 Radial Temperature Profiles at the base (k = 2)

Note : The corresponding locations for i and k grid points are listed in Sec. 6.1

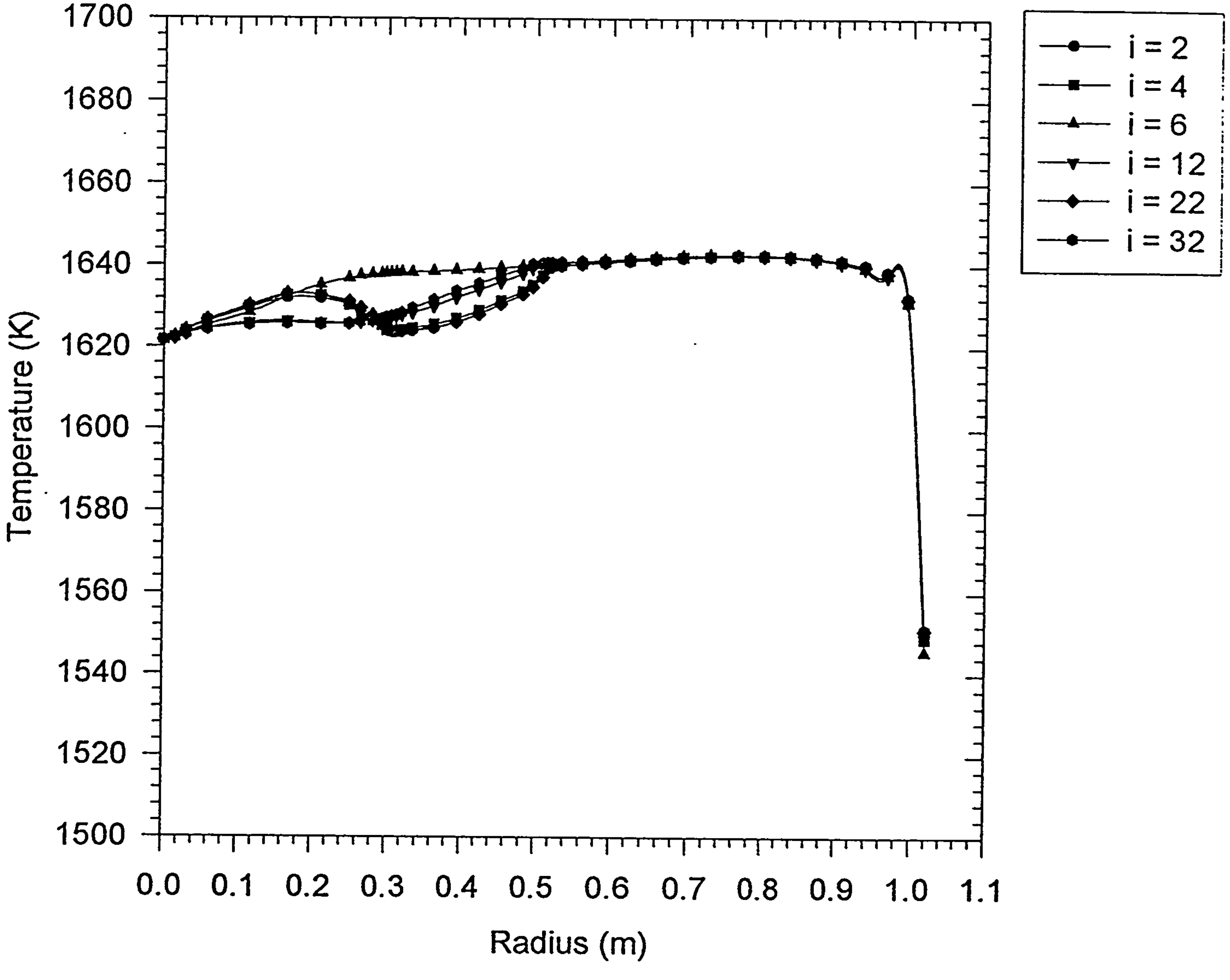


Figure 2.2 Radial Temperature Profiles at k = 8

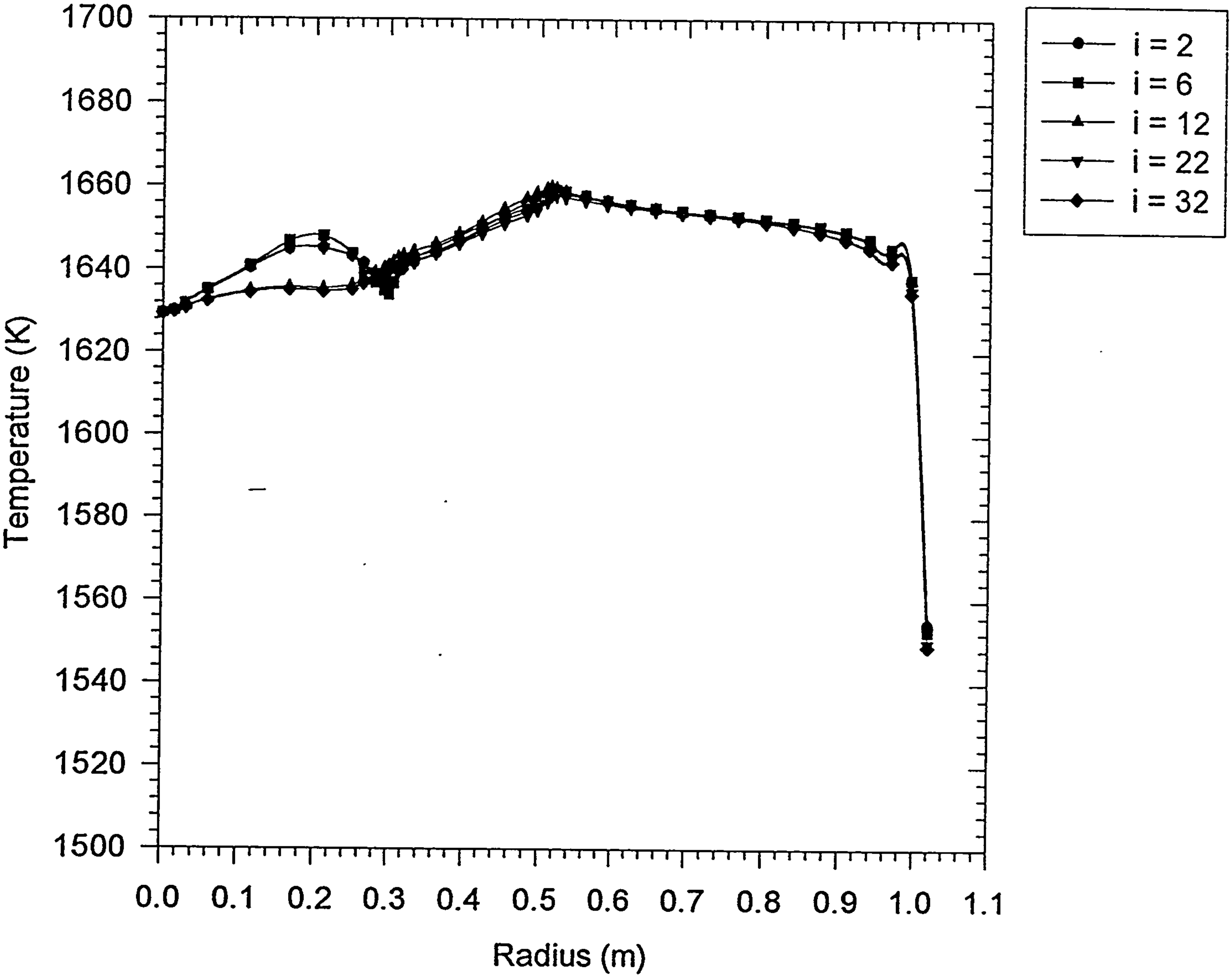


Figure 2.3 Radial Temperature Profiles at k = 16

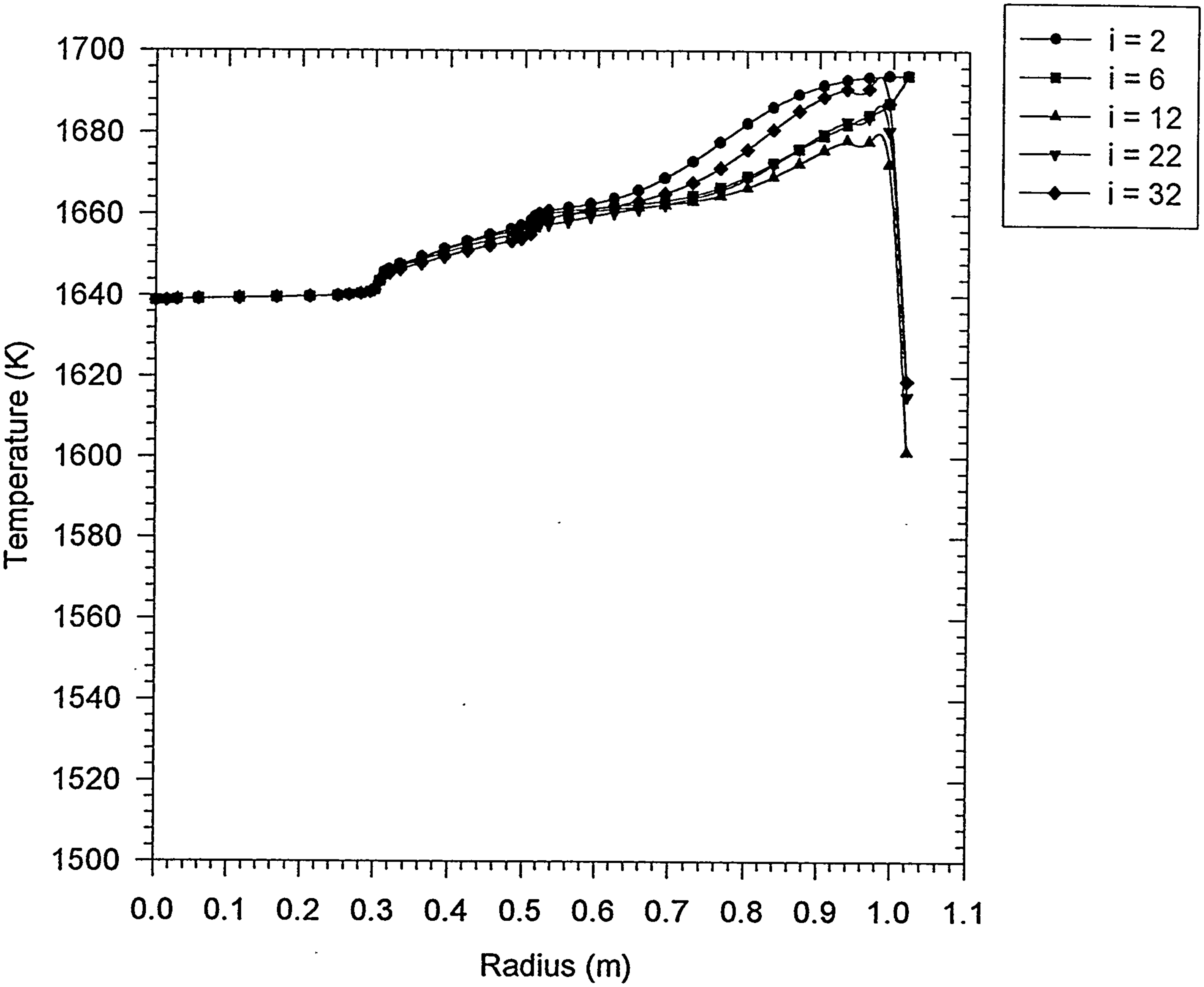


Figure 2.4 Radial Temperature Profiles at inlet (k = 54)

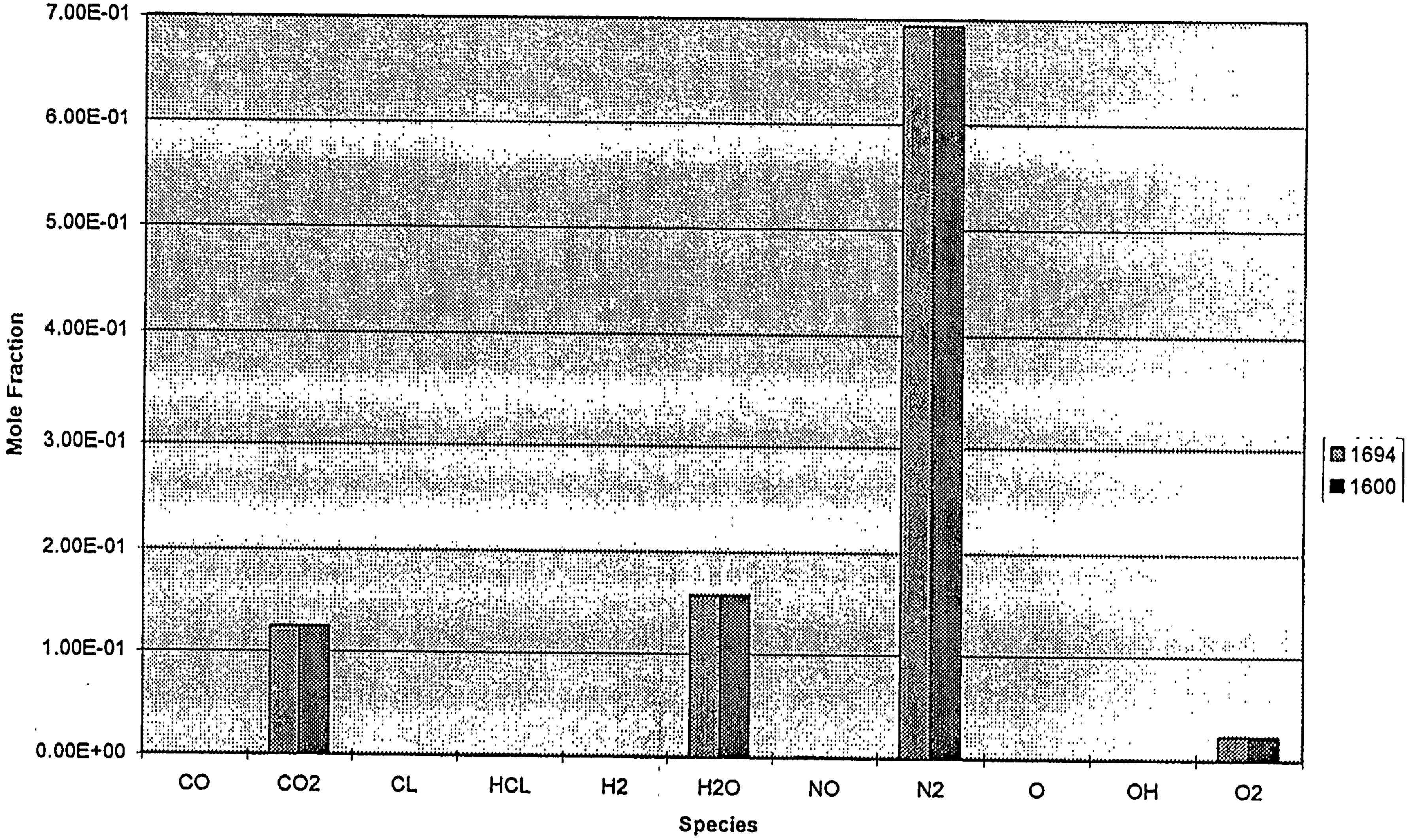


Figure 3.1 Mole Fractions of Flue Gas at Equilibrium (Major Species Only)

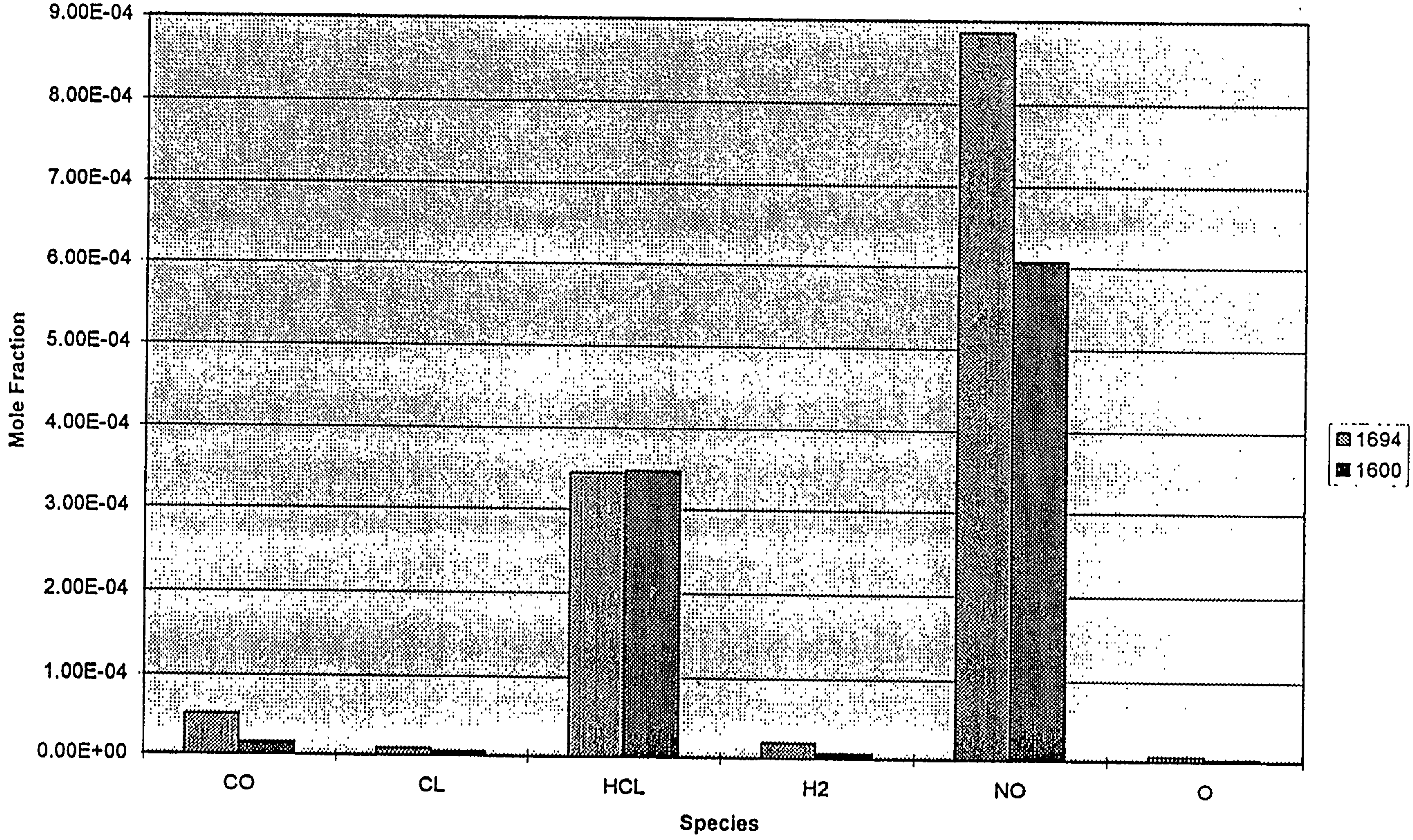


Figure 3.2 Mole Fractions of Flue Gas at Equilibrium (Minor Species Only)

APPENDIX C:
Conversion Rates and Times for Selected Hydrocarbons and PCB Compounds

Conversion rates and destruction times for some selected hydrocarbons and PCB compounds have been calculated. The operation conditions for the residence chamber are given in Table 1 in accordance with Vortec's specifications. As a first step we have considered one global reaction of the form

$$C_aH_bCl_d + [a+(b-d)/4]O_2 \Longrightarrow aCO_2 + dHCl + (b-d)/2\ H_2O \qquad (1)$$

with an Arrhenius reaction rate, K*, such that

$$d[HC]/dt = -K^*\ [HC] \qquad (2)$$

where [HC] is the concentration of the hydrocarbon being considered

We have compiled the reaction rates from the literature where K* is usually given for a situation where the Oxygen concentration is taken to be constant. We have devised a method to estimate the oxygen concentration and made the reaction rate coefficient K be dependent on the concentration of the hydrocarbon as well as that of oxygen. The resulting expression for K is

$$K = [HC]^{(m-1)}\ [O_2]^n\ A\ Exp(-E/Ru\ T) \qquad (3)$$

It should be mentioned that for the present operation conditions the equivalence ration, ϕ, is very small (typically less than 0.01) indicating that the mixture is very much fuel-lean, and hence assuming the oxygen concentration to be constant should be a good approximation.

The reaction rates and the associated parameters as defined in Equation (3) are listed in Table 2. In some case we had to do extrapolation by curve fitting using carbon to hydrogen ratio as an independent variable, e.g. for Naphthalene. Using these reaction rates we have calculated destruction times for 99.99% and 99.9999% conversion rates (see Table 2) at a reaction temperature of 1694 K (2590 °F). It is seen that the residence times required even for six-nine conversion rates are very small. The longest time requirement is 0.5 sec which is in case of Dichloromethane. These results confirm our previous report which basically stated that the kinetic reactions for most of these compounds are very fast hence equilibrium should prevail for a perfectly well mixed reactor and a premixed mixture which are the conditions specified currently.

Fluid dynamics computations with FLUENT computer code indicated that there can be a localized temperature drop of as much as 100 K from the initial temperature of 1694 K (2590 deg. F) as a result of heat loss from the walls amounting to about 5% of the total energy content of the flue gas at the inlet of the residence chamber. To account for this temperature drop we computed the conversion rates and residence times for selected PCB and hydrocarbon compounds at a temperature of 1600 K (2421 deg. F). The results of these calculations area listed in Table 2b. It is seen that this reduction temperature leads to a significant increase in the conversion times (see Table 2a for the results at 1694 K).

The longest conversion time is still for Dichloromethane, which is approximately one second for a six nines destruction rate.

We also performed equilibrium calculations at two temperatures, namely 1694 K (2590 deg. F) and 1600 K (2421 deg. F) assuming all of the given 175 PPM of PCB is composed of Dichloromethane at the inlet of the residence chamber. The molar concentrations of the other species entering the residence chamber are given in Table 1 for reference. The computed equilibrium concentrations for various species are listed in Table 3 which are also presented in Figures 1 and 2 as bar charts. It is observed that some traces of O, Cl, and CO are present. The NO concentration is considerably large at both temperatures, which is 885 PPM and 606 PPM at the higher and lower temperature, respectively.

Table 1a Mass Fractions of Exhaust Gas Entering the Residence Chamber
(Chloromethane as PCB)

Compound	N_i	X_i	M_i	eta_i	mass	Y_i
	(kmole/s)		*(kmole/kg)*		*(kg/s)*	
CO2	0.004355	0.123184	44.00	1.531905	0.191600	0.188706
H20	0.005537	0.156629	18.00	0.626688	0.099663	0.098158
N2	0.024379	0.689635	28.00	0.974849	0.682600	0.672290
02	0.000839	0.023724	32.00	1.114113	0.026837	0.026432
SO2	0.000000	0.000000	0.00	0.000000	0.000000	0.000000
Particulate Mass (PM)	0.000235	0.006653	60.00	2.088962	0.014110	0.013897
CH2Cl2	0.000006	0.000175	85.00	2.959362	0.000526	0.000518
Total	0.035350	1.000000	267.00		1.015336	1.000000
Average			28.72			
Total - PCB	0.035344					

note: CH2Cl2 is calculated from the given concentration of 175 PPM PCB present in the flue gas.
CH2Cl2 was assumed to represent the whole mixture of the compounds.

note: The Particulate Mass was assumed to be quartz, SiO2.
It's molecular weight is 60 kg/kmole.

Symbols	Representation
i	each species component
n	the number of kmoles per second (kmole/s).
y	the molar fraction, M is the molecular weight (kg/kmole)
eta	the molecular weight divided by the total (computed) molecular weight
x	the mass fraction

Table 1b Mass Fractions of Exhaust Gas Entering the Residence Chamber (Chlorobenzene as PCB)

Compound	N_i	X_i	M_i	eta_i	mass	Y_i
	(kmole/s)		*(kmole/kg)*		*(kg/s)*	
CO2	0.004355	0.123184	44.00	1.531649	0.191600	0.188674
H20	0.005537	0.156629	18.00	0.626583	0.099663	0.098141
N2	0.024379	0.689635	28.00	0.974685	0.682600	0.672177
O2	0.000839	0.023724	32.00	1.113926	0.026837	0.026427
SO2	0.000000	0.000000	0.00	0.000000	0.000000	0.000000
Particulate Mass (PM)	0.000235	0.006653	60.00	2.088612	0.014110	0.013895
C6H5Cl	0.000006	0.000175	112.50	3.916147	0.000696	0.000685
Total	0.035350	1.000000	294.50		1.015506	1.000000
Average			28.73			
Total - PCB	0.035344					

note: C6H5Cl is calculated from the given concentration of 175 PPM PCB present in the flue gas.
C6H5Cl was assumed to represent the whole mixture of the compounds.

note: The Particulate Mass was assumed to be quartz, SiO2.
It's molecular weight is 60 kg/kmole.

Symbols	Representation
i	each species component
n	the number of kmoles per second (kmole/s)
y	the molar fraction, M is the molecular weight (kg/kmole)
eta	the molecular weight divided by the total (computed) molecular weight
x	the mass fraction

Table 2a Destruction Times for Selected Hydrocarbon and PCB Compounds

				Destruction T=1694 K	
Formula	Name	A *m^3/kmole*s*	E *J/kmole*	Time at 99.99% *s*	Time at 99.9999% *s*
C6H6	Benzene	2.59E+24	4.01E+08	4.83E-08	7.24E-08
C6H5Cl	Chlorobenzene	4.83E+19	3.21E+08	8.68E-06	1.30E-05
C6H4Cl2	m-Dichlorobenzene	1.71E+17	2.62E+08	3.79E-05	5.69E-05
C2H4Cl2	1,2-Dichloroethane	1.52E+14	1.91E+08	2.75E-04	4.13E-04
CH4	Methane	6.88E+13	2.18E+08	4.13E-03	6.20E-03
CH3Cl	Methyl Chloride	2.87E+11	1.71E+08	3.52E-02	5.28E-02
CH2Cl2	Dichloromethane	4.00E+10	1.75E+08	3.36E-01	5.03E-01
CHCl3	Trichloromethane	1.27E+14	1.93E+08	3.81E-04	5.71E-04
CCl4	Tetrachloromethane	5.02E+10	1.48E+08	3.93E-02	5.90E-02
C10H18	Decahydro-Napthalene*	4.04E+08	1.26E+08	4.13E-04	4.13E-04
C2H3Cl	Vinyl Chloride	1.24E+17	2.65E+08	6.43E-05	9.65E-05

note: K= Ae^(-E/RuT)[Hc]^(m-1)[O2]^n

	m	n
Decahydro-Napthalene	0.25	1.5
All Others	1	1

Table 2b Destruction Times for Selected Hydrocarbon and PCB Compounds

				Destruction T=1600 K	
Formula	Name	A	E	Time at 99.99%	Time at 99.9999%
		m3/kmole*s	_J/kmole_	_s_	_s_
C6H6	Benzene	2.59E+24	4.01E+08	2.43E-07	3.64E-07
C6H5Cl	Chlorobenzene	4.83E+19	3.21E+08	3.13E-05	4.69E-05
C6H4Cl2	m-Dichlorobenzene	1.71E+17	2.62E+08	1.07E-04	1.60E-04
C2H4Cl2	1,2-Dichloroethane	1.52E+14	1.91E+08	5.77E-04	8.65E-04
CH4	Methane	6.88E+13	2.18E+08	9.69E-03	1.45E-02
CH3Cl	Methyl Chloride	2.87E+11	1.71E+08	6.78E-02	1.02E-01
CH2Cl2	Dichloromethane	4.00E+10	1.75E+08	6.58E-01	9.87E-01
CHCl3	Trichloromethane	1.27E+14	1.93E+08	8.04E-04	1.21E-03
CCl4	Tetrachloromethane	5.02E+10	1.48E+08	6.89E-02	1.03E-01
C10H18	Decahydro-Napthalene*	4.04E+08	1.26E+08	6.67E-04	6.68E-04
C2H3Cl	Vinyl Chloride	1.24E+17	2.65E+08	1.84E-04	2.75E-04

note: K= Ae^(-E/RuT)[Hc]^(m-1)[O2]^n

	m	n
Decahydro-Napthalene	0.25	1.5
All Others	1	1

Table 3 Mole Fractions of Flue Gas at Equilibrium

Temperature	1694 K	1600 K
	mole fraction	*mole fraction*
CO	5.15E-05	1.60E-05
CO2	1.24E-01	1.24E-01
CL	8.43E-06	5.70E-06
HCL	3.44E-04	3.47E-04
H2	1.93E-05	6.78E-06
H2O	1.57E-01	1.58E-01
NO	8.85E-04	6.06E-04
N2	6.94E-01	6.94E-01
O	6.35E-06	2.20E-06
OH	2.58E-04	1.31E-04
O2	2.32E-02	2.34E-02

SITE REMEDIATION TECHNOLOGIES

DRAIN-ENHANCED SOIL FLUSHING (DESF) FOR ORGANIC CONTAMINANTS REMOVAL
METC TASK 1.2

Quarterly Report XVI for Period
July 1 through September 30, 1996

Work Performed Under Contract
No. DE-FC21-92MC29467

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

Principle Investigator:

M.A. Gabr, Ph.D., P.E.

Research Assistants:
Doug Szabo
Mike Sabodish
Alan Williamson

Department of Civil Engineering
West Virginia University
Morgantown, West Virginia 26505-6103
October 1996

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ABSTRACT

The main objective of this research is to investigate major parameters affecting the efficiency of contaminant extraction using PVD systems. The concept aims at enhancing the effectiveness of the soil flushing process by installing the PVD to shorten the drainage path of the flow and provide redundancy and wide coverage to capture the subsurface pollutants. The laboratory phase and pilot scale field test have proven the feasibility of the concept. The objective of this proposed phase is to prove and evaluate the technology performance in the field on a full scale. Hence, the overall cleanup of a chosen site in accordance with acceptable groundwater standards. In addition, a comparison with the conventional vapor extraction system, proposed at the same test site, will be conducted.

Work conducted so far on the project included laboratory tests, pilot scale testing and analytical model development. Laboratory tests included flexible wall permeability tests on soil specimens with and without a PVD installed. Bench-scale tests using large scale reactors, referred to as Smear Cells, were conducted to evaluate the equivalent diameter, long term performance, and zone of influence of the PVDs. In addition, several pilot scale Contaminant Recovery Cells (CRCs) were used to evaluate the flow characteristics of the soil samples and the concept of soil flushing using the PV drains in various installation arrangements. Sodium chloride (NaCl) tracer (100 and 1,000 mg/l) was used for discerning the concept of contaminant recovery using the PVDs system. Soil samples were prepared from sand and a mixture of 80% Ottawa sand and 20% kaolinite. An analytical model for the design of PVD systems for soil remediation was also developed. The field site selection and investigation was done following procurement of a site in Weston, WV. This site is owned by Ashland Petroleum Co. and has LNAPL and DNAPL contamination present in the groundwater. A Pilot Scale field experiment was performed to find air and fluid flowrates per PVD. Also, several grab samples were acquired during testing and tested for BTEX concentrations.

1.0 INTRODUCTION

This project researches the use of Prefabricated Vertical Drain (PVD) systems for remediation of contaminated sites with fine-grained soils. PVD systems are relatively simple in operation and installation. This type of system can be expected to reduce remediation costs while at the same time increase the efficiency of the remediation effort.

Currently, PVDs are used for soil improvement and consolidation. Other than the work performed in the first two phases of this project, no research has been done to test the feasibility of using PVDs for enhanced soil flushing.

2.0 PURPOSE

The primary phase accomplished throughout this quarter was the successful performance of the pilot size field scale test. Specific objectives are as follows:

1) Measurement of fluid flowrate extracted to develop upper limits for vacuum pressure to minimize groundwater drawdown.

2) Development of a functional relationship between BTEX concentrations in groundwater extracted with operation time to determine extent of decontamination.

3) Measurement of groundwater levels via monitoring wells for approximation of the effected area during the extraction operation of the pilot scale.

4) Injection process was completed through a reversal of the pilot scale setup. Groundwater levels were also measured to estimate extent of area effected due to the positive pressure applied.

The secondary phase accomplished throughout this quarter deals with a site specific test using the soil collected from Weston, WV. Gradient Ratio tests (ASTM D-5101) were conducted to determine the clogging potential of the geotextile jacket encasing the flow channels.

3.0 BACKGROUND

The PVD system is used to shorten the drainage path of the groundwater flow and therefore promote subsurface liquid movement and expedite the soil flushing process in low permeability soils. Proved technologies exist for the in situ soil flushing for sites with granular soils (hydraulic conductivity approximately 1×10^{-2} cm/s). However in the case of sites where the soil is fine-grained with corresponding hydraulic conductivity ranging 1×10^{-3} to 1×10^{-7} cm/s, implementation of the traditional soil flushing technology is ineffective and cost prohibitive.

Previous work conducted on the project included laboratory tests, pilot scale testing and analytical model development. Laboratory tests included flexible wall permeability tests on soil specimens with and without a PVD installed. Bench-scale tests using large scale reactors, referred to as Smear Cells, were conducted to evaluate the equivalent diameter, long term performance, and zone of influence of the PVDs. In addition, several pilot scale Contaminant Recovery Cells (CRCs) were used to evaluate the flow characteristics of the soil samples and the concept of soil flushing using the PV drains in various installation arrangements. Sodium chloride (NaCl) tracer (100 and 1,000 mg/l) was used for discerning the concept of contaminant recovery using the PVDs system. Soil samples were prepared from sand and a mixture of 80% Ottawa sand and 20% kaolinite. An analytical model for the design of PVD systems for soil remediation was also developed. The field site selection and investigation was done following procurement of a site in Weston, WV. This site is owned by Ashland Petroleum Co. and has LNAPL and DNAPL contamination present in the groundwater. A Pilot Scale field experiment was performed to find air and fluid flowrates per PVD. Also, several grab samples were acquired during testing and tested for BTEX concentrations.

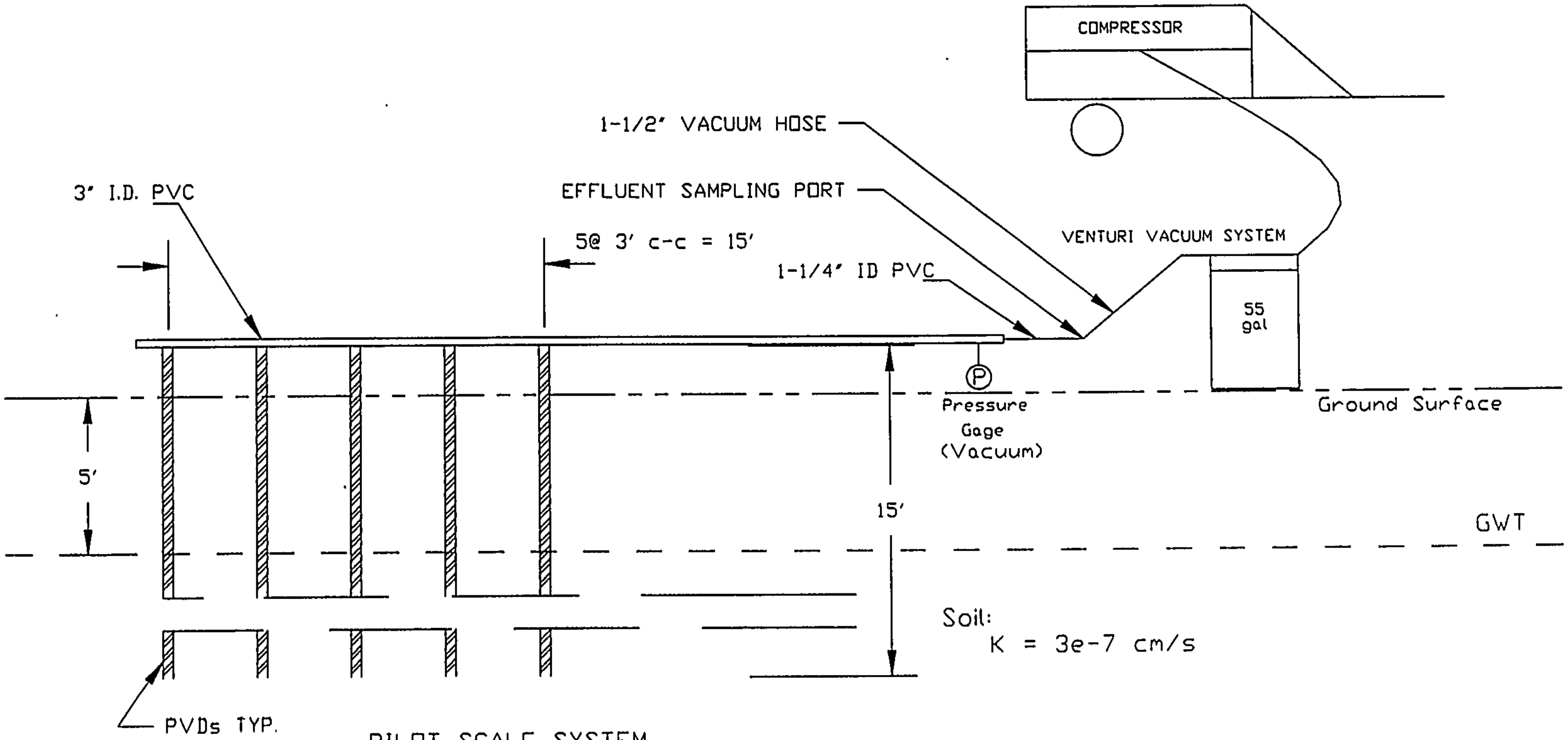
4.0 WORK PERFORMED THIS PERIOD

The pilot scale field test consisted of five (5) PVD drains installed in a linear fashion with a nominal three foot spacing between the wells. The drains were then connected to a collector pipe, consisting of 3 inch inside diameter PVC pipe. To complete the PVD grid prototype setup, a vacuum system was adjoined to one end of the collector pipe. Refer to Figure 1 for the schematic representation of the pilot scale field test. The vacuum system, a Venturi Vacuum System, employs a standard 55-gallon drum (DOT 17C) along with compressed air to generate the vacuum or negative pressure required to operate the extraction component for the DESF technology.

Groundwater levels measured via monitoring wells were taken prior to activation of the vacuum system and also during the operation of the pilot scale. Figure 2 shows the placement of the pilot scale at the Weston site along with the location of the monitoring wells incorporated for data retrieval. A water level indicator was employed to determine the depth of the water level in each well and the depth to the free product level if applicable to a well. The free product is lighter than water and floats above the water level. The general depth to the liquid surface in each well was relatively small and easily visible with the aid of a flashlight.

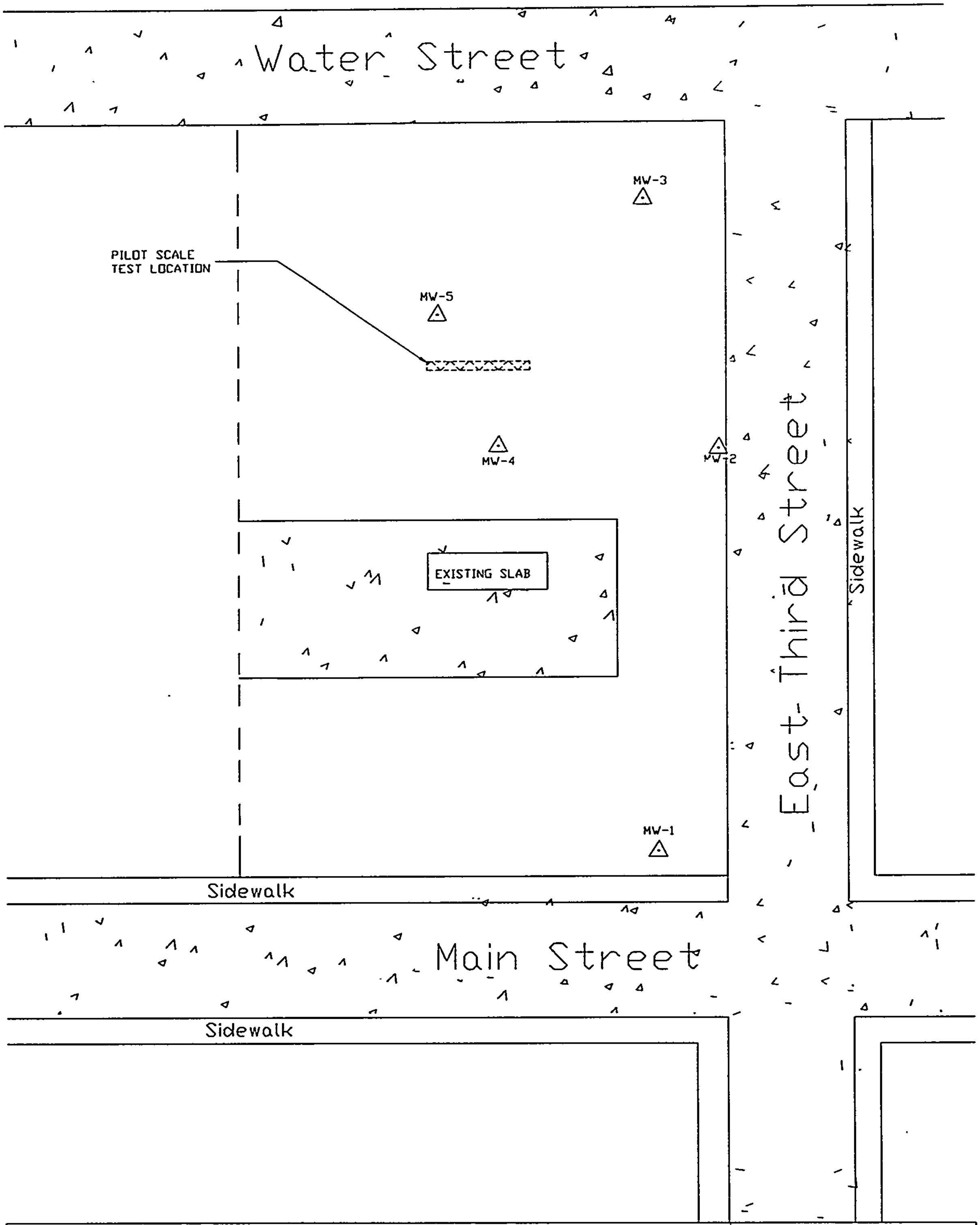
Fluid flowrates for the extracted effluent were determined by measuring initial and final depths of effluent in the 55-gallon drums over a known period of time. Calculating the volume, the flowrate for each time interval could be determined. These time intervals coincided with the retrieval of grab samples taken from the collector pipe.

The grab samples were kept at an approximate temperature of 0°C to prohibit sample decay. The samples were then shipped to Ashland's R&D Lab via United Parcel Service (UPS). The R&D



PILOT SCALE SYSTEM

Figure 1
Note: drawing not to scale



ASHLAND BRANDED MARKETING, INC.
FACILITY #289-020 257 Main Street
Weston, West Virginia

FIGURE 2
DESF PILOT SCALE
Scale: 1" = 30'

West Virginia University
Department of Civil and Environmental Engineering
P.O. Box 6103
Morgantown, West Virginia 26506

Drafted by:
Alan Williamson 8/13/96

Lab tested the grab samples for Benzene, Toluene, Ethyl Benzene, Xylene (BTEX) concentrations in the groundwater extracted.

Injection was completed by reversing the system and applying a positive head pressure into the collector pipe and through the PVDs. The prototype injection consisted of the compressor and a 55-gallon drum fitted with a cover designed to receive piping consistent with the connections to the compressor and collector pipe. The drum was filled with 45 gallons of water to be used as the injected solution. An approximate pressure of 5 psi was applied for the injection portion of the pilot scale field test. A total time of 15 minutes elapsed and the 45 gallons of water had been injected through the PVDs. Only 4 PVDs were used for injection because one had been previously removed from its well after the extraction process to observe possible clogging in the geotextile and flow channels.

The PVD has a geotextile jacket surrounding the flow channels to prevent piping of soil into the channels. Gradient ratio (ASTM D5101) tests were performed to determine the clogging potential of the soil-geotextile interface. Soil collected from the site and the geotextile surrounding the PVDs chosen for the Weston site were used for the gradient ratios.

5.0 RESULTS AND DISCUSSION

BTEX Concentrations: The operation schedule of the extraction process covered two days of run time. Day 1 elapsed six hours and then a shut down period until 8:30 A.M. on Day 2. Day 2 lasted eight hours. Figure 3a shows the relationship between BTEX concentrations of the grab samples taken versus cumulative time, which includes the shut down period. During Day 1, the concentrations increased with time linearly with time until shut down for Day 1. As can be seen in Figure 3a, the concentrations for Day 2 started significantly higher than the ending concentrations of Day 1. However, in contrast to Day 1, the concentrations proceeded to decrease linearly during the operation time of Day 2. Figure b displays the flowrate versus cumulative time which coincides with the grab sampling time. For the sampling times shown, the flowrate calculated is an average over one hour. As can be seen in Figure b, the flowrates calculated remained relatively consistent throughout the two days of operation. This flowrate was controlled from the vacuum pressure applied to overcome the elevation head along the length of the PVD. Previous laboratory work on PVD well resistance was used to determine the magnitude of vacuum pressure applied to overcome the elevation head. The length of the PVDs was 15 feet, refer to Figure 1. From the lab scale tests for well resistance, a pressure of approximately 29 kPa(kiloPascals) was needed to overcome an elevation head of 5 meters (~15 feet).

Gradient Ratios: Four gradient ratio tests were set up this quarter to check for clogging potential of the soil-geotextile interface specific to the soil at the Weston site and the geotextile shroud around the PVDs chosen for the DESF project. These tests are referred to as Cell 1, 2, 3, and 4 respectively. Figure 4 and Figure 5 show graphical results from Cells 2 and 4 respectively. These two cells showed two different aspects of theorized results expected from the gradient

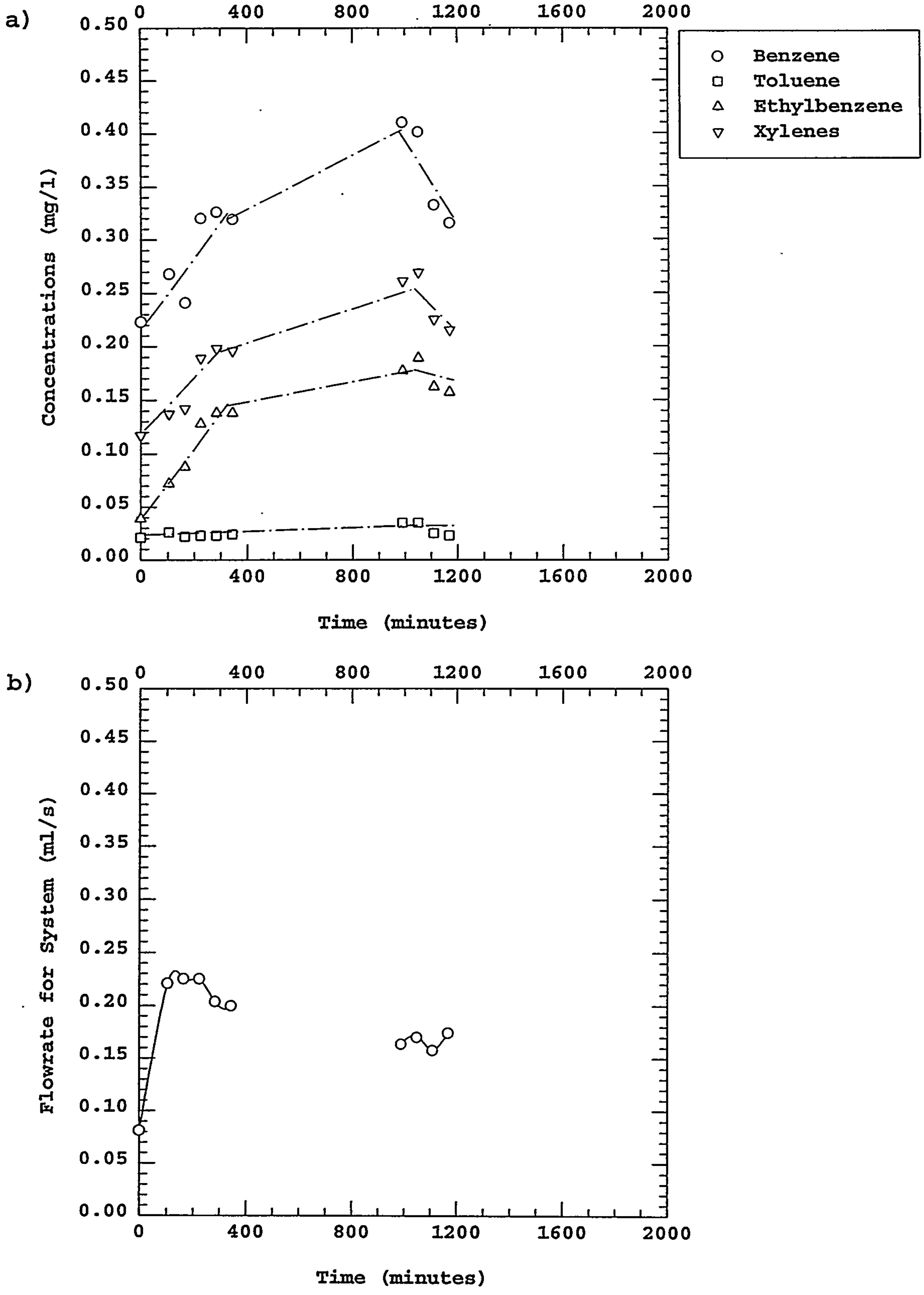
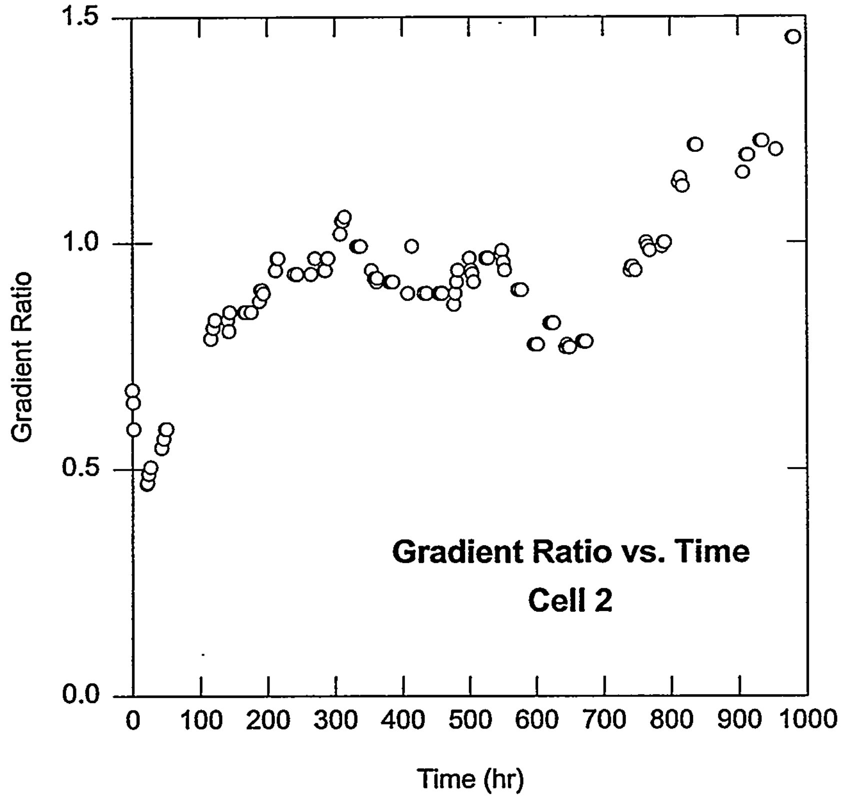


Figure #3 Results From Pilot Scale Field Test Conducted on Ashland Facility in Weston, WV
a) BTEX Concentrations for Pilot Scale Test
b) Flowrate from System versus Time



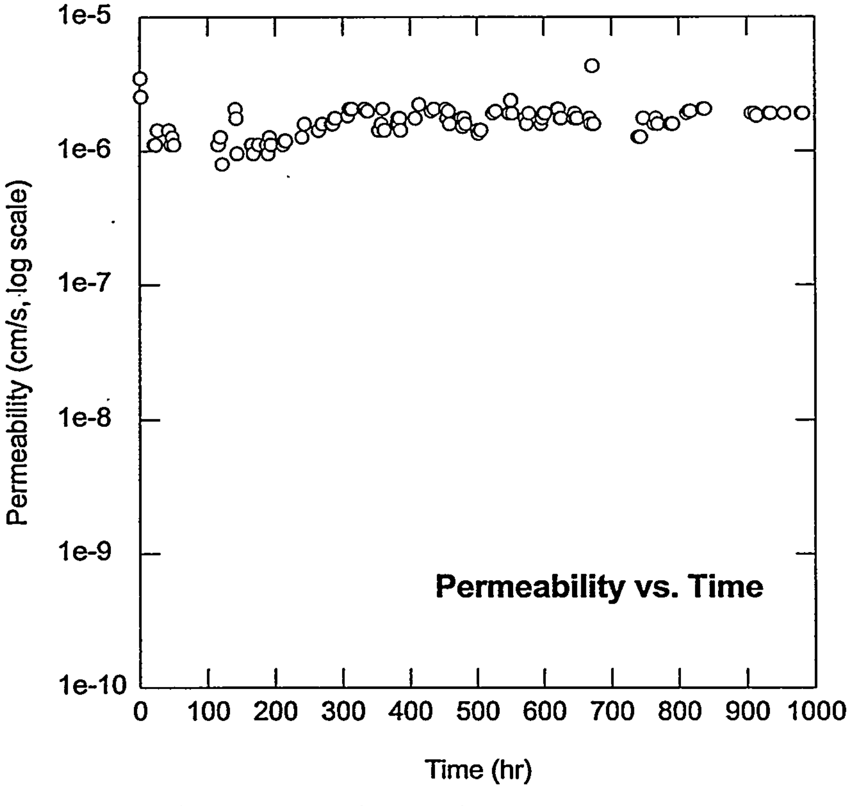
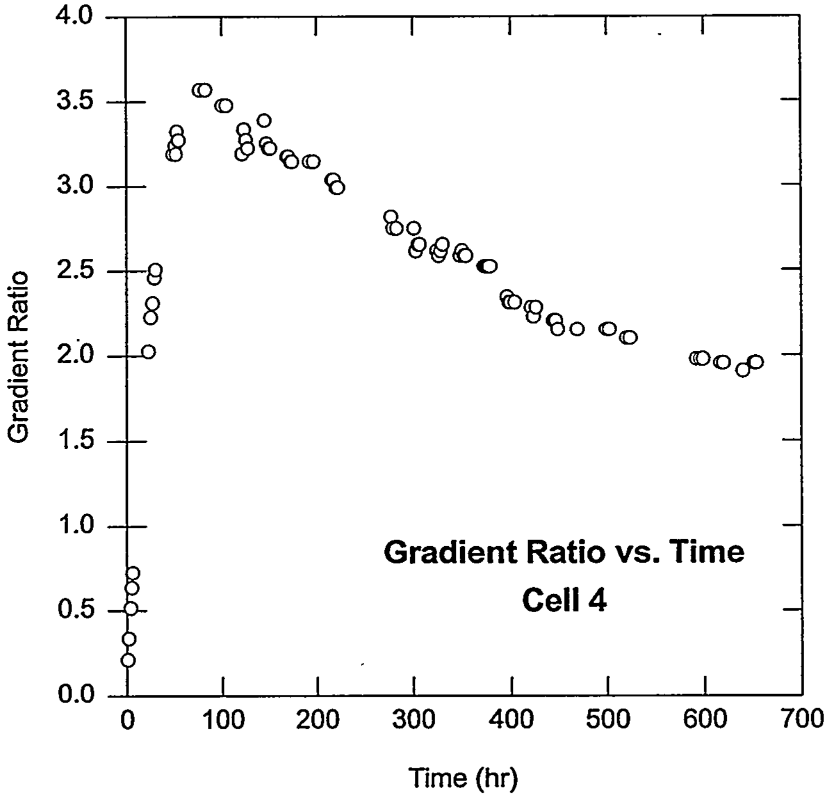


Figure 4 : Gradient Ratio Test for Cell 2
a) Gradient Ratio versus Time
b) Permeability versus Time



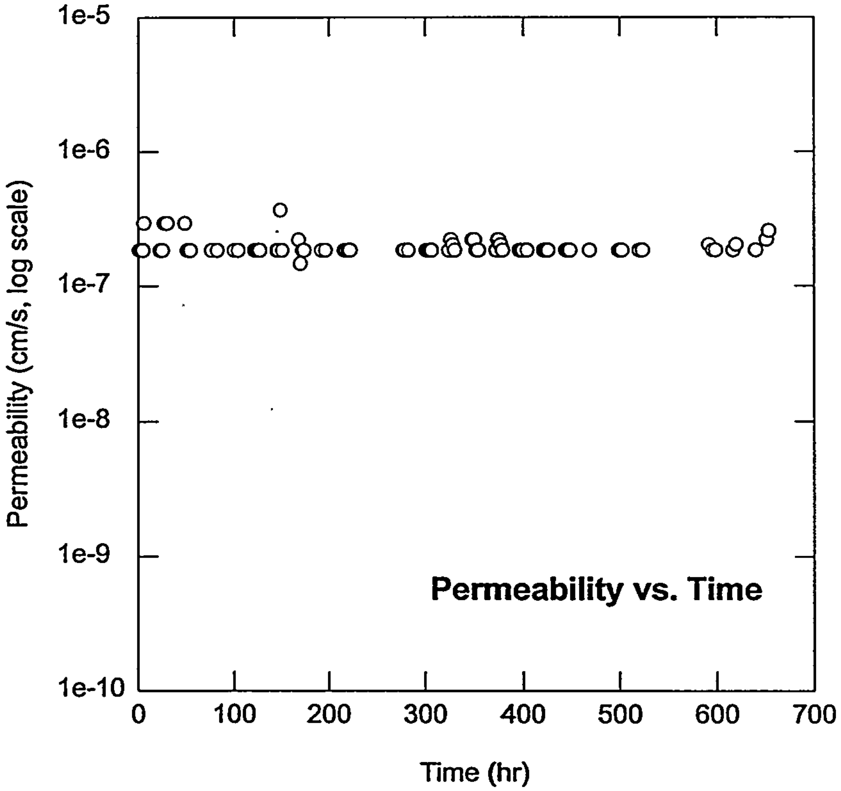


Figure 5 : Gradient Ratio Test for Cell 4
a) Gradient Ratio versus Time
b) Permeability versus Time

ratio test. Cells 1 and 3 were inconclusive due to leaking from equipment failure and hence caused a head loss in the cell and therefore were eliminated. From Figures 4 and 5, it can be seen that the total time for Cell 2 is significantly longer than Cell 4. However, these two tests displayed different results with respective cumulative times. The peaks in the Gradient Ratio versus Time curve in Figure 4a signify a point when the geotextile became clogged with soil and therefore creating a increase in the hydraulic gradient in the soil column. Refer to ASTM D5101 in the appendix for methodology of the gradient ratio test. In contrast, Cell 4 shows an initial peak or clogging at the start of the test and then a constant decrease in the gradient ratio to a value of 2.0. Also shown in Figures 4 and 5 is the Permeability versus Time for Cells 2 and 4 respectively. The permeability for each cell remained constant and the difference of the average permeabilities between each cell is very small.

6.0 CONCLUSIONS

The results from operating the pilot scale field test verify that contaminated ground water can be substantially retrieved with respect to the size of the pilot scale. The data obtained creates a good bridge for the jump to the full scale implementation. Decisions such as drain spacing can be altered for site specific qualities.

7.0 WORK PLANNED FOR NEXT PERIOD

Tasks to be performed for next reporting period include further testing with the gradient ratios, which in contrast to the first cells, will have compacted soil placed in the soil column. Permeability tests will be performed on clogged and unclogged geotextiles along with the possibility of obtaining electron microscope photographs of a clogged geotextile.

Advancements for the decision to use a surfactant are in process. This testing follows a procedure of contaminating soil collected from Weston with a main constituent of BTEX found at Weston. Proposal to use the surfactant Tergitol, specifically the clone TMN-10, will be studied along with water to determine effectiveness as a flushing solution. A gas chromatograph will be implemented to evaluate concentrations of the contaminant before and after a flushing solution is used to quantitatively determine the amount of decontamination in the selected soil samples.

APPENDIX

Designation: D 5101 – 90

Standard Test Method for
Measuring the Soil-Geotextile System Clogging Potential by the Gradient Ratio[1]

1. Scope

1.1 This test method is a performance test applicable for determining the soil-geotextile system permeability and clogging behavior under unidirectional flow conditions.

1.2 The values stated in SI units are to be regarded as standard. The values in parentheses are for information only.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[2]
D 653 Terminology Relating to Soil and Rock[3]
D 737 Test Methods for Air Permeability of Textiles[4]
D 4354 Practice for Sampling of Geotextiles for Testing[3]
D 4439 Terminology Relating to Geotextiles[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *clogging potential, n—in geotextiles,* the tendency for a given geotextile to decrease permeability due to soil particles that have either lodged in the geotextile openings or have built up a restrictive layer on the surface of the geotextile.

3.1.2 *geotextile, n*—any permeable textile material used with foundation, soil, rock, earth, or any other geotechnical engineering related material, as an integral part of man-made product, structure, or system.

3.1.3 *gradient ratio, n—in geotextiles,* the ratio of the hydraulic gradient through a soil-geotextile system to the hydraulic gradient through the soil alone.

3.1.4 *hydraulic gradient, i, s* (D)—the loss of hydraulic head per unit distance of flow, dH/dL.

3.1.5 For definitions of other textile terms, refer to Terminology D 123. For definitions of other terms related to geotextiles, refer to Terminology D 4439 and Terminology D 653.

3.2 *Symbols and Acronyms:*

3.2.1 *CO_2*—the chemical formula for carbon dioxide gas.

3.2.2 *CHD*—the acronym for constant head device.

4. Summary of Test Method

4.1 This test method requires setting up a cylindric clear plastic permeameter (see Figs. 1 and 2) with a geotexti and soil, and passing water through this system by applyi various differential heads. Measurements of differenti heads and flow rates are taken at different time intervals determine hydraulic gradients. The following test procedu describes equipment needed, the testing procedures, a calculations.

5. Significance and Use

5.1 This test method is recommended for evaluating t performance of various soil-geotextile systems under co trolled test conditions. Gradient ratio values obtained m be used as an indication of the soil-geotextile system cloggi potential and permeability. This test method is not appr priate for initial comparison or acceptance testing of vario geotextiles. The test is intended to evaluate geotextile pe formance with specific on-site soils. It is improper to utili the test results for job specifications or manufacturer certifications.

5.2 It is important to note the changes in gradient rat values with time versus the different system hydraul gradients, and the changes in the rate of flow through t system (see Section 11).

NOTE 1—A gradient ratio of approximately one (1.0) indicates th the geotextile has no effect on the hydraulic flow through the so geotextile system and that the soil is internally stable. A gradient ratio <1.0 may indicate internal instability of the soil with some of tho particles adjacent to the geotextile moving out of the system. It may al indicate clogging or restriction at the manometer ports due to a bubbles or algae. A gradient ratio >1.0 indicates system clogging restriction at or near the surface of the geotextile. The allowable gradie ratio value for various soil-geotextile systems will be dependent on t specific application. It is the responsibility of the design professional establish this allowable value on a case-by-case basis.

6. Apparatus

6.1 *Soil-Geotextile Permeameter,* (three-piece uni equipped with support stand, soil-geotextile support scree piping barriers (caulk), clamping brackets, and plastic tubi (see Fig. 2).

6.2 *Two Constant Water Head Devices,* one mounted o a jack stand (adjustable) and one stationary (Fig. 3).

6.3 *Soil Leveling Device* (Fig. 4).

6.4 *Manometer Board,* of parallel glass tubes and me suring rulers.

6.5 *Two Soil Support Screens,* of approximately 5 m (No. 4) mesh.

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[1] This test method is under the jurisdiction of ASTM Committee D-35 on Geosynthetics and is the direct responsibility of Subcommittee D35.03 on Permeability and Filtration.

Current edition approved July 27, 1990. Published October 1990.

[2] *Annual Book of ASTM Standards,* Vols 07.01 and 07.02.

[3] *Annual Book of ASTM Standards,* Vol 04.08.

[4] *Annual Book of ASTM Standards,* Vol 07.01.

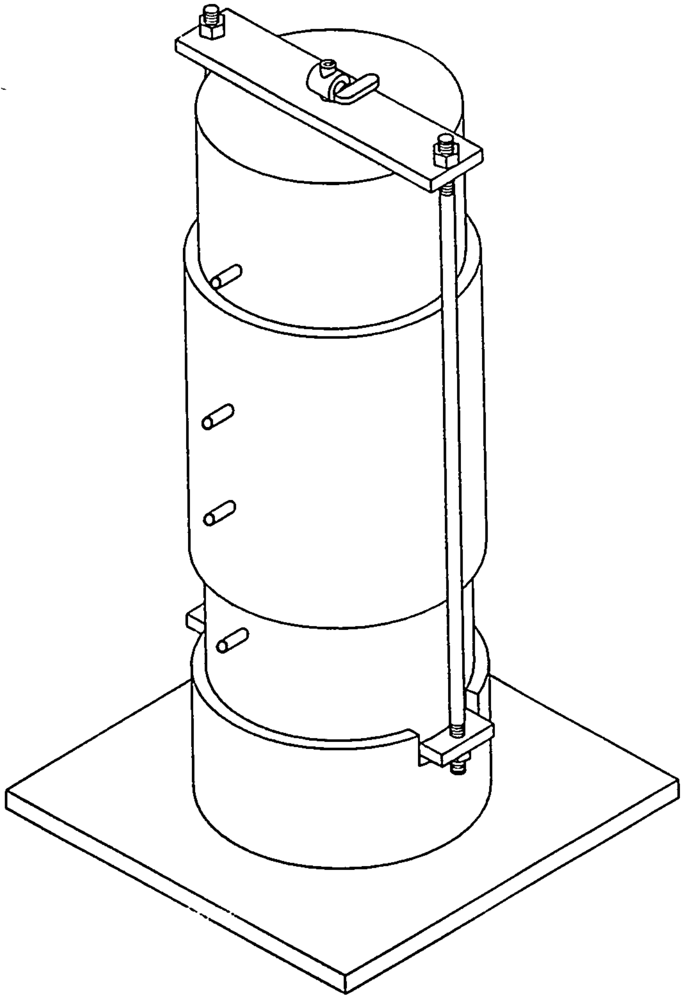


FIG. 1 Geotextile Permeameter

6.6 *Soil Support Cloth*, of 150 μm (No. 100) mesh, or equivalent geotextile.

6.7 *Thermometer* (0 to 50°C ± 1°C).

6.8 *Graduated Cylinder*, 100 cm^3 capacity.

6.9 *Stop Watch.*

6.10 *Balance*, or scale of at least 2-kg capacity and accurate to ±1 g.

6.11 *Carbon Dioxide*, (CO_2), gas supply and regulator.

6.12 *Geotextile.*

6.13 *Water Recirculation System.*

6.14 *Water Deairing System*, with a capacity of approximately 1700 L/day (500 gal/day).

6.15 *Algae Inhibitor*, or micro screen.

6.16 *A 150 μm Mesh Screen, (No. 100)*, or equivalent geotextile for manometer ports.

6.17 *Soil Sample Splitter (optional).*

6.18 *Pan*, for drying soil.

6.19 *Mortar and Pestle*, for pulverizing soil.

6.20 *Wooden rod*, 20 mm (¾ in.) diameter by 150 mm (6 in.) long.

7. Sampling and Test Specimens

7.1 *Lot Sample and Laboratory Sample*—Take a lot sample and laboratory samples as directed in Practice D 4354. For laboratory samples, take a full width swatch of geotextile from each roll of material in the lot sample at least 1 m (3 ft) long cut from the end of the roll after discarding the first meter of material from the outside of the roll.

7.2 *Test Specimens*—Cut three circular specimens from each swatch in the laboratory sample with each specimen having a diameter of 110 mm (4.33 in.). Locate two specimens no less than 300 mm (11.8 in.) from each edge of the swatch and one at the center of the swatch width.

8. Conditioning

8.1 *Test Water Preparation:*

8.1.1 Test water should be maintained at room temperature about 16 to 27°C (60 to 80°F), and deaired to a dissolved oxygen content of 6 parts per million (ppm) or less before introducing it to permeameter system. This will reduce or eliminate the problems associated with air bubbles forming within the test apparatus.

8.1.2 An algae inhibitor or micro screen should be used to eliminate any algae buildup in the system.

8.2 *Specimen Conditions:*

8.2.1 Condition the specimen by soaking it in a container of deaired water for a period of 2 h. Dry the surface of the specimen by blotting prior to inserting in the permeameter.

9. Procedure

9.1 *Preparation of Apparatus:*

9.1.1 Thoroughly clean and dry permeameter sections.

9.1.2 Close all valves and cover the inside openings of all manometer ports with fine wire mesh or lightweight nonwoven fabric (the equivalent of No. 100 mesh).

9.1.3 Lubricate all O-ring gaskets.

9.2 *Permeameter Preassembly:*

9.2.1 Stand center section of the permeameter on end and place a soil support cloth 110 mm (4.33 in.) in diameter on recessed permeameter flanges.

9.2.2 Insert support screen 110 mm (4.33 in.) diameter on top of support cloth with mesh side against the cloth.

9.2.3 Align and insert top section of the permeameter into center section and press until there is a tight fit to secure the support cloth and screen in place. Assure that all gasket edges secure against the support cloth, support bracket, and between the center and top permeameter sections.

9.2.4 Invert and place permeameter into holding stand.

9.3 *Process Soil:*

9.3.1 Thoroughly air dry the soil sample as received from the field. This shall be done for a minimum of three days. Pulverize the sample in a mortar with a rubber-tipped pestle (or in some other way that does not cause breakdown of individual grains), to reduce the particle size to a maximum of 10 mm (⅜ in.). Select a representative sample of the amount required (approximately 1350 g) to perform the test by the method of quartering or by the use of a soil splitter.

9.3.2 Select that portion of the air-dried sample selected for purpose of tests and record the mass as the mass of the total test sample uncorrected for hygroscopic moisture. Separate the test sample by sieving with a 2-mm (No. 10) sieve. Pulverize that fraction retained on the 2-mm (No. 10) sieve in a mortar with a rubber-covered pestle until the aggregations of soil particles are broken up into the separate grains.

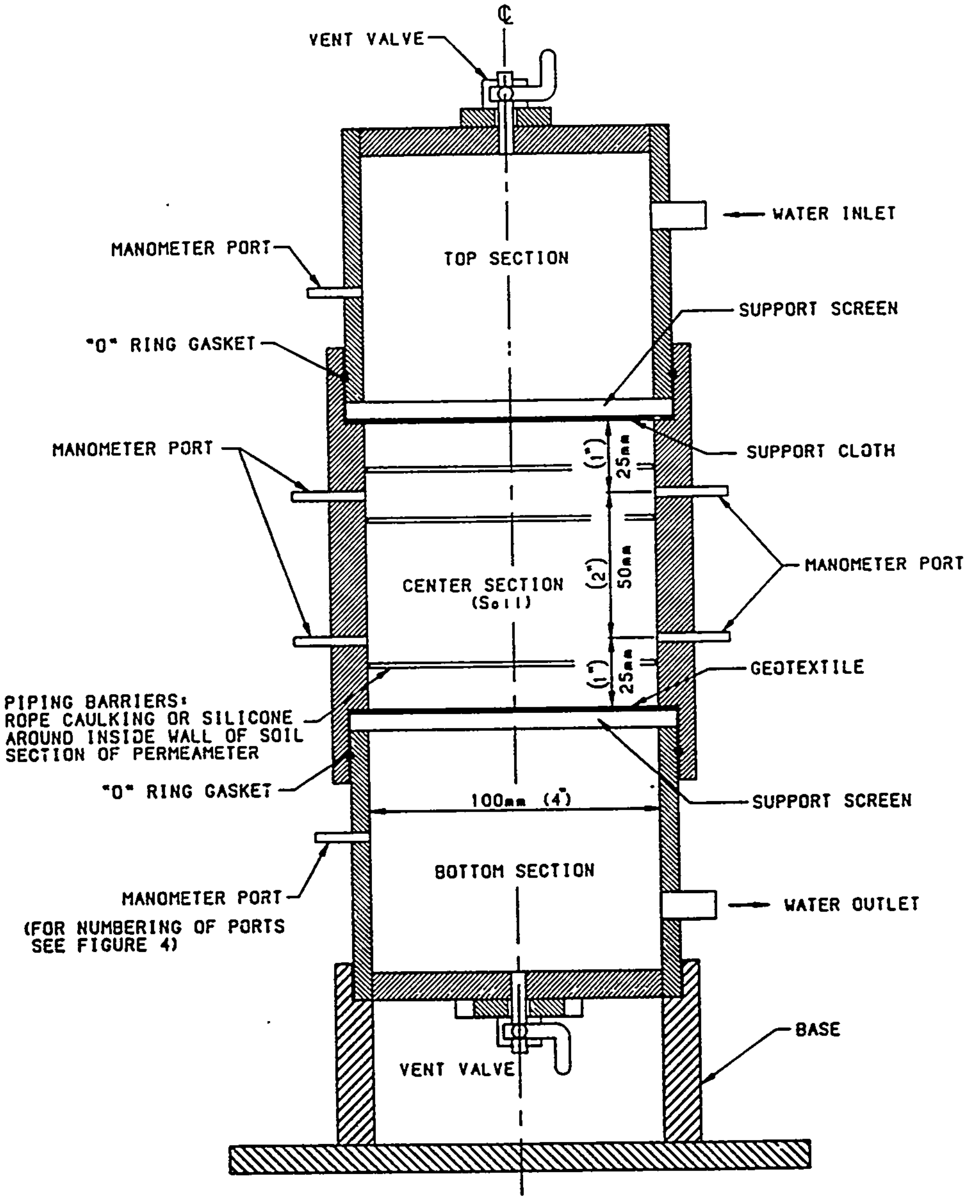


FIG. 2 Section—Geotextile Permeameter

9.3.3 Mix the fractions passing the 2-mm (No. 10) sieve along with the portion that was retained on the 2-mm (No. 10) sieve to form the test soil. All particles larger than 10 mm (3/8 in.) should be eliminated.

9.4 *Soil Placement:*

9.4.1 Weigh out approximately 1350 g of air dried processed soil.

9.4.2 Place air dried processed soil above the support cloth to a depth of 110 mm (4.33 in.). The final depth of soil after settlement will be approximately 100 mm (4 in.). The soil should be placed in 25 mm (1-in.) to 40-mm (1½-in.) layers, making sure that no voids exist along the permeameter walls at manometer ports, or the caulk piping barriers. The soil shall be placed carefully into the permeameter with a scoop or appropriate tool with a maximum drop of the soil no greater than 25 mm (1 in.). Consolidation of each layer shall consist of tapping the side of the permeameter six times with a 20 mm (3/4 in.) diameter by 150 mm (6 in.) long wooden rod.

9.4.3 When the level of the soil in the permeameter reaches a depth of 100 mm (4 in.), insert the soil leveling device (Fig. 4), with the notch down, on the top edges of the permeameter. Continue placing soil and rotating the leveling device until the total soil height of 110 mm (4.33 in.) is reached.

9.4.4 Remove the soil leveler and any excess soil. Determine the mass of the soil in the permeameter for unit weight calculations.

NOTE 2—The specified soil placement procedure results in a relatively loose soil condition and is conservative for many applications. If a density approximating actual field soil conditions is desirable, the test could be run at this specified soil density. It should be recognized, however, that predicting field soil conditions may be very difficult due to construction installation procedures that generally disturb and loosen soils adjacent to the geotextile.

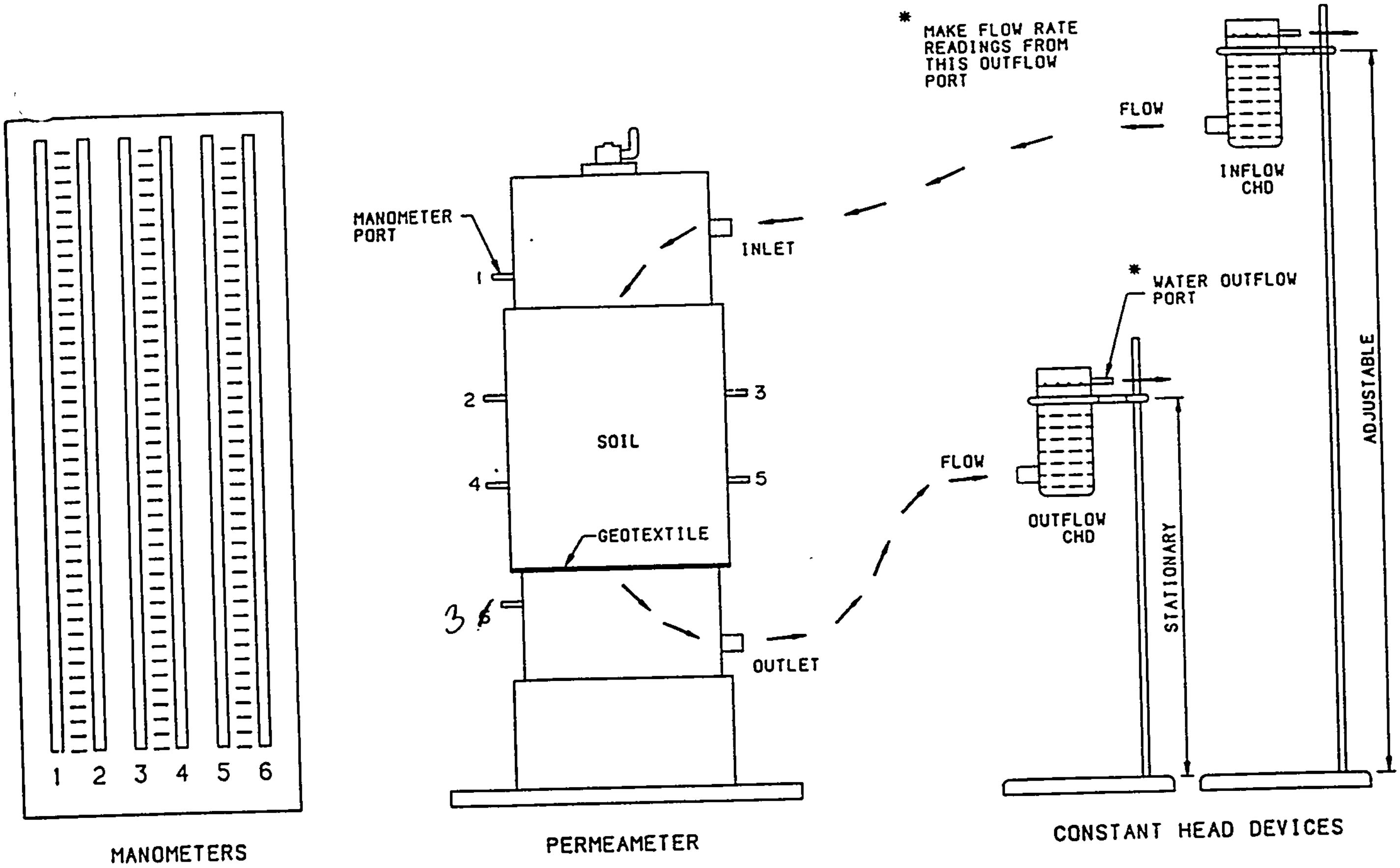


FIG. 3 Geotextile Permeameter "Set Up" Diagram

9.5 *Permeameter Assembly and Setup:*

9.5.1 Clean the inner flange of the center section of the permeameter and insert the geotextile to be tested.

9.5.2 Insert support screen on top of geotextile with the mesh side against the geotextile.

9.5.3 Align and insert the bottom section of the permeameter into the center section and press tightly to secure the geotextile and support screen. The soil will compress from 110 mm (4.33 in.) to approximately 100 mm (4 in.) when the bottom section is secured. Check gaskets to assure contact is made between permeameter sections, support screen, and geotextile.

9.5.4 Secure the permeameter sections together within clamp brackets and tighten bolts on bracket rods evenly.

9.5.5 Invert permeameter into holding stand so that the geotextile will be below the soil level.

9.5.6 Connect the inflow and outflow constant head devices (CHD) to their corresponding permeameter ports (see Fig. 3) with plastic tubing. The outflow CHD is attached to the bottom permeameter port and inflow CHD is attached to the top permeameter port.

9.5.7 Connect all manometer tubes (1 through 5) to their corresponding permeameter manometer ports, and all overflow tubes to their corresponding outlet ports.

9.6 *Saturating the Soil/Geotextile System:*

9.6.1 Open the top vent valve, and close off the permeameter water outlet hose.

9.6.2 Backfill permeameter with water through the outflow CHD until the water level is approximately 10 mm (3/8 in.) below the open manometer port 6. Stop waterflow into the permeameter by clamping off the hose between outflow CHD and permeameter.

9.6.3 Expel oxygen and other gases in permeameter and soil system by (*1*) attaching a carbon dioxide (CO_2) line to manometer port 6, and (*2*) regulating the gas flow at 2 L/min and purging the system for 5 min.

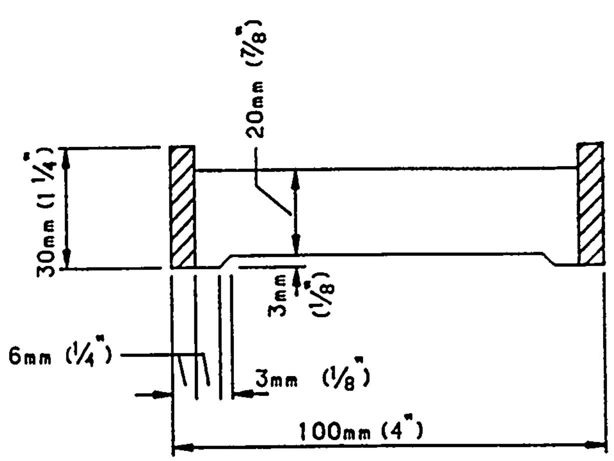
NOTE 3—The permeameter may be backfilled without purging with CO_2, however, the potential for air pockets within the soil to cause erratic results for flow and pressure measurements will be greater without the purging.

9.6.4 After 5 min of gas saturation, seal off (plug) the open end of each manometer tube (1 through 5) and continue to purge the system with CO_2 for an additional 5 min with only the top vent valve open.

9.6.5 Remove the CO_2 gas line and replace the No. 6 manometer hose. Remove the seals (plugs or clamps) from all manometer tubes (1 through 5).

9.6.6 Loosen hose clamp between outflow CHD and permeameter, and fill soil section of permeameter with water. Filling is accomplished by adding water to and raising the level on outflow CHD slowly. Start with outflow CHD at 25 mm (1 in.) above the geotextile level and raise 25 mm (1 in.) every 30 min until water level is 50 mm (2 in.) above the top support screen bracket. This slow saturating process is necessary to prevent air pockets or internal soil movement during loading.

9.6.7 Clamp hose between outflow CHD and permeameter to prevent flow. Continue to raise the water level in the



SECTION A-A

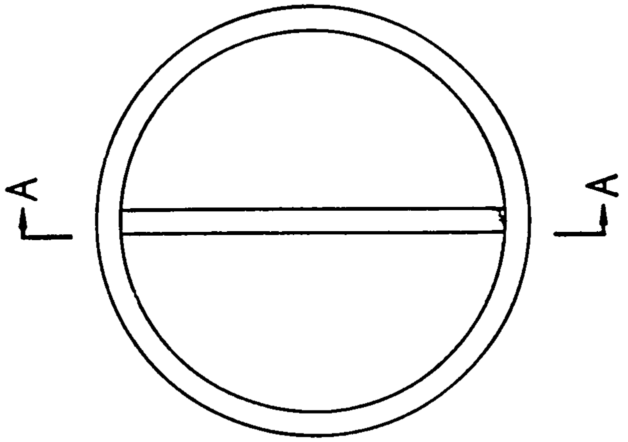


FIG. 4 Plan—Soil Leveling Tool

permeameter by filling from the top inlet through the inflow CHD. The outflow CHD should be clamped so that no flow occurs through the system. The water level should be raised until water flows from top vent valve. Position outflow CHD so that its overflow outlet is approximately 25 mm (1 in.) above the permeameter soil level. The system should be in no-flow condition and the manometers should all read the same.

9.6.8 Close off top vent valve and allow the system to stand overnight in a static condition. This should ensure complete saturation of the system with water. The system should be in a no-flow condition overnight.

9.6.9 Check for and remove air bubbles found in the tubes or manometers by light vibration or tapping. It may be necessary to disconnect tubing from the manometer board and slowly lower the tubing, allowing water and entrapped air to run out.

9.6.10 Place a thermometer into the inflow CHD to monitor temperature of water flowing into permeameter.

9.7 *Running the Test:*

9.7.1 Check to make sure that all scales on the manometer board are set to a common reference elevation.

9.7.2 Adjust the inflow CHD to a level so that a hydraulic gradient (i) of 1 is obtained (see 10.1).

9.7.3 Unclamp hoses between the permeameter and CHD's to allow flow, and record the initial starting time.

9.7.4 Record the following data (using Fig. 5) at 0, ½, 1, 2, 4, 6, and 24 h from the initial starting time:

9.7.4.1 The time in hours (accumulated).

9.7.4.2 The flow rate from the system (outflow CHD); time (t) in seconds for a measured quantity of flow (Q) in cubic centimetres. Measure for a minimum duration of 30 s and a minimum quantity of flow of 10 cm^3.

9.7.4.3 The temperature (T) in degrees celsius of the water in the system.

9.7.4.4 The water level readings from the individual manometers.

9.7.4.5 The date and time of day.

9.7.5 After the 24-h reading, raise the inflow CHD to obtain a system hydraulic gradient (i) = 2.5. Record time. After ½ h at this level, record all data.

9.7.6 Raise the inflow CHD to obtain i = 5. Repeat measurements as in 9.7.4.

9.7.7 After 24-h reading, raise the inflow CHD to obtain i = 7.5. Record time. After ½ h, record all data.

9.7.8 Raise the inflow CHD to a level to obtain i = 10. Repeat measurements as in 9.7.4.

NOTE 4—This test can be run at hydraulic gradients other than those specified in this procedure, for example, $i = 3$ for 24 h. In all cases, the system hydraulic gradient should be increased gradually and in increments no greater than $i = 2.5$ and maintain those incremented levels for a minimum of 30 min. The test may also be run at longer intervals than 24 h, until some recognizable equilibrium or stabilization of the system has occurred.

9.7.9 The test must be run continuously. Once the test has started, it cannot be stopped and then resumed.

10. Calculation

10.1 *Hydraulic Gradient*—Calculate the hydraulic gradients for the system i, using Eq 1. Figure 6 shows the meaning of the values in the equation schematically.

$$i = \Delta h / L \tag{1}$$

where:

Δh = difference in manometer readings for soil zone analyzed, manometer 1 minus manometer 6, cm, and

L = length or thickness of soil between manometers being analyzed, cm.

10.2 *System Permeability*—Calculate the system permeability at the temperature of the test and corrected to 20°C using Eqs 2 and 3:

$$k_T = Q/[(iAt)\cdot 100] \tag{2}$$

$$K_T = k_{20}\mu_T/\mu_{20} \tag{3}$$

where:

k_T = system permeability at test temperature, m/s,

k_{20} = system permeability at 20°C, m/s,

GEOTEXTILE I.D. _____ UNIT WT. OF DRY SOIL IN PERMEAMETER _____ SOIL I.D. _____

DATE	TIME OF TEST (hrs.)	i ($\Delta h/L$)	Δh (cm) (1-6)	MANOMETER READINGS (cm)						Δh_s	Δh_{sf}	G.R.	Q (cc)	Flow Time (s)	Temp (°C)	$k_{20°C}$ (m/s)
				1	2	3	4	5	6							

FIG. 5 Gradient Ratio Permeameter Data

Q = quantity of flow measured, cm^3,
i = hydraulic gradient of the system,
A = cross-sectional area of the specimen, cm^2,
t = time for measured quantity of flow, s,
μ_T = water viscosity at temperature of the test, and
μ_{20} = water viscosity at 20°C.

10.3 *Gradient Ratio*—For each hydraulic gradient, report the gradient ratio, *GR*, for the system using Eq 4 and data for a 24-h period. Figure 5 shows the meaning of the values in the equation schematically.

$$GR = (\Delta h_{sf}/L_{sf})/(\Delta h_s/L_s) = L_s\,\Delta h_{sf}/L_{sf}\,\Delta h_s \quad (4)$$

where:

$$\Delta h_s = \frac{(M_2 - M_4) + (M_3 - M_5)}{2}$$

$$\Delta h_{sf} = \frac{(M_4 - M_6) + (M_5 - M_6)}{2}$$

(M_n = the manometer reading, cm, for the manometer numbered n.)
L_s = 5.10 cm (2 in.), and
L_{sf} = 2.55 cm (1 in. + the geotextile thickness) (Test Method for Measuring Thickness of Geotextiles, Geomembranes, and Related Products[5]

Calculate values from two sets of manometers, as shown above, to detect any changes in pressure from one side to the other. If a significant difference exists between manometers, the system should be investigated for air bubbles, algae buildup, plugged manometer tube, or a plugged port.

11. Report

11.1 State that the specimens were tested as directed in Test Method D 5101. Describe the material or product tested and the method of sampling used.

11.2 Report the following information:

11.2.1 Unit weight of dry soil in the permeameter,

11.2.2 All instrument readings, such as flow volume, flow time, temperature, and manometer readings,

11.2.3 System permeability corrected to 20°C,

11.2.4 Gradient ratio for the system,

11.2.5 A plot of the gradient ratio to the nearest 0.1 unit against time (hours) for each hydraulic gradient tested,

11.2.6 A plot of the permeability to three significant digits against time (hours), and

11.2.7 A plot of the gradient ratio versus the system hydraulic gradient (i).

12. Precision and Bias

12.1 *Precision*—Precision of this test method is being established.

12.2 *Bias*—The procedure in Test Method D 5101 for measuring the soil-geotextile system permeability and clogging potential has no bias because the value of the gradient ratio and permeability can be defined only in terms of a test method.

13. Keywords

13.1 clogging potential; gradient ratio; soil-geotextile system

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[5] This document is currently under development and may be obtained by contacting the Committee D-35 staff manager.

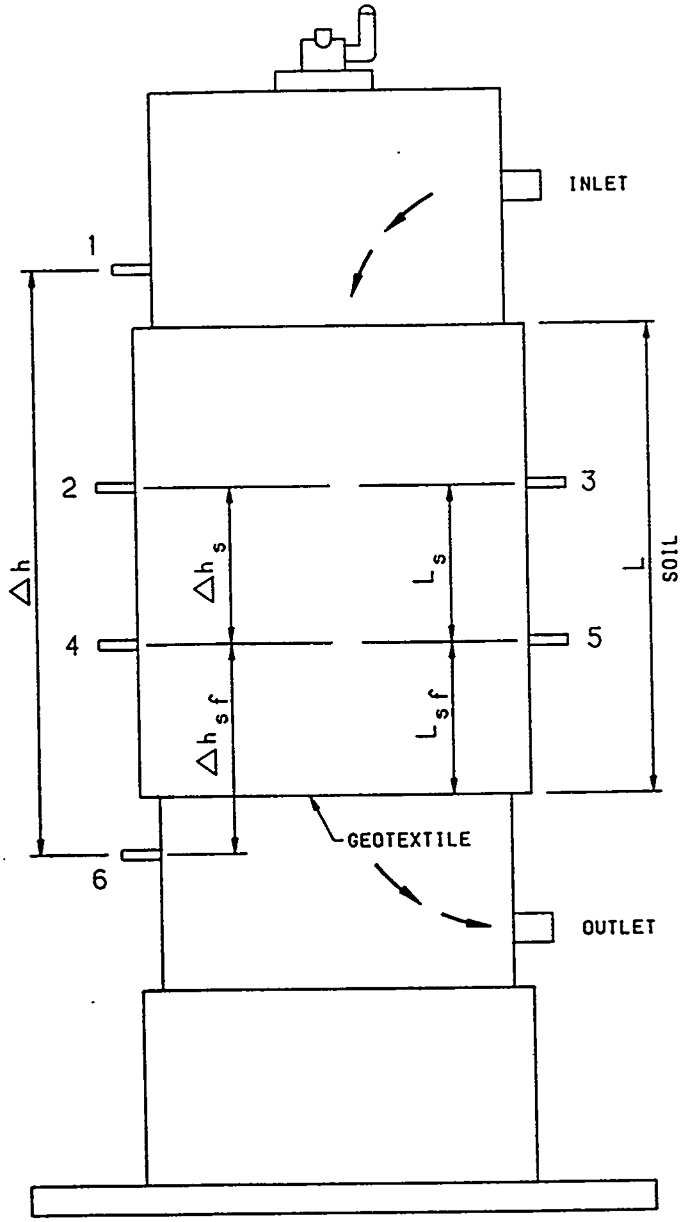

FIG. 6 Values for Gradient Ratio and Hydraulic Gradient Calculations

PERFORMANCE AND CHARACTERISTICS EVALUATION OF ACRYLATES AS GROUT BARRIERS

METC Task No. 1.3
(Chemical Grout Barriers)

PROGRESS REPORT
Reporting Period: June 15, 1996-September 7, 1996

Work Performed Under Contract No.
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For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, WV

Department of Civil and Environmental Engineering
West Virginia University
Morgantown, WV 26506

Evaluation of Acrylate Grouts for Subsurface Containment of Wastes

Abstract: A significant effort in the U.S. and the world is undertaken to develop remediation technologies for contaminated soils and waters. However, in some cases a permanent technology in response to some urgent environmental problems is yet to be developed. For this situation, a temporary barrier may be needed to retard the advancement of the plumes until a permanent remediation technology is implemented. As a result, extensive research is being conducted to evaluate the feasibility of using permeation grouts to develop subsurface containment/confinement systems to retard, or even prevent, the advancement of subsurface contaminant plumes.

A research program was conducted to assess the effectiveness of a urethane (Scotch-Seal (TM) Chemical Grout 5610 Gel manufactured by 3M Company) and a vinyl monomer (Duroseal™ manufactured by Beton-Bau-Zubehor (BBZ) of Germany) grout in controlling the subsurface migration of waste. The study was carried out on the laboratory scale, and was intended to evaluate the chemical grouts ability to create a low-permeability barrier when injected into the soil and develop standard grout evaluation and testing protocols.

The experimental program was implemented in four phases: a) Grout Development, b) Performance Testing, and c) Grout Testing Chamber. The first phase included the evaluation of the grouts' properties and development of viable mix ratios. The second phase of the experimental program aimed at examining the feasibility of injecting the urethane grout into a uniformly graded Ottawa sand (i.e., groutability) in large diameter (29.2 cm), acrylic columns. Samples were collected from the grout bulb and subjected to additional testing which yielded results on the grouted-sands hydraulic conductivity, compressive strength, and durability under permeation and immersion with various classes of chemicals. Phase 3 involves the design and construction of the Grout Testing Chamber (GTC) (essentially a 91.4 cm flexible-wall permeameter) and manufacture of a sand rain device to prepare uniform specimens for the GTC. The first phase of the study resulted in four urethane mixes and one vinyl monomer mix being developed for the next phase. All the urethane mixes were 10:1 (activator:gel) ratios; however, the mix additives varied. The vinyl monomer mix was comprised on 50% water and 50% gel concentrate.

Results from the second phase indicated that two of the urethane grout mixes could be easily injected into the Ottawa sand at its porosity extremes. Additionally, the larger the perforation size and perforation length on the injection probe, the larger the resulting grout bulb and bulb radius. Samples collected from the injected bulbs yielded hydraulic conductivities (k) of 2.4×10^{-7} cm/s with water and significantly increased when using chemical permeants. Grouted-sand samples from both of the grout mixes swelled excessively and decreased in strength when immersed in water, bases, and ethylene glycol solutions. Cupric sulfate, methanol, acetone, and acids appeared to have few observable affects on the samples.

The GTC was designed and is currently under construction. Additionally, a sand rain device was designed and manufactured to produce uniform density specimens for the GTC. It is currently being calibrated.

Within this paper, testing methodologies, data, and results from each phase of the experimental program are presented.

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2.0 EVALUATION OF A URETHANE GROUT FOR CONTAINMENT OF WASTES

3.0 DESIGN AND CONSTRUCTION OF THE SAND RAIN DEVICE

3.8 Technical/Administrative Difficulties

LIST OF FIGURES

1.0 INTRODUCTION

There is a significant effort in the U.S. and the world to develop remediation technologies for contaminated soils and waters. For some contaminated medias, no technologies are feasible or fully developed to meet today's environmental problems. For example, radioactive contaminated soils are impossible to treat with current technologies. Only waste minimization/segregation techniques are applicable which results in a highly concentrated (i.e., radioactive) residual material. Because of the lack of treatment methods, confinement and containment technologies are needed to retard, or even prevent, the advancement of subsurface contaminant plumes into the environment until the development of appropriate remediation technologies are identified and implemented.

There are numerous confinement/containment (C/C) methods available in today's market. Though these techniques are fully capable of preventing lateral migration of contaminants in soil and water, longitudinal migration of contaminants is generally not suppressed or retarded. One method that appears suitable for developing both vertical cut-off walls and/or horizontal bottom barriers is permeation (injection) grouting utilizing chemical grouts which is defined as the process of filling small interstitial voids and fissures in rock or soil (Karol 1990). If permeation grouting is proven to be preventative measure (i.e., secondary containment), as well as a post-release alternative, it could potentially be utilized at numerous sites (Superfund and RCRA) throughout the country.

Because of the potential benefits of this technology and the large number of sites where it could be implemented, research was conducted at West Virginia University to evaluate two chemical grout's effectiveness and applicability as a subsurface barrier. Unlike prior and other current studies, instead of immediately advancing from the lab-scale grout investigation to field scale injection testing, a meso-scale testing program was developed to investigate the material Injectability. Two types of chemical grouts were evaluated for barrier use: Duraseal™ (a vinyl monomer compound from Germany) and Scotch-Seal (TM) Chemical Grout 5610 (a urethane gel product manufactured by 3M). Both grout materials were evaluated on their ability to form in-situ barriers (i.e., injectability/viscosity), toxicity, resistance to chemical degradation, strength properties, and permeability. The research work was divided into the following tasks:

I. Grout Development
II. Performance Testing
III. Grout Testing Chamber (GTC)

Grout Development involves the identification of the grout mix ratio which exhibits the optimum properties for use as a subsurface barrier. Performance Testing utilizes large diameter acrylic columns to investigate the injectability of the grout into Ottawa sand using different grout mixes and varying injection strategies. The structural and hydraulic performance of the formed barrier is also evaluated by performing immersion/strength testing (triaxial and unconfined compression) and permeability testing on samples collected from the grouted-sand bulb. Task III involves the design and construction of the Grout Testing Chamber (GTC), essentially a large-scale flexible wall permeameter with 3 feet diameter specimens which will be capable of simulating in-situ stress conditions. A sub-task was the development of a device and method for preparing specimens of a uniform density which can be accomplished through the use of a sand rain device.

2.0 EVALUATION OF A URETHANE GROUT FOR CONTAINMENT OF WASTES

2.1 Purpose

This research presented in this section was performed to successfully fulfill the requirements of Task I (Grout Development) and Task II (Performance Testing). Only urethane grout is performance is currently being pursued. The objectives of this research program are to establish grout testing protocol, evaluate grout performance, and determine grout characteristics for several injection strategies.

2.2 Background

Although the principles of permeation grouting were developed in the 1940's and numerous companies are in existence which specialize in this practice, none have utilized permeation grouting for waste containment. Past field studies (May et al. 1986, Dwyer 1994, Moridis 1995) conducted to evaluate subsurface barrier characteristics have identified the need for a barrier verification technique. A limited number of research programs (Bodocsi et al. 1988, Bodocsi et. al. 1991, Carson 1988, Heiser et al. 1992, Sherer 1986) have investigated the properties of various types of chemical grouts which make them amenable as a subsurface barrier. At present, there is a considerable amount of research being conducted on this topic at government facilities.

The urethane grout evaluated in this research was supplied by the 3M Company under the trade name Scotch-Seal (TM) Brand Chemical Grout 5610 (Gel). This was chosen after reviewing the product literature and past studies as well as discussing the grout's capabilities with various distributors and manufacturers with research objectives in mind. Furthermore, 3M defines Scotch-Seal (TM) as groutable. The 5610 gel is a hydrophilic material most commonly used for sewer and water line rehabilitation projects.

2.3 Methodology

The experimental program of this investigation was performed to develop a standard testing protocol to evaluate the performance (i.e., durability, hydraulic conductivity, and strength) of permeation grouts. Experimental testing was implemented in two phases: a) Materials Testing Phase and b) Performance Testing Phase. The first phase consisted of testing material properties which included: moisture content (ASTM D 2216), specific gravity (ASTM D 1429), minimum and maximum densities (ASTM D 4254 83 and D 4253 83), and triaxial compression (ASTM D 2850). Additionally, the permeability the material was determined using a rigid wall permeameter conducted in accordance with ASTM Draft Standard Test Method for Measurement of Hydraulic Conductivity Using a Rigid Wall, Compaction-Mold Permeameter. Performance testing examined the injectability (groutability) of the grout in large diameter columns and Post-grout testing (permeability testing, immersion testing, and strength testing).

2.4 Work Performed this Period

Work performed during this period included the following tasks:

1. Viscosity tests on various vinyl monomer mixtures
2. Injection of urethane grout into large diameter (11.5" I.D.) columns
3. Flexible wall permeability tests on urethane grout samples
4. Compaction tests on various ratios of 20/30 sand and kaolinite

During this period the process of optimizing the vinyl monomer grout was initiated. The first acceptance criteria used for the grout was a plot of viscosity versus time. By using a LVT model Brookfield Viscometer characteristics such as initial viscosity and gel time were determined. Once a mix achieved a geltime greater than 15 minutes, it was considered to be acceptable.

Compaction tests were ran to determine the maximum dry density and optimum water contents for various ratios of 20/30 sand and kaolinite.

Flexible wall permeability tests on the urethane grout mixtures were performed using three permeates, water, TCLP#1, TCLP#2.

2.5 Results and Discussion

The results of the flexible wall permeability tests to this date are shown in the Table 1.

Figure 2.1 Results from Flexible Wall Permeability Tests

Gel Time Extender, %	Water, %	Gel Reinforcer, %	Initial Viscosity, cP	Gel Time, s	K, cm/s (water)	K, cm/s (TCLP#1)	K, cm/s (TCLP#2)
100	0	0	212	15	2.0E-9	1.1E-5	
70	30	0	91	17	1.7E-9		
50	50	0	43	21	3.0E-9		
90	9	1	225	15	1.7E-9		
70	29	1	96	16	1.4E-9	1.8E-5	1.2E-5
52	47	1	51.5	17	2.4E-9		
50	48	2	55	16			
55	42	3	45	15	1.6E-9	1.9E-5	
50	45	5	55.5	15	4.6E-9		

Note: The blanks in Table 1 are results from tests that are still in progress

These results are encouraging when water was used as the permeate, but the hydraulic conductivity values when TCLP#1 and TCLP#2 were used are somewhat high.

2.6 Conclusion

At this point in the testing process, it is to soon to make any conclusions about which urethane grout mix is optimal. Once the permeability tests are completed, it will be more apparent which mixes will proceed to the next phase of testing.

2.7 Work Planned for next Period

Urethane Grout

During the next period, three of the urethane grout mixes that yielded the best results from the permeability tests will be chosen. At this point the optimum thickness of the grout will be determined. This will be accomplished by using the flexible wall permeameter and varying the thickness of the sample until there is no infiltration of the chemicals. The next step will be to refine the injection process. This will involve reducing the size of the grout bulb by varying the injection well pattern, hole diameter (in the well pattern), and injection flow rate.

Vinyl Monomer

During the next work period, the vinyl monomer grout will be injected into the large diameter columns. An analogous procedure to the urethane grout will be used, but the soil matrix will also be a variable. Upon curing, samples will be extracted from the grouted soil and flexible wall permeability tests will be preformed.

3.0 SAND RAIN DEVICE

3.1 Purpose

The grout testing chamber (GTC) is to be used to test grout characteristics on a larger or meso-scale to reproduce field conditions. This is to observe the effects of multiple injection wells on barrier formation and grout performance in this environment. Obviously, a large soil sample is needed to execute such an experiment. The sand rain device will be able to deliver such a sample at a uniform and desired relative density.

3.2 Background

Hydraulic conductivity is the soil characteristic that is believed will most affect the grout's performance. Sand was chosen as the soil for grout performance testing since it has a very high hydraulic conductivity and represents the worst case scenario for barrier formation in non-fractured soil. Controlling the value of hydraulic conductivity can be done by fixing the relative density of the test specimen.

Fabricating such sand specimens, especially on a large scale, presents a few obstacles, because guaranteeing a homogenous sample on any scale is difficult without automation. ASTM offers methods for finding the maximum and minimum densities that are adequate for relatively small sand samples, but are both impractical and inaccurate for large scale samples. Therefore, testing grout performance on meso-scale sand specimens dictates the need for a device. This can be done by constructing an apparatus that is able to insure the following in large scale test specimens:

- they must have the requested relative density
- they must have properties that are reproducible independent of the operator
- they must be homogenous

Although such a device is not commercially available, success has recently been realized by a handful of researchers in creating a machine to produce large-scale, homogenous sand samples. The machine they designed is called a sand rain device.

3.3 Methodology

The most important considerations for the design process were cost, durability, and the practical limitations of the technician employed to perform the actual construction. With this in mind sand rain device was designed much like those in the literature. Normally the upper sand reservoir allows flow out through a stopcock and funnel assembly. The sand rain device at WVU has dual perforated plates in place of this assembly to better facilitate dispersion. A scale drawing of the final design can be seen in FIG 3.1.

3.4 Work Performed this Period

Work during this past quarter has been very progressive in the area of sand rain device research. The funnel platform has been completed. A locking mechanism was constructed by

SAND RAIN DEVICE

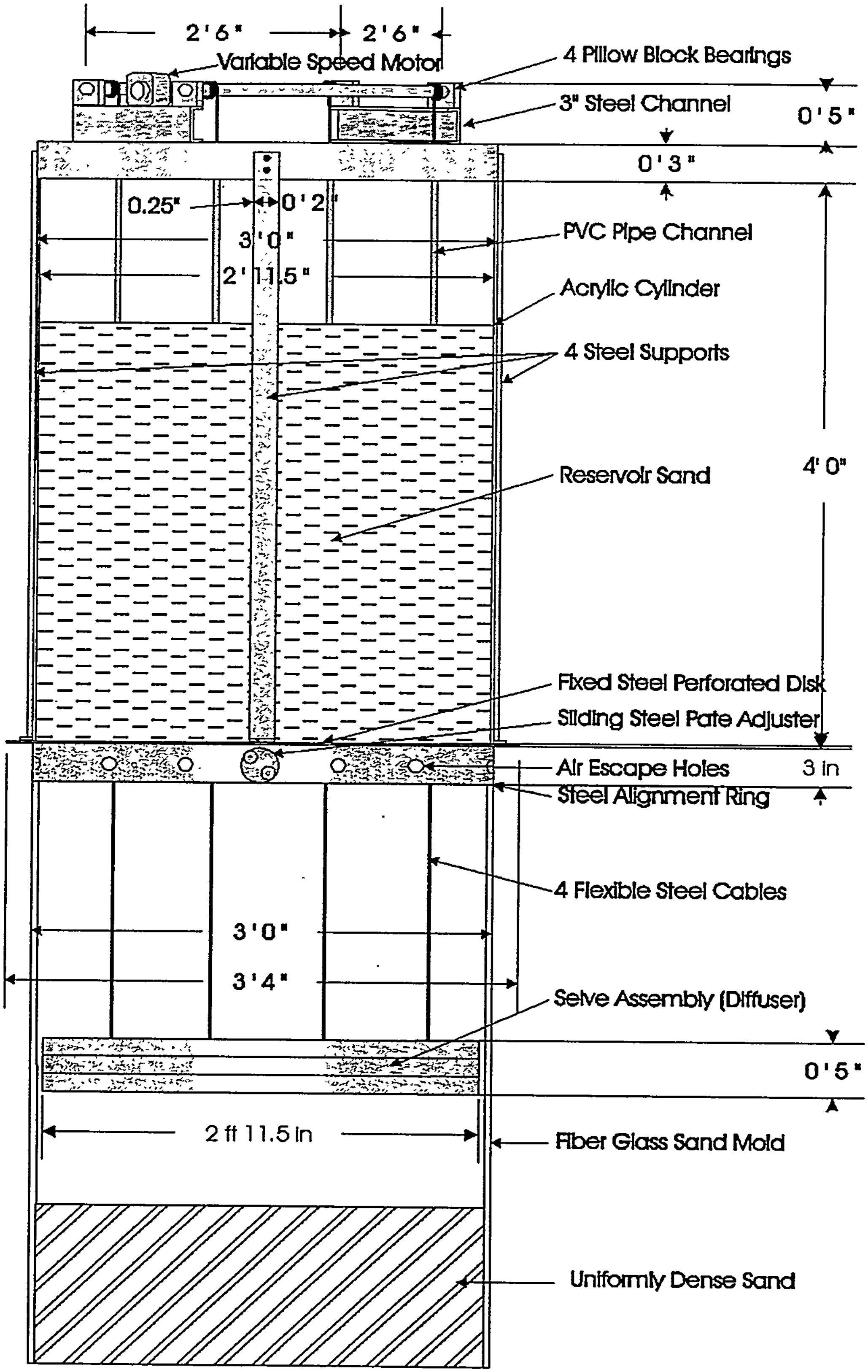

FIG 3.1. Scaled Drawing of the Sand Rain Device

simply holding the two doors open by simple, 4-ton hydraulic jacks like those sold in auto parts stores. The doors are dropped by releasing the pressure in the jacks simultaneously which funnels the sand down and toward the front via a trough. The sand is allowed out by a sliding door so that the sand falls into a 5-gallon bucket placed at the end of the trough which are poured into the top of the sand rain device. With this procedure, testing time is reduced greatly when making full size samples (completely filling the sand mold) than by leaving the mold on the floor. On the floor, a significant amount of time is spent scooping out sand through the top of the mold with small shovels before the mold can be physically lifted to allow the remainder of the sand to pour out onto the floor to be shoveled into buckets and then poured again into the top of the sand rain device. The funnel base nearly eliminates shoveling altogether and significantly reduces loss onto the floor. Furthermore, it keeps the sand cleaner since it never reaches the floor. The funnel base was designed to hold the SRD while full of sand for storage purposes. It is not currently being used as such because it has been more convenient to rest it on two steel I-beams that became available. However, the funnel base is fully capable of holding the sand rain device while full if it became necessary in the future. Finally, the amount of time reloading the SRD has been reduced from three people with shovels taking approximately 1 hour 20 minutes to two people taking requiring less than 30 minutes. If the funnel base were to be placed on some other platform high enough to allow the trough to be above the top of the SRD, refilling the device could be further reduced to about five minutes.

The struts and PVC pipe were installed successfully as projected in the last report. Although the sand streams are still not quite centered over the sample, the strut assemble greatly reduced the diagonal effect mentioned in the last report. It remains to be seen if this is still a problem in the final product, but preliminary results seem to indicate acceptable results.

The speeds required of the electric motor have been narrowed so that the speeds needed can be achieved with the new sprocket design which was simply a matter of increasing the gear ratio to 4 to 1. With the installation of the new sprocket and new, smaller diameter cables for easier winding, the mechanics of raising the sieves at a constant rate are completed.

It became obvious for a need to keep the sand relatively clean because of the obvious need for a homogenous material, but more importantly to keep the sand rain device from clogging when the ports were opened only a small amount. In cases where one port was closed due clogging, the entire sample makeup was altered. Therefor, a large square sieve made from 2x4s, plastic reinforcing mesh, and windows screen was constructed to use when cleaning became necessary. Cleaning the entire supply of sand requires about 4 hours.

Radial density tests have been completed with the quality being quite good. Two series of tests have been performed with different volumes. Tests with these concrete testing cylinders have shown agreement to within less than 5%. Once acclimated to the tests, between 4 and 6 tests could be run in one day.

Vertical testing has also been nearly completed using the nuclear gage. Care was taken to insure that the readings would not be adversely affected by the close quarters of the boundary and it appears that the gage is adequate. Still, the readings by the nuclear gage have not been reasonable in reference to the concrete molds nor have they been constant with depth. Possible reasons for this is discussed below. This testing required raining the entire sample so no more than two could be completed in a day.

Perhaps the most important accomplishment was devising the new equipment needed as it came up and developing testing protocol for the radial vertical tests for the quality of the samples. Both of these tests can be run with little difficulty and efficiently.

3.5 Results and Discussion

As was mentioned vertical testing results were not as good as expected. It appears at this point that a dense sample can be created with little difficulty and the longitudinal change in density is not great. However, in most samples the density increases with depth. The question is whether this increase is due to overburden stress or a flaw in the reading of the nuclear gage due to the near boundary. Tests made on two samples under the same conditions using both the cylinders and the gage for measuring seem to indicate that the readings are true since the gage agrees with the density calculated from the molds to within 1-5% while still showing the same increasing trend with depth. However not enough tests have been conducted to draw this conclusion for certain. If this were true, then it may not be possible to achieve low density samples using the current methods. Currently, tests are being conducted to measure the lower third of the sample with and without overburden to see if there is any change in the readings.

In a related matter, it was discovered that raining sand into the diffuser when it consisted of only the largest sieve and when it had the full compliment made almost no difference on the speed required for raising the sieves. This may indicate that the sieves are not performing properly and they may not be fine enough or the mass flow is too little. It would be worthwhile to explore those possibilities.

3.6 Conclusions

Much progress has been made over the past quarter and the sand rain device is nearing completion. Most of the unforseen problems have been worked out and it is both mechanically and structurally sound. Dense samples can already be made with a reasonable degree of accuracy while loose samples lose much homogeneity longitudinally. However, there is reason to believe that these things may be overcome with further testing.

3.7 Work Planned for next Period

Further radial and longitudinal uniformity testing will take place if needed and the exact nature of why low density samples will be explored.

3.8 Technical/Administrative Difficulties

There are no difficulties to report at this time.

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DEVELOPMENT OF STANDARD TEST PROTOCOLS AND DESIGN MODELS FOR DESICCATION BARRIERS
METC Task No. 1.4

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For:
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Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By:
K. Aminian, S. Ameri
Petroleum and Natural Gas Engineering
West Virginia University
P.O. Box 6070
Morgantown, WV 26506

ABSTRACT

Significant research effort is currently being conducted in the United States and the world for remediation of contaminated soils and groundwater. In parallel to remediation efforts, confinement technologies are needed to retard, or even prevent, the advancement of contamination plumes into the environment until the implementation of remediation efforts is completed. Research proposed herein aims at developing and investigating containment/confinement technology using desiccation for in situ formation of barriers.

The desiccant barrier proposed to be investigated will be the recently developed circulating air barrier (CAB). The CAB utilizes the injection of dry gas (air, nitrogen, CO_2, etc.) to create an ultra dry zone in situ; beneath or adjacent to the zone of contamination. The CAB relies on the removal (drying) of in situ liquids (water) so that any contaminating liquids entering the zone of desiccation must replace the previously removed liquids before the contaminants can migrate further. In field application, the CAB maybe established by means of specific patterns of vertical or horizontal (or a combination of vertical and horizontal) injection and withdrawal wells which enable the injected dry gas to travel through and dry the formation prior to exiting via a withdrawal well.

This research will be conducted using laboratory and meso-scale setups. In addition to conventional laboratory equipment proposed research will utilize a large scale reactor for simulating in situ conditions. Conducting the research based on a meso-scale setup will minimize problems associated with scale effects. Such problems are usually encountered when a method is developed in the laboratory with no considerations given to correlation of laboratory scale results to field implementation. The B-17 facility in METC with its infrastructure will be utilized to conduct the proposed work. The proposed experimental methodologies will simulate field conditions and should provide laboratory data to test and enhance predictions of CAB performance made by existing models and computer simulations.

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EXECUTIVE SUMMARY

This study aims at developing and investigating containment/confinement technology using a "Circulating Air Barrier (CAB)". The CAB utilizes the injection of dry air to create an ultra dry zone in situ; beneath or adjacent to the zone of contamination. The CAB system can provide many advantages, however, there are no laboratory data or CAB field performance data available in the literature. There are however are a number of issues that must be settled before it can be decided if CAB technology is applicable. Key among these issues are the influence of the soil characteristics (such as porosity, permeability, water saturation, grain size distribution, and clay content) on the drying profile. This research will be conducted using laboratory and meso-scale setups to evaluate the soil characteristics influence on CAB performance and will determine the effects of flow and sweep efficiency for fixed and mixed grain size soils. These experiments will validate and/or improve model predictions of system behavior and drying profiles of different soil types.

Sand-packs are being utilized for bench-scale testing to determine the relationship between soil characteristics and in situ barrier quality. Sand-packs are being calibrated to determine porosity, permeability, and saturation characteristics. The soil characteristics will be varied by changing grain size and sorting of the pack materials. A detailed CAB vessel assembly has been designed and been approved for experimental studies at B-17 facilities. The procurement of materials and fabrication of the assembly is in progress by EG&G at METC. Additional laboratory tests on core samples have been undertaken to evaluate the influence of porous media on air-water equilibrium and mass transfer.

1.0 INTRODUCTION

Many contaminated subsurface sites can not be economically decontaminated because of the limitations in technologies for decontamination. In such cases, containment becomes the primary technology for environmental protection. For non-excavatible sites, in situ containment is the most viable approach for preventing further spread of contamination. Different approaches for achieving in situ containment have been proposed, however, the study herein is focused on the evaluation of in situ barriers created by soil desiccation.

The desiccant barrier proposed to be investigated is the recently developed (1,2) Circulating Air Barrier (CAB). The CAB utilizes the injection of dry gas into a porous media to create an ultra dry zone in situ by removing water and reducing the water saturation below the level required for liquids to flow in order to prevent migration of underground contaminants. The CAB, once established, will also provide a means of monitoring and removing contaminants entering the zone. The proposed experimental methodology should provide laboratory data to test and enhance predictions of CAB performance made by existing models and computer simulations.

2.0 PURPOSE

The concept of the Circulating Air Barrier was conceived (1,2) to solve the subsurface contamination problems at the Hanford site used by the former Atomic Energy Commission. The references to CAB (1,2,3) are mainly related to CAB as a concept, and no field or laboratory data is available in the literature. The previous studies (1,2) concluded that a CAB system provided many advantages, however, the concept needs to be demonstrated to develop data necessary for scale-up and regulatory acceptance.

Among issues that must be settled before it can be decided if CAB technology is applicable to all DOE sites are the influence of the soil characteristics (such as, porosity, permeability, water saturation, grain size distribution, and clay content) on the drying profile and CAB performance. The laboratory study to be undertaken will evaluate the soil characteristics influence on CAB performance and will determine the effects of flow and sweep efficiency for fixed and mixed grain size soils. These experiments will validate and/or improve model predictions of system behavior and drying profiles of different soil types.

3.0 BACKGROUND

The Circulating Air Barrier (CAB) system is a desiccant-type barrier designed to prevent the movement of liquid contaminants toward groundwater, using air circulation and a processing system to lower water saturation in a targeted subsurface zone. The concept of Circulating Air Barrier was introduced in a study (1) that evaluated the barrier systems that could be installed beneath and around the tank farms with a minimum of excavation at DOE's Hanford Site. The CAB can be installed using either verticalor horizontal wells, establishing a pattern of air injection and extraction wells. The moving airvaporizes water in the zone and carries the water vapor to an extraction well. In time, circulation of the air reduces the water saturation in the swept interval, and continues to remove, by evaporation, liquids that move into the zone such as a leaked plume. No liquids can flow through this interval until a critical saturation is achieved, a saturation level that is well above the initial saturation. In the event that a leak occurs, the CAB system serves as a tool for early leak detection and providesa means to withdraw volatile contaminants for surface treatment. The study concluded that CAB system provided many advantages including non-physical confinement, active monitoring and leak detection, commercially available oil and gas technologies and equipment, emergency response and rapid deployment capability, and high potential for integration with other remediation technologies. However, the concept needs to be demonstrated to develop data needed for scale-up and regulatory acceptance.

A follow up study by K&M/BDM (2) which addressed modeling, cold test design, and surface processing and control design for the CAB demonstration at Hanford concluded that several issues must be settled before it can be decided if CAB technology is applicable to all DOE sites. Key among these issues is the influence of the soil characteristics such as porosity, permeability, water saturation, grain size-distribution, and clay content on the drying profile and CAB performance. This report further recommended a laboratory study to be undertaken to evaluate the soil characteristics influence on CAB performance and to find out the effects of flow and sweep efficiency for fixed grain-size and mixed grain-size soils. This would also serve to validate and/or improve modelpredictions of system behavior and drying profiles of different soil types. In addition, a range of geologic and climatic environments within which the CAB could successfully operate, baseline equilibrium moisture curve for a specific grain-size, and relative humidity for a specific temperature could be determined.

4.0 METHODOLOGY

Experimental procedures will be developed to address the problem of evaluatinga desiccant barrier, i.e., a barrier based on creation of a dry zone of earth beneath a contaminated zone to store contaminating liquids, thus preventing their further migration. Two phases of experimental research are proposed in this study. The B-17 facility in METC with its infrastructure will be utilized to conduct the proposed work. The first phase deals with bench-scale experiments using laboratory setups. The second phase will deal witha meso-scale setup that will utilize large tanks for simulations of in situ conditions in order to minimize problems associated with scale effects.

Bench-scale testing of the CAB process parameters will utilize sand packs to determine the relationship between soil characteristics and in situ barrier quality. Sand packs will be calibrated to determine porosity, permeability, and saturation characteristics. The soil characteristics will be varied by changing grain size and sorting of the pack materials. Residual water saturation of the pack will be established by first saturating the pack with water and then desaturating the pack by injection of water-saturated air. The pack will then be dried at various rates of air injection to determine drying characteristics as a function of rate, remaining water saturation, and soil characteristics. The final phase of sand pack testing will be to resaturate the packs with simulated contaminant fluids injected at low pressure (near atmospheric) at the top while monitoring any effluent at the bottom. This will allow determination of contaminants migration rate as well as total storage capacity of the packs.

Meso-scale performance testing will be used to investigate the effectiveness of the CAB system in containment/confinement of the study waste under simulated field conditions. The meso-scale tests of the CAB system will utilize large rectangular tanks in DOE/METC's B-17 building to create a three-dimensional test bed to verify, refine, and extrapolate the results of sand pack experiments to field like conditions. The tanks will be filled to a desired depth by slurring sand of predetermined grain size range intothe tanks with water. All or part of bed will then be desaturated, dried, and "contaminated" with a safe liquid to simulate the CAB process on the meso-scale. Instrumentation and sampling ports and tubes will be placed in strategic locations in or at the tanks to monitor the status of the CAB.

5.0 WORK PERFORMED THIS PERIOD

5.1 Task 1: SARS Activity

The CAB vessel assembly component parts were re-drawn to scale and and several drawings were modified to reflect the space limitations in Room 16 in B-17 Building. The 'Operating Procedure' was then revised and submitted to METC for approval in preparation to begin conducting experiments in B-17 Building. The operating plan was approved and the operating permit was issued on Septemeber 15, 1996.

5.2 Task 2.1: Literature Review

A comprehensive literature review relative to air-watermass transfer in porous media has been conducted. Considerable work has been reported in the literature on drying of porous materials, and related heat and mass transfer processes. Gamson et al.(18) were the first to establish the correlations for heat and mass transfer coefficients by measuring the rate of evaporation in packed beds at high Reynolds numbers ($300<Re<3000$). Acetis and Thodos(19) concluded that the surface temperature decreased as the air velocity increased and approached the wet-bulb temperature of the air only at very high air velocities. Petrovic and Thodos(20) and Wakao and Kaguei(21) introduced similar correlations to extend the mass transfer correlations to low Reynolds numbers region ($3<Re<300$).

Most of the above mentioned studies were conducted in saturated porous media consisting of only few layers of relatively large spheres or cylinders with very well defined geometry at high Reynolds numbers. These conditions are more representative of the industrial applications such as adsorption of gases in granular beds or drying of solids by through circulation of air. The information relative to dryingof naturally formed unsaturated porous media is very limited. Attempts to develop mass transfer correlations in unsaturated porous media have been limited only to the case of steady-state liquid infiltration(22,23,24) Rogers and Kaviany(25,26) distinguished two distinct regimes, funicular and pendular, in convective drying process in granular beds. Funicular regimes occurs when the liquid phase is continuous. Pendular regime occurs when liquid phase is immobile. They concluded that evaporation rate during funicular regime was constant and decreased substantially during pendicular regime indicating surface drying.

5.3 Task 3: Laboratory Equipment and Preliminary Testing

The CAB vessel assembly components were transferred to Room 16 in METC Building B-17. The CAB vesseland the monitoring equipments was then assembled. A new air flow manifold was constructed for the set-up in Room 16. The chambers have been calibrated. The previously developed protocols forsand compaction have been implemented using the chambers. Compaction tests to determine the optimum water saturation for compaction of 40/50, 50/70, 70/100, and 100/140 mesh sizes have been undertaken. In addition, a mixture of sand has been prepared to study the influnece of size distribution.

5.3.1 Sand Pack Assembly Characteristics Evaluation

The modified "Standard Ordinary Compaction Test" was utilized to determine the optimum moisture content. Porosity was measured using gas expansion method. This method provided the effective porosity. Total porosity was estimated by adding the known water content of the wet sand pack to effective porosity. Prior to porosity measurements, the total volume of the system without sand pack was also measured with gas expansion method. This allowed accurate detetermination of the sand-pack porosity. Traditionally, sand-pack permeability is measured by liquid flow (constanthead procedure). However, to conduct this test the sand-pack has to be fully-saturated. This procedure was not applicable since we were interested in studying unsaturated media.Therefore, a new approach for sand-pack permeability measurement was develpoed by measuring the pressure-drop as result of air flow through sand-pack at constant rate. Generally, the pressure-drop due to air flow through sand-pack is extremely small because of the magnitude of sand-pack permeability. However, a differential pressure transmitter with accuracy of 0.05 inch of water was used to measured the pressure-drop. The air flow rate was maintained during tests by mass flow meter and controller. To assure that the measured pressure-drop is entirely caused by the sand-pack, the pressure-drop for air flow through the system without sand was measured. It was concluded that the system did not introduce any significant resistances which could affect the permeability measurements. The permeability was measured for 10 different air flow rates to assure that the results are consistent and reliable.

The developed protocols for determining sand pack characteristics have been used to measure the porosity and permeability of sand packs of various mesh sizes compacted at the optimum water saturation. These experiments have been conducted at the METC building B-17. The porosity and permeability measurements indicate that the compaction procedure is valid and repeatable.

5.3.2 Sand Pack Drying Characteristics Determination

Dry air was injected through the sand pack and pressure-drop and the outlet pressure as well as relative humidity and temperature at inlet and oulet were continuously monitored. The experiments were conducted at the flow rates dictated by the field conditions. The Reynolds numbers for the experiments ranged from 0.09 to 1.32 (air flow rate ranged from 1 to 14.6 liter/minute). Relative humidity of air from laboratory supply system ranged from 6% to 10.5%. Relativehumidity and temperature of the air were measured in the oulet and inlet by hygrometers with chilled mirror sensor and temperature probes. The accuracy of the relative humidity and temperature probes were ±1.25 % and ±0.2% respectively. The air injection was continued until the sand pack was completely dried. This was achieved when the outlet and inlet relative humidity readings were equalized. The porosity and permeability of the dried sand pack was measured upon conclusion of each experiment. Finally, the dried sand pack was weighted. The total mass of water evaporated was then calculated by subtracting mass of dry sand pack from the mass of initial wet sand pack. This verified that the sand was completely dried. The results of experimental investigations for 20/30 mesh size sand pack drying have been studied to obtain a better understanding of the processes that takes palce during drying of the unsaturated unconsolidated porous media.

5.3.3 Design and Experimental Plans for the Scaled Models

The design of the scaled model, which isreferred to here as the Circulating Air Test Cell (CATC), the evaluation of the monitoring system and experimental procedures for Phase II experiments has been initiated.

6.0 RESULTS AND DISCUSSION

6.1 SARS Activity

The CAB Vessel has been assembled at B-17 Building, has passed various inspections, and the operating permit has been granted on Septemeber 15, 1996.
The next phase of SARS activity is submission of the 'Operating Permit' for Phase II experiments to METC for approval.

6.2 Sand Pack Assembly Characteristics Evaluation

The protocols for determining sand-pack characteristic namely, optimum moisture content, porosity, and permeability were developedusing the CAB vessel assembly. Porosity and permeability of the sand packs as representative macroscopic parameters were measured and used for comparison. The use of very accurate differential pressure transmitter allowed us to accurately measure the pressure-drop.. The measured porosity and permeability values based on the developed protocols have been found to be reasonable and repeatable.

6.3 Sand Pack Drying Characteristics Determination

The results of the sand pack drying experiments are illustrated in Figure 1. Two periods of decline appear to be present. The first and main decline period begins after early transient period that is characterized by increase in evaporation rate has ended. The second and final decline period occurs near the end of experiment. The main decline cannot be attributted to reduction in surface contact area due to surface drying. This is because there is significantly more (several order of magnitude) surface contact area available than required for achieving the observed evaporation rates. Figure 2 illustrates the processes that take palce during drying of a unsaturated thick porous media. The evapoation mainly occurs in a very thin zone. As this thin zone or evaporative front penetrates the sand pack, it evaporates the water. As the water evaporates, it absorbs the latent heat of evaporation from the air and the sand, causing the air and the surface temperature to decline. The air leaving the front is highly saturated. This saturated air then flows through the wet sand pack and absorbs more heat before it exits the sand pack. As a result, a secondary mass transfer takes place during this period due to heating of the air. However, the secondary mass transfer rate declines as the evaporative front approches the end of the sand pack. Figure 3 illustrates the temperature drop for differnet drying experiment. The maximum temperature drop occurs at the end of the experiment when the evaporative front reaches the exit. Therefore, theobserved main decline in is due to the secondary mass transfer decline. The final decline period is mainly due to non-uniform movement of the evaporative front which results in dry air break-through and reduction in sweep efficiency. This is principally very similar to other displacement processes in porous media.

It should be noted that the main decline in mass transfer is more pronounced at higher Reynolds numbers. Referring to Figure 3, it can be seen that as Reynolds number increase the temperature drop increases. This is because at low Reynolds number (low flow rates) small amount of water is required to saturate the air causing a very small temperature drop. Therefore, the secondary mass transfer is not significant at low Reynolds numbers. However, as the Reynolds number increases, more water is evaporates to saturate the air causing larger temperature drop. As a result, the secondary mass transfer becomes significant at higher Reynolds numbers. In addition, the secondary mass transfer decline more rapidly as Reynold numer increases bcause the evaporative front moves faster through the sand pack. It can be concluded that the mass transfer at low Reynolds numbers is nearly isothermal and approaches adiabatic at higher Reynolds numbers.

6.4. Design and Experimental Plans for the Scaled Models

A preliminary design of the CATC consist of a large tank which accommodates the sand bed and the necessary plumbing to simulate various well configurations. The construction of CATC will be undertaken upon approval of the construction permit which is imminent. Phase II experiments will be initiated by installation of CATC and assembly of the necessary the monitoring system at B-17 at METC.

7.0 CONCLUSIONS

7.1 SARS Activity

The next phase of SARS activity is submission of the 'Operating Permit' for Phase II experiments and presentation to METC for approval.

7.2 Sand Pack Assembly Characteristics Evaluation

The CAB vessel sand pack chamber, coupled with the procured instrumentation, have enabled generation of high quality data. The sand pack tests have useful in establishing experimental protocols. Compaction tests will be performed with assorted sand particle sizes to evaluate this relationship between particle size and optimum water conent. These compaction tests, coupled with porosity and permeability measurements, could provide baseline data for the larger scale experiments to be performed at METC building B-17.

7.3 Sand Pack Drying Characteristics Determination

The results of the drying tests indicate that as the Reynolds number (or the air flow rate) increases, the efficiency of drying process decreases. This is because the mass transfer coefficient declines as Reynolds number increases. It should however be noted that the real time of drying declines as the flow rate increases. Therefore, it is a matter of optimization between the time required for barrier development (drying) and horse-power requirements (flow rate). Two types of water evaportion takes palce in the sand pack. The primary evaporation takes palce in a thin zone. Thesecondary evaportion is the result of heat transfer and is only significant at higehr flow rates.

8.0 WORK PLANNED FOR NEXT PERIOD

8.1 SARS Activity

Prepare 'Construction/Operating Permit' for larger scale experiments in METC Building B-17.

8.2 Grain Size and Sorting Influence on Sand Pack Characteristics

Complete packing 40/50, 50/70, 70/100, and 100/140 meshsize sand as per modified 'Standard Ordinary Compaction Test', and measuring porosity and permeability.

8.3 Sand Pack Drying Characteristics Determination

Continue drying tests on sand packs to evaluate the effect of grain size on drying process.

9.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES

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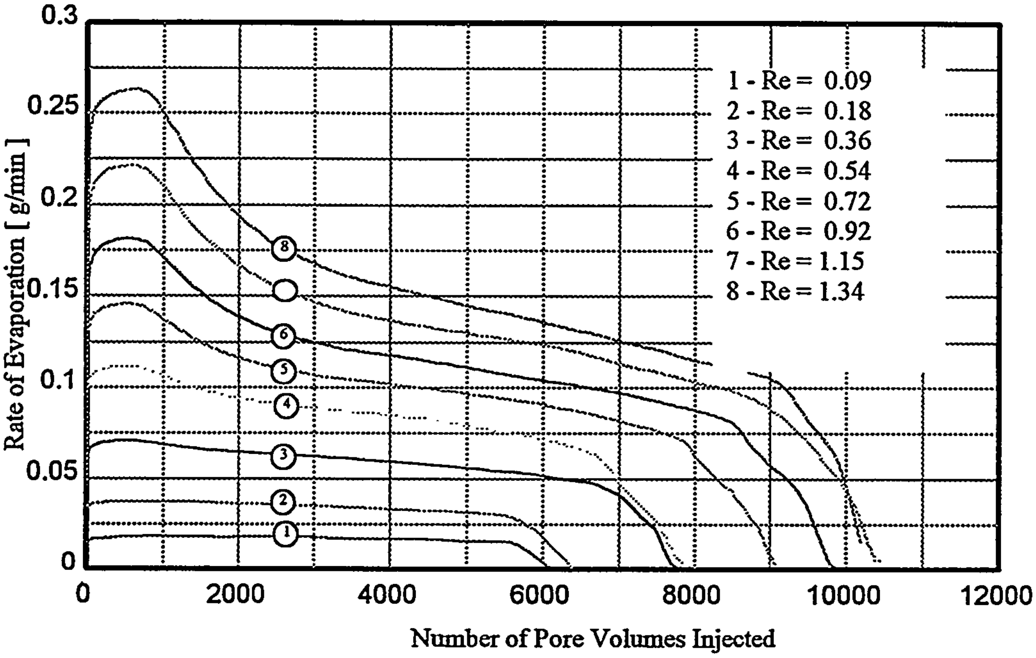

Figure 1. Water Evaporation Rates During Sand Pack Drying

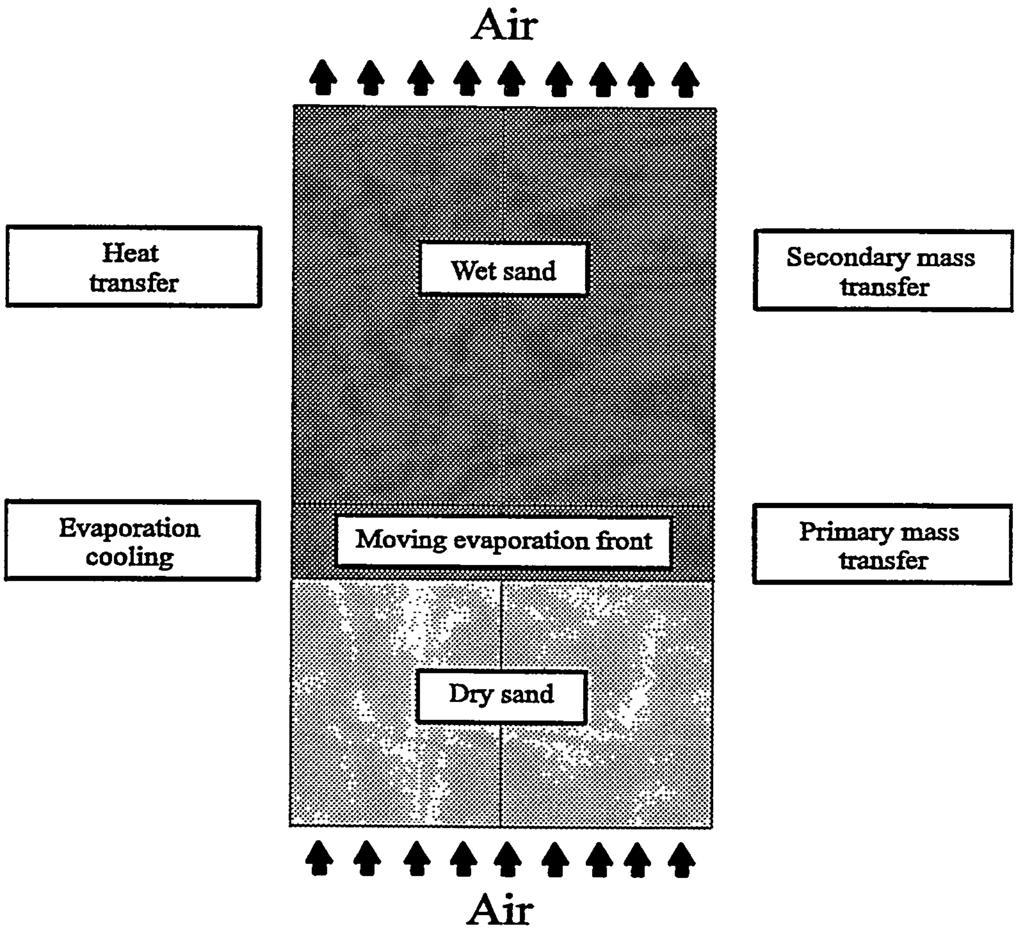

Figure 2. The Drying Process in the Sand Pack

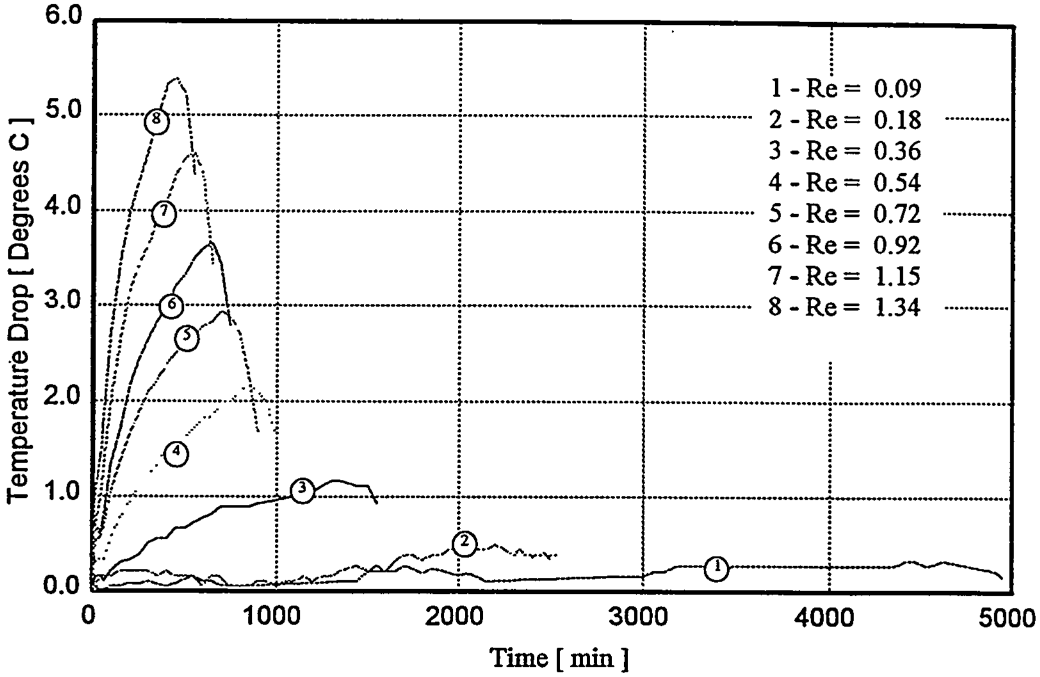

Figure 3.Temperature Drop During Sand Pack Drying

TECHNICAL SUPPORT FOR DEVELOPMENT OF STANDARD TEST PROTOCOLS AND DESIGN MODELS FOR INSITU FORMED BARRIERS

Quarterly Technical Progress Report
Reporting Period:
July 1, to September 30, 1996

Work Performed Under Contract
No.:DE-FC21-92MC9467

For:
US Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By:
BDM Federal, Inc.
Morgantown, West Virginia

Abstract

This work is designed to support the on-going research at DOE/METC aimed at the development of standard test protocols for in-situ-formed barriers. The immediate support effort is directed toward establishing DOE/METC's B-17 building as a center for laboratory, bench-scale, and large-scale experiments needed to evaluate in-situ-formed barrier systems. Two systems are being evaluated initially: (1) a grout barrier system, and (2) a circulating air or desiccant barrier system. The grout and desiccant barrier systems currently being tested were identified previously by DOE researches as high-potential alternatives for containing the movement of liquid contaminants which may have leaked from underground storage tanks existing at various DOE former weapon sites. Other barrier approaches are being independently evaluated under the DOE program, such as the freeze-wall systems and systems that totally encapsulate a leaking tank. The contractor will continue to monitor these programs for the development of standard test protocols in the B-17 facility. The goal is to establish the B-17 facility as the Focus Area Test and Evaluation Facility (FATE) for the entire DOE program.

The focus of the CY 1996 work is to (1) Support the operation of the circulating air barrier and grout laboratory experiments to be moved into the B- 1 7 facility; (2) prepare for the scale-up of these experiments in 1996, and (3) support the planning for testing additional barrier systems in 1997 and beyond.

Documents will be developed for METC's Lab Safety Committee to obtain construction and/or operating permits for the B-17 experiments and will include, but are not limited to, line drawings and definitions of operating parameters. The contractor will plan and coordinate the installation of equipment and utilities needed to support the experimental work to be conducted in B-17, with a special focus on using the existing equipment for this research. The contractor will also assist principal investigators with the planning, hands on operation, and analysis of experimental operations.

In the conduct of this support, the contractor personnel will meet with the principal investigators and review the key design elements, operational procedures, quality assurance and control procedures, and initial design sketches for experimental equipment. After collecting the required supporting data, the contractor will prepare draft drawings and operational procedures for construction and subsequent operation of the test cells. Applications for construction permits and operating permits will be prepared and submitted to the DOE/METC Lab Safety Committee.

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Executive Summary

During this period, a number of accomplishments were achieved. Construction and fabrication of equipment for the B-17 lab area was completed. This included installation of equipment racks, and electrical wiring, and instrumentation air as an air supply for the Circulating Air Barrier project. Fabrication of a work bench and hood for conducting chemical grout experiments was also completed.

OSHA, Chemical Hygiene, and Lab Safety Committee inspections were conducted as a prerequisite to obtaining an operating permit for each project. Both projects were issued operating permits. Tests are currently being conducted by the Circulating Air Barrier Project.

Preparation of a Construction Permit for the Circulating Air Test Cell (CATC) was completed. The permit was submitted to DOE for study.

Several study sessions were held with WVU PNGE faculty to discuss the instrumentation requirements for the pilot scale testing in the CATC vessel. Resistivity, Capacitance, and accoustic imaging were all discussed as potential methods for determining the distribution of layers of water within the CATC desiccation zone. Another problem discussed was the development of a suitable protocol for compaction of the sand in the vessel for the pilot scale tests. Accoustic transmission was discussed as a potential method of determining that the density of packing was consistent from one layer to the next.

Signs identifying the project work space were also installed during the quarter.

1.0 Introduction

This work is designed to support the on-going research now underway at DOE/METC; research that is aimed at the development of standard test protocols for in situ formed barriers. The immediate support effort is directed to establishing the DOE/METC B-17 building as a center for laboratory bench-scale and large-scale experiments needed to evaluate in-situ formed barrier systems. Two systems being evaluated by researchers are identified as the initial systems to be tested in the facility: (1) a grout barrier system, and (2) a circulating air barrier system, also referred to as a desiccant barrier system.

2.0 Purpose

The focus of the CY 1996 work is to: (1) support the installation, permitting, and operation of the circulating air barrier and grout laboratory experiments in DOE/METC's B-17 facility; (2) prepare for the scale-up of these experiments in 1996, and (3) support the planning for testing additional barrier systems in 1997 and beyond.

3.0 Background

Subsurface barrier technology development is an important and growing part of the Department of Energy (DOE) environmental program. In support of this program, the contractor has, under separate contract since 1991, evaluated alternative barrier approaches. The grout and desiccant barrier systems currently being tested were identified by a team of DOE contract researchers as high-potential alternative approaches to contain the movement of liquid contaminants from underground storage tanks existing at various DOE former weapon sites.

The desiccant barrier concept is based on existing petroleum technology, but proof of concept is needed and will be provided in the B-17 facility. Specific variables being evaluated include: Vapor phase transport phenomena, effects of adiabatic cooling near injection points, and effects of changes in grain size, porosity, and permeability on sweep efficiency within the target subsurface interval.

Grout barriers are a more mature technology, but specific protocols are being developed in the B-17 facility for application to DOE sites. Specific variables include: Injectability of the grout (viscosity), setting time, immersion, durability, permeability, shear strength, compressibility, and toxicity of the cured injected material.

Other barrier approaches are being independently evaluated under the DOE program, and these approaches include freeze wall systems and systems that totally immerse a leaking tank. The contractor will continue to monitor these programs for the development of standard test protocols in the B-17 facility. The goal is to establish the B-17 facility as the Focus Area Test and Evaluation Facility (FATE) for the entire DOE program.

4.0 Methodology

The methodology involves the development of documents for DOE/METC's Lab Safety Committee that are needed to obtain construction and/or operating permits for the B-17 experiments. Installation of necessary equipment utilities is coordinated in support of the experimental work to be conducted in B-17, with a special focus on using the exiting equipment for this research. Principal Investigators are assisted with the planning, hands on operation, and analysis of experimental operations.

In the conduct of this support, Contractor Personnel meet with the Principal Investigators and review the key design elements, operational procedures, quality assurance and control procedures, and initial design sketches for experimental equipment. After the required supporting data are collected, draft drawings and operational procedures for construction and subsequent operation of the test cells are prepared. Applications for construction permits and operating permits are prepared and submitted to the DOE/METC Lab Safety Committee with a goal of having permits returned approved within a 60 day period. This requires prioritized response by responsible DOE/METC Lab Safety Committee personnel, in order to keep the project on schedule and within budget.

5.0 Work Performed This Quarter

Milestones planned for this reporting period were as follows:

- Operating Permit for CAB
- Operating Permit for Chem. Perm. Grouting
- Construction Permit for CATC
- Construction Permit for GTC

Three of the four planned milestones were completed. The Construction Permit for GTC could not be completed because of a lack of data pertaining to the Grout Test Cell and the planned series of tests. The following is a brief discussion of the accomplishments during the period.

Subtask 1.5 Technical Support to West Virginia University

Task 0.0 - Planning and Technology Transfer

Planning for the instrumentation system to be used for CATC operations was conducted during the quarter. Papers describing the experiments on the Circulating Air Barrier were submitted for presentation to Eastern Regional SPE Meetings in conjunction with WVU PNGE faculty and student authors. BDM will prepare a a formal document for technology transfer after all experimental data is collected.

Task 1.0 Circulating Air Barrier Test {Protocol Development Support

Task 1.3 Obtain Operating Permit for Bench Scale CAB Operations

The request for Operating Permit was submitted to DOE in June and the permit was received in September. During this period, construction and fabrication of equipment for room 16 in B-17 was completed in support of the circulating air barrier project. The project began collecting data in late September

Task 1.4 Prepare Quality Assurance - Quality Control Guidelines for CAB

The Quality Assurance and Quality Control guidelines were completed for general and specific instances of operation of the circulating air barrier. The draft document was submitted for comment to West Virginia University in early August and the final Deliverable Item was submitted in September.

Task 1.5 Obtain Construction Permit For Scale-Up of CATC Unit

Work on development of the construction permit for the circulating Air Test Cell (CATC) was initiated in August. Several meetings were helt with faculty and staff concerning the type of instrumentation that would be used for detection of water saturation in the large test cell. Examination of the pro's and con's of resistance, capacitance, and accoustic instrumentation packages indicated that this was in reality a research project in itself. The group decided to investigate resistivity systems and accoustic systems as the most likely system to provide the measurements required. The accoustic system is interesting because it can also be used to determine the density of packing of the sand grains in the development of the test protocols.

Task 2.0 Chemical Grout Barrier Test Protocol Development Support

2.1 Obtain Operating Permit for Bench Scale Chemical Grout Studies

Paper work for the Bench scale Chemical Grout material testing experiment was submitted in August. The OSHA and Chemical Hygiene Lab safety Committee inspections were completed in September and the permits awarded on October 1, 1996.

2.2 Prepare Quality Assurance and Quality Control Guideline for Chemical Grout

Studies.

Quality Assurance and Quality Control Guidelines for Chemical Grouting Experiments were submitted in the same package as for the CAB.The report was delivered in early September, 1996.

2.3 Obtain Construction Permit for Scale -Up Operations on GTC

Initial design of the circulating air test cell (CATC) was completed in August ,1996. A fiberglass tank 8 foot high and 8 ffot in diameter was designed to contain the initial set of scaled-up experiments to examine vaporphase transport phenomena on a larger scale. This scale will allow closer examination of the effects of adiabatic cooling of the formation and any effects it may have on sweep efficiency.

Task 3.0 Freeze Barrier Test Protocol Development Support

3.1 Report on initial planning for testing Freeze Barrier

Initial planning was undertaken relative to development of a test cell that could be used for the scale-up testing which might conducted at approximately the same time as the CATC tests are conducted, and using the same instrumentation package.

Task 4.0 Dual Barrier System test protocol development support

4.1 Report on initial planning for testing of a dual barrier system

A second barrier system which is a chemical barrier system has been considered as part of a dual barrier system that may be examined in the dual barrier test cell(DBTC). That system needs to be considered for evaluation as part of a dual barrier system.

6.0 Results and Discussion

The purpose of BDM's technical support to West Virginia University is to provide guidance from an industry point of view on methodologies for designing and operating research project which will eventually be commercialized.
Providing technical support to groups is relatively easy when there is an interest and mutual co-operation between those providing the support and those who are to receive the support. It is sometimes difficult to perform research in a less than co-operative atmosphere. BDM has experienced a much higher level of co-operation from DOE/ METC personnel in getting the building prepared for installation of the experimental

apparatus and the supporting utilities. BDM hopes the cooperative atmosphere between all parties of the research projects will continue in the future to continue the progress at a faster pace than has been accomplished to date.

The conversion of B-17 Building into a DOE Focus Area Test and Evaluation Facility (FATE) is well under way and the validity of the concept has been established with the installation of the first two sets of experiments in the building. The Building will soon house both bench scale and pilot scale test projects.

7.0 Conclusions

Early results of the bench scale air drying process for the circulating Air Barrier proof of concept testing, supports the contention by the K&M-BDM Team of its viability.

8.0 Work Planned for Next Quarter

Obtain construction Permit for GTC

Obtain Construction Permit for GTC

Prepare Reports on Freeze Barrier and Dual Barrier Systems

9.0 Technical/Adminstrative Difficulties

Most of the difficulties which occured during the past quarter were those of an administrative nature, primarily of scheduling for inspections by a team when several members are on vacation. There are other apparently adminstrative problems associated with making funds available for commitment to support students, or to purchase equipment before the work has been completed, or planned and approved. Purchasing of equipment to be used cannot be approved until the permit for construction has been granted. This sometimes has the effect of delaying equipment purchases for an entire semester (three months) period. BDM has proposed to speed up this process by purchasig the equipment as soon as the construction Permit is approved. This also requires the placing of requested funding for equipment in the BDM financial plan.

SITE REMEDIATION TECHNOLOGIES
IN SITU BIOREMEDIATION OF Chlorinated Solvents
METC TASK No. 1.6

Work Performed Under
Contract No.: DE-FC21-92M29467

For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O.Box 880, Collins Ferry Road
Morgantown, West Virginia

By:
Patrick E. Carriere, Department of Civil Engineering
William A. Sack, Department of Civil Engineering
Wei Lin, Department of Civil Engineering
Tim Mackey, Department of Civil Engineering
Chris Cunningham, Department of Civil Engineering

West Virginia University
Morgantown, West Virginia 26506

October 1996

Disclaimer

Abstract

Chlorinated aliphatic hydrocarbons (CAHs) contamination in soils and groundwater is a wide-spread problem at Portsmouth Gaseous Diffusion Plant Site (PORTS) and many other DOE sites. These solvents often move downward through the soil profile and the groundwater accumulating as dense nonaqueous aqueous phase liquid (DNAPL) over an aquitard. Reliable and cost effective field technologies are needed to cleanup these sites. To date, when remedial measures are implemented to achieve source containment and/or plume containment at DNAPL sites, the measures nearly always include pump-and-treat. Approaches based on extracting contaminated groundwater with pumps and subsequently treating it above ground have proven ineffective for removing much of the immiscible DNAPL phase mass. To accomplish source mass reduction, another approach is to use surfactants to enhance the dissolution of DNAPLs followed by in situ bioremediation. In situ bioremediation of CAHs often involves amendments to enhance the degradation processes. These will include injection of primary substrate, nutrients, and electron acceptors into the plume after flushing of DNAPL zone to stimulate activity of natural microbes for destruction of contaminants that will be desorbed from the porous medium. In situ bioremediation offers a number of advantages over other processes for destruction of organics in groundwater. Advantages include the potential for complete or near complete destruction of contaminants in place, avoidance of transfer of pollutants to another medium, less risk of health hazards due to human exposure and cost-effectiveness in many cases.

This research seeks to exploit the natural symbiotic relationship between methanogenic and methanotrophic microorganisms. The methanogens are able to carry out the anaerobic reductive dehalogenation (RD) of highly chlorinated solvents and producing methane. The methanotrophs, in turn, utilize the end products of the methanogens, including methane, to aerobically degrade the residual compounds to environmentally acceptable end products. Both groups of organisms work cometabolically and require a primary substrate.

The proposed research will mimic the field conditions in the Gallia sand and gravel aquifer at PORTS. The proposed research will be to evaluate biopolishing of contaminants at PORTS after surfactant flooding. The surfactant flooding will probably utilize an anionic surfactant and an alcohol such as isopropanol. Due to all unknown factors involved in the surfactant flood, it is not possible at this time to develop a good estimate of the concentrations of contaminants and surfactant that will be left in the formation after the flood. Therefore, a range of levels will be evaluated in both columns and microcosms. It is possible that the isopropanol or perhaps other contaminants already present in the formation may serve as primary substrate for methanogenic and methanotrophic organisms. This possibility will be evaluated by not adding a separate primary substrate (typically methanol) during the study. Again, both column and microcosms will be used to evaluate biopolishing at PORTS.

Executive Summary

Chlorinated aliphatic hydrocarbons (CAHs) contamination in soils and groundwater is a wide-spread problem at Portsmouth Gaseous Diffusion Plant Site (PORTS) and many other DOE sites. These solvents often move downward through the soil profile and the groundwater accumulating as dense nonaqueous aqueous phase liquid (DNAPL) over an aquitard. Reliable and cost effective field technologies are needed to cleanup these sites. To date, when remedial measures are implemented to achieve source containment and/or plume containment at DNAPL sites, the measures nearly always include pump-and-treat. Approaches based on extracting contaminated groundwater with pumps and subsequently treating it above ground have proven ineffective for removing much of the immiscible DNAPL phase mass. To accomplish source mass reduction, another approach is to use surfactants to enhance the dissolution of DNAPLs followed by in situ bioremediation. In situ bioremediation of CAHs often involves amendments to enhance the degradation processes. These will include injection of primary substrate, nutrients, and electron acceptors into the plume after flushing of DNAPL zone to stimulate activity of natural microbes for destruction of contaminants that will be desorbed from the porous medium. In situ bioremediation offers a number of advantages over other processes for destruction of organics in groundwater. Advantages include the potential for complete or near complete destruction of contaminants in place, avoidance of transfer of pollutants to another medium, less risk of health hazards due to human exposure and cost-effectiveness in many cases.

Two approaches are being taken to simulate the site conditions and to predict the effectiveness of bioremediation at PORTS. Continuous flow column studies and anaerobic batch reactor (microcosm) studies are being performed. Two column studies, a simultaneous anaerobic and aerobic column and a sequential anaerobic and aerobic column, are in progress along with the construction of a second simultaneous column. It is hoped that various influent TCE concentrations and the possible use of surfactant as a substrate will be explored using these columns. Higher overall TCE destruction has been achieved in the simultaneous column with TCE removal as high as 98%. Moderate vinyl chloride (VC) concentrations remaining in the column effluent have continued to be a problem with the simultaneous column. Attempts of increasing aerobic degradation of VC via increased hydrogen peroxide addition has greatly improved VC destruction. The sequential column has been showing lower percentage of TCE destruction, 70-80%, but has shown greater daughter product destruction and lower effluent concentrations of VC. Better pH control, through buffers, and reinoculation of the anaerobic and aerobic zones of the sequential column has shown some improvement in TCE degradation.

Preliminary microcosm studies have been performed to establish an experimental procedure for their use in this study. Factors such as head space volume, substrate addition, biomass concentrations, trace metal and nutrient concentrations, and possible agitation have been considered. Initially testing has focused on comparisons between substrates and initial TCE concentrations. The substrates being studied are methanol and the surfactant used in the surfactant flood.; initial TCE concentrations are between 20 and 100 ppm. Progress in both the column and microcosm studies has been delayed by continued instrument failure.

Technical difficulties were experienced this quarter when the gas chromatograph (GC) failed. No analytes were detected by the GC for 4 weeks. GC measurements are vital in determining the amount of TCE that exists in the influent, throughout the columns, and in the microcosms.

Further discussions were held this quarter with Intera and METC/DOE personnel in order to obtain samples of soil and groundwater from the Potrsmouth, Ohio DOE site. However, there appears to be a number of political and technical barriers to obtain these samples. In order to accomplish project objectives, it has been decided to simulate groundwater from the Portsmouth site for use in our microcosms/ column studies.

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1.0 INTRODUCTION

Chlorinated aliphatic hydrocarbons (CAHs) contamination in soils and groundwater is a wide-spread problem at Portsmouth Gaseous Diffusion Plant Site (PORTS) and many other DOE sites. These solvents often move downward through the soil profile and the groundwater accumulating as DNAPL over an aquitard. Reliable and cost effective field technologies are needed to cleanup the site. To date, when remedial measures are implemented to achieve source containment and/or plume containment at DNAPL sites, the measures nearly always include pump-and-treat. Approaches based on extracting contaminated groundwater with pumps and subsequently treating it above ground have proven ineffective for removing much of the immiscible DNAPL phase mass. To accomplish source mass reduction, another approach is to use surfactants to enhance the dissolution of DNAPLs followed by in situ bioremediation.

In situ bioremediation of CAHs will often involve amendments to enhance the degradation processes. These will include injection of substrate, nutrients, and electron acceptors into the plume after flushing of DNAPL zone to stimulate activity of natural microbes for destruction of contaminants that will be desorbed from the porous medium. In situ bioremediation offers a number of advantages over other processes for destruction of organics in groundwater. Advantages include the potential for complete or near complete destruction of contaminants in place, avoidance of transfer of pollutants to another medium, less risk of health hazards due to human exposure and cost-effectiveness in many cases.

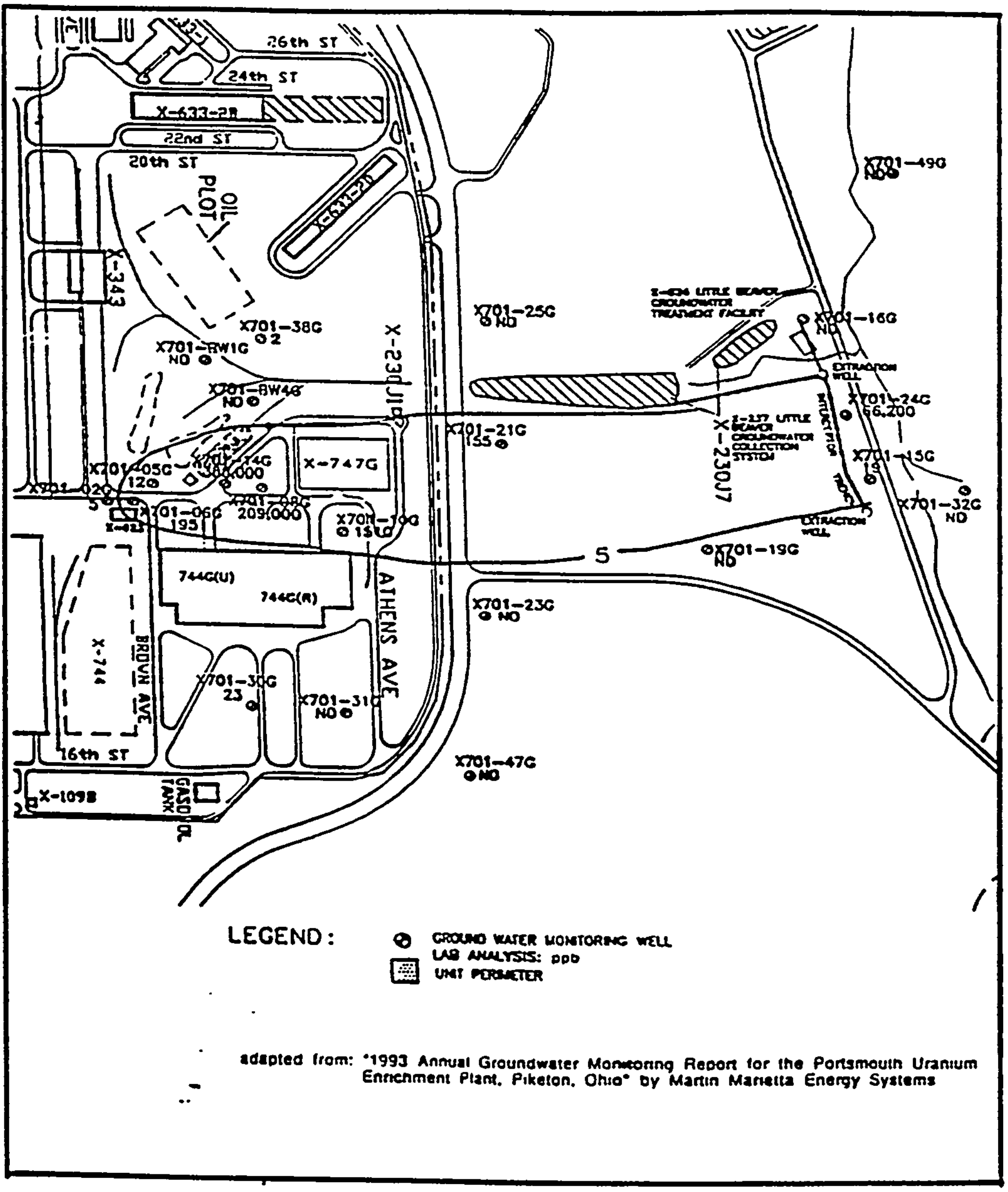
2.0 PURPOSE

The purpose of the research is to evaluate biopolishing of contaminants at PORTS after surfactant flooding. The surfactant flood will probably utilize an anionic surfactant and an alcohol such as isopropanol. Due to all unknown factors involved in the surfactant flood, it is not possible at this time to develop a good estimate of the concentrations of contaminants and surfactant that will be left in the formation after the flood. Therefore a range of levels will be evaluated in both columns and microcosms. It is possible that the isopropanol or perhaps other contaminants already present in the formation may serve as primary substrate for methanogenic and methanotrphic organisms. This possibility will be evaluated by not adding a separate primary substrate (typically methanol) during the study. Both columns and microcosms will be used to evaluate biopolishing at PORTS and to evaluate the ability of methanotrophic, methanogenic, and other selected bacteria for cost-effective biotransformation of TCE and other volatile organic compounds (VOC's) found at PORTS.

3.0 BACKGROUND

A number of exciting developments are occurring in the field of environmental biotechnology (engineering applications of microbial ecology). For example, while trace concentrations of some organic contaminants cannot support microbial growth as the sole electron donor, they can still be biotransformed by engineering the system so that the microbial population obtains the majority of its energy and carbon from a different compound that serves as the primary substrate. This is sometimes referred to as *secondary utilization*. There are also many organic contaminants that are biotransformed in the environment for which no microorganisms have been found which are able to use them as sole carbon source. This is a special case of secondary metabolism often termed *cometabolism*. Cometabolism has been defined (Dalton, 1982) as the "transformation of a non-growth substrate in the obligate presence of a growth substrate or another transformable compound". As an example, halogenated methanes, ethanes, and ethylenes are poor growth substrates for bacterial growth but may be degraded by methanotrophs growing aerobically on methane (Henson et al., 1988).

The test area at PORTS is designated X701B and is illustrated in Figure 1. This area originally contained a holding pond which received liquid wastes, including chlorinated solvents, from industrial operations elsewhere on



adapted from: "1993 Annual Groundwater Monitoring Report for the Portsmouth Uranium Enrichment Plant, Piketon, Ohio" by Martin Marietta Energy Systems

Figure 1. Site Map of the X701B Test Area (Source: Intera)

site. The X701B area is underlain by lacustrine silts and clays and by deeper alluvium. These Quaternary units lie on the Sunbury shale which, in turn, overlies the Berea sandstone. The geologic and hydrologic properties of the proposed test site is presented in Table 1 and the hydrostatigraphy of the proposed test site is presented in Figure 2. The Gallia alluvium was deposited by the ancient Portsmouth river, which left abandoned alluvial and fluvio-lacustrine deposits across the PORTS site. Hydraulic conductivities in the Gallia are of the order of 10^{-2} to 10^{-3} cm/s. The Gallia is underlain by the Sunbury shale, which is estimated to have a vertical hydraulic conductivity on the order of 10^{-8} cm/s.

Groundwater in the Gallia underlying X701B flows from the north and west across the X701B holding pond and continues east toward Little Beaver Creek. Groundwater flow is directly affected by the thickness of the Gallia which is generally two to six feet thick in the vicinity of X701B. A groundwater divide is present west of X701B. The groundwater flow direction is constant throughout the year with the gradient exhibiting very little seasonal change. The gradient is flattest in the vicinity of X701B and steepens east of X230J7 toward Little Beaver Creek. The steeper gradient is due to decreasing thickness of the Gallia.

The DNAPL within X701B has a specific gravity of approximately 1.4 and the viscosity is measure as 4 centipoises. The DNAPL is a multi-component liquid composed of TCE, PCE, and a number of minor components including PCB and 1,1-DCE. The approximate concentration of DNAPL from the X701B area is presented in Table 2. It should be noted that the chemical analysis only identified a small fraction of the DNAPL components and the listed quantities are probably significantly underestimated, in particular TCE. Recent measurements of TCE indicate that TCE concentrations at the perimeter fence have reached 800 mg/L.

4.0 METHODOLOGY

The principal focus of the investigation is to evaluate the performance of biopolishing of contaminants at PORTS after surfactant flooding. This research was organized into five phases, as follows:

Phase 1: Literature Review.

Phase 2: Synthesize Water And Soil Samples

Phase 3: Characterize Groundwater and Soil samples

Phase 4: Evaluation of Biopolishing Using Columns

Phase 5: Evaluation of Biopolishing Using Microcosms

5.0 WORK PERFORMED THIS PERIOD

In this quarter, partial site data were obtained from Intera, Inc. and reviewed; the existing columns continued to operate in both sequential and simultaneous modes; and microcosm studies have begun. A second simultaneous column has been constructed and is presently undergoing tracer and integrity testing. Using an Orion Model 96-17BN chloride probe initial measurements of chloride concentrations in the influent and effluent flows have been measured. In correlation to GC analysis this will allow more accurate assessment of column performance and mass balance calculations. Microcosm work has proven the ability of methanogens to completely degrade TCE, and a previously unknown acclimation phase has been discovered. This discovery has led to the creation of a new maintenance reactor for the express purpose of acclimating methanogens to TCE. Other microcosms have been created to study the effects of the surfactant sodium dihexyl sulfosuccinate on biodegradation.

Table 1. Geologic and Hydrogeologic Properties of the X701B Area (Source: Intera)

FORMATION	LITHOLOGY	HYDRAULIC CONDUCTIVITY (cm/s)	APPROXIMATE AVERAGE THICKNESS (m)
Minford	Clay	8.1E-08	4.6
Minford	Silty clay	1.5E-06	3.0
Gallia	Sand	E-02 to E-03	1.5
Sunbury	Shale	K_v = E-08	3.0
Berea	Sandstone	5.6E-05	9.1
Bedford	Shale	2.1E-05	30

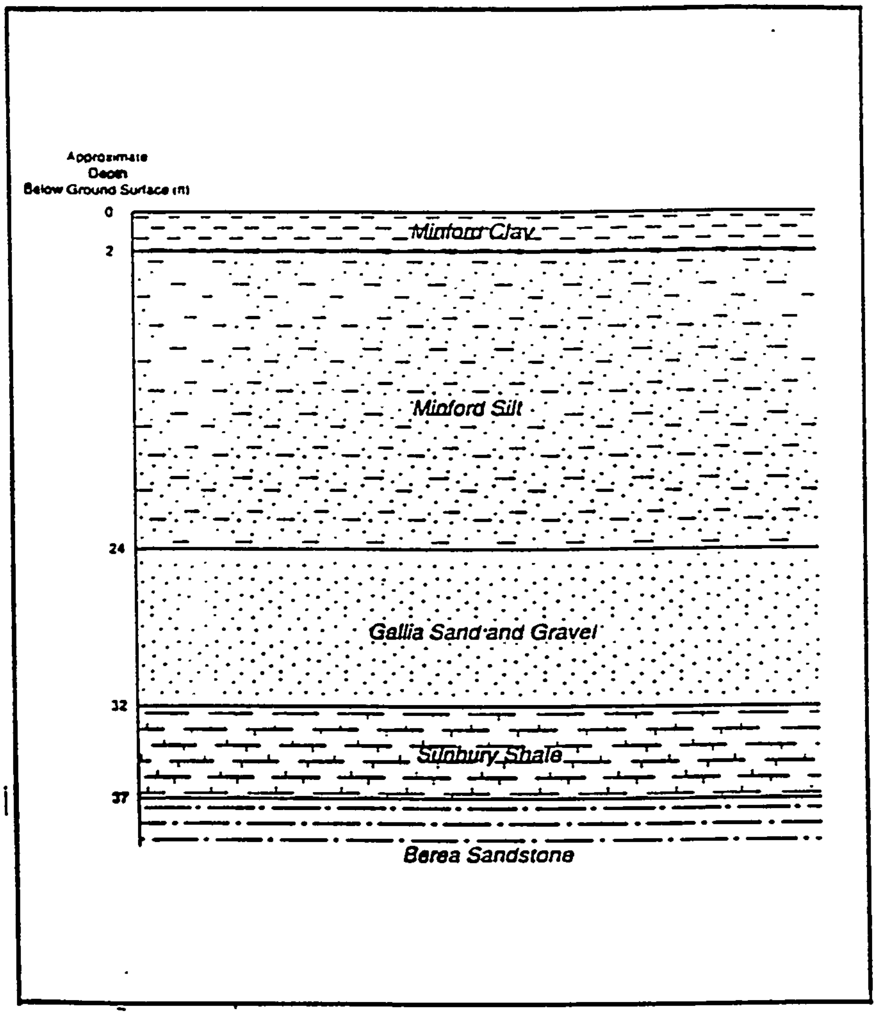


Figure 2. Hydrostatigraphy of the X701B Test Area (Source: Intera)

COMPOUNDS IDENTIFIED IN DNAPL SAMPLE	QUANTITY (g/L)	NOTES
Trichloroethene	12	
Tetrachloroethene	3.6	Exceeds initial calibration range
Perchloromethane	0.22	"Carbon tetrachloride"
1,1-dichloroethene	0.034	
1,1,2-trichloro-1,2,2-trifluoroethane	1.6	(CFC-113), tentatively identified
1,1,1-trichloroethane	2.3	Tentatively identified
1,1,2-trichloroethane	0.12	Tentatively identified
Toluene	0.028	Tentatively identified
Xylenes	0.060	Tentatively identified
1,2,4-trimethylbenzene	0.080	Tentatively identified
Polychlorinated biphenyls	820 ug/g (0.08%)	Identified as PCB-1254

Table 2. DNAPL Composition from Pumping Well BW2G (Source: Intera)

1. Literature Review--- 80% complete

Numerous journal articles on microcosm preparation and studies have been consulted in fine-tuning the microcosm batch studies.

2. Procure/Synthesize Water and Soil Samples--- 0% complete

It is unlikely that ground water or soil samples will be obtained from the PORTS site for use in this research. Instead synthetic ground water will be used for experimentation.

3. Characterize Groundwater and Soil Samples--- 70% complete

Data has been collected from Intera on groundwater and soil contents. More in-depth and up-to-date information is being sought from Intera.

4. Evaluation of Biopolishing Using Columns--- 60% complete

Column studies are ongoing. Determination of column nutrient requirements to aid destruction of TCE and its daughter products has been completed. Further studies include optimization of electron donor concentration and pH to increase TCE/daughter product destruction even further.

5. Evaluation of Biopolishing Using Microcosms--- 60% complete

Preliminary determination of experimental parameters which will provide effective and reliable results have been completed. Microcosms using synthetic groundwater have been made to evaluate the effect of surfactant as an additional substrate; and to determine the effect of various initial TCE concentrations.

6. Preparation and Submission of Quarterly and Final Reports--50 % Complete

Assuming equal weight for each of the 6 project tasks noted above, it is estimated that the overall project is 53% complete.

6.0 RESULTS AND DISCUSSION

Experimental studies performed in this quarter include column operations and anaerobic microcosm testing. Results of these studies are presented in this section.

6.1 Sequential Columns.

A schematic diagram of the sequential column setup is presented in Figure 3. The anaerobic and aerobic columns have been operated in a sequential mode continuously in this quarter. Table 3 shows the substrate and inorganic concentrations entering the anaerobic column for various time periods in this quarter. Aerobic condition in the second column is achieved by injecting H_2O_2 to the column. CH_3OH is added to the aerobic column as the primary substrate. Doses of H_2O_2 and CH_3OH are presented in Table 4.

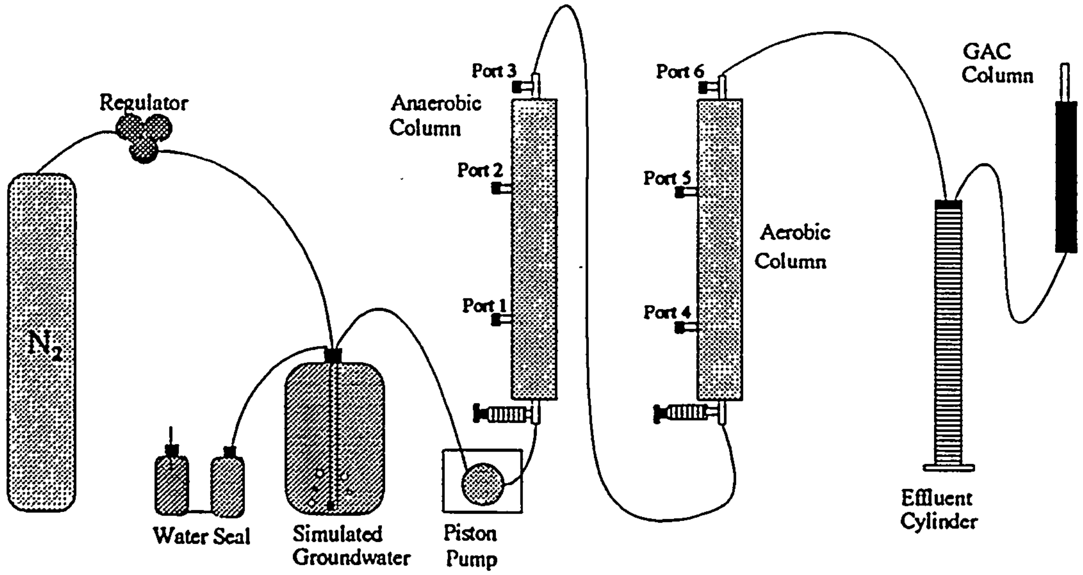


Figure 3 Schematic Diagram of the Sequential Column Setup

Table 3. Sequential Anaerobic Column Operational Conditions (Concentrations, mg/L)

Chemical	7/1-7/14	7/15-7/28	7/29-8/4	8/5-9/30
NH_4Cl	28.35	28.35	28.35	28.35
K_2HPO_4	5.84	5.84	5.84	6.05[3]
KH_2PO_4	4.22	4.22	4.22	4.48[3]
$MgCl_2.6H_2O$	5.76	5.76	5.76	5.76
$MnCl_2.4H_2O$	1.92	1.92	1.92	1.92
$CoCl_2.6H_2O$	3.84	3.84	3.84	3.84
$ZnCl_2$	1.92	1.92	1.92	1.92
H_2SeO_3		0.08[1]	0.08	0.08
$CaCl_2.2H_2O$	6.34	6.34	6.34	6.34
H_3BO_3	0.58	0.58	0.58	0.58
$NiCl_2.6H_2O$	1.92	1.92	1.92	1.92
$Na_2MoO_4.2H_2O$	0.768	0.768	0.768	0.768
L-Cysteine	24.0	24.0	24.0	24.0
$Na_2S.9H_2O$	40.0	40.0	40.0	40.0
$NaHCO_3$	76.8	76.8	76.8	76.8
Yeast Extract	5.0	5.0	5.0	5.0
$FeCl_2{\bullet}4H_2O$	6.0	6.0	6.0	6.0
Vitamin B-12	4.81	4.81	4.81	4.81
Methanol	800	800	800	1100[4]
Na_2CO_3	528	528	0[2]	0
TCE	20	20	20	20
HRT, hours	24	24	24	24

[1] To provide additional trace metals based on literature review.

[2] To return pH to acceptable range.

[3] To provide additional pH buffering.

[4] To encourage growth of methanogens throughout entire column, 300 ppm added at 2/3 column height.

Table 4. Sequential Aerobic Column Operational Conditions

Chemical	7/1 -8/11	8/22 - 9/12	9/13 - 9/30
H_2O_2, mg/L	220	170[1]	200[2]
CH_3OH, mg/L	54	54	54
HRT, hours	24	24	24

1 Reduced peroxide concentration during inoculation period to prevent toxic effects.
2 To improve DP degradation after inoculation

TCE removal rates in the aerobic and aerobic columns, and the overall TCE removal in the sequential column system are presented in Table 5. During the initial part of this quarter, low TCE removal was observed in the column effluents (Figure 4). The first step taken to improve the percent removal was determining whether the column ground water could be lacking any trace minerals. After a literature review of typical trace minerals found in similar types of research selenic acid was added to the concentrated given in Table 3 to supply the recommended concentration of selenium. Additionally both the aerobic and anaerobic columns were reinoculated with methanogens and methanotrophs. The pH was also a major concern in the operation of both the sequential and the simultaneous column. In hindsight the high pH measured is believed to be the real reason for the fall off in TCE degradation in both columns. Based on previous work by Davis and Raman sodium carbonate was being added via the TCE and substrate syringe to prevent the microorganisms from creating overly acidic conditions in the column. It became apparent that the dosage of sodium carbonate was too high for the flow rates being encountered. The addition of sodium carbonate was halted and instead of adding a base to keep the pH around 7 additional phosphate buffer was added via the TCE and substrate syringe. This change yield marked improvements in pH (Figure 5) and began to show significant improvement in the percent removal. However GC analysis became questionable and was eventually eliminated shortly after so further changes were not possible.

Table 5. Average TCE Removal throughout the Sequential Columns (%)

	7/1-7/3	7/3-7/9	7/10-7/11	7/12-7/18	7/19-7/28	7/29-8/5	8/6-8/11
% TCE Removal Anaerobic Column	49	42	73	70	45	49	40
% TCE Removal Aerobic Column	22	22	-26	8	33	12	-14
Overall % Removal	53	55	64	72	63	55	31

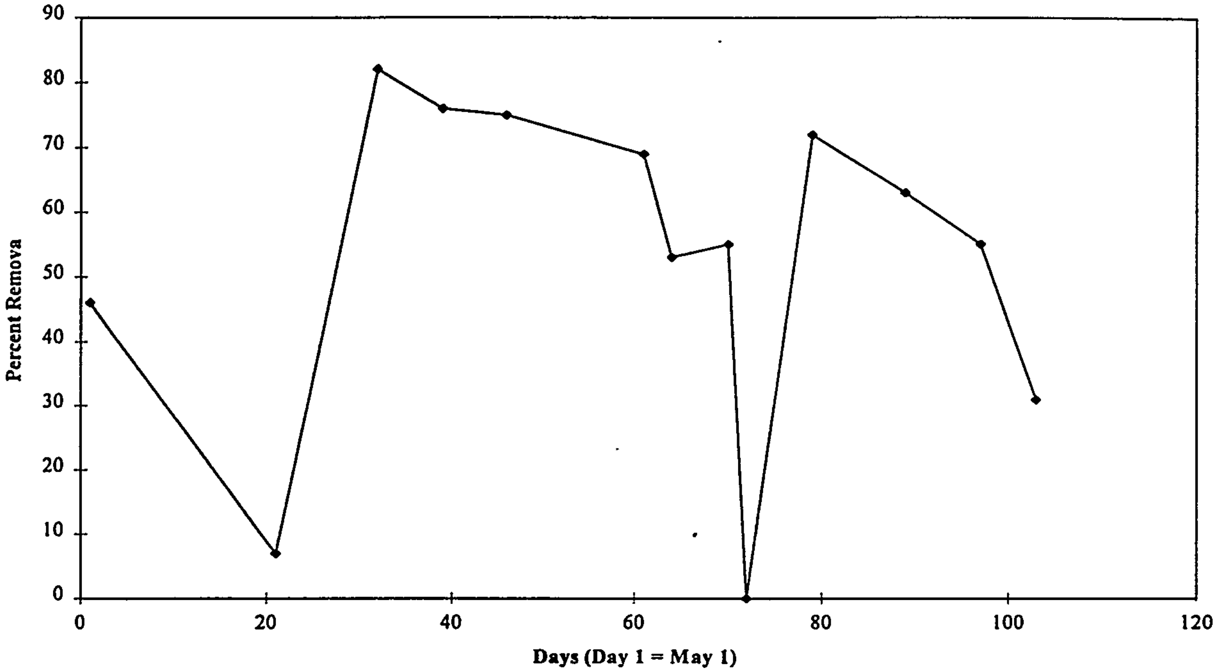


Figure 4. Sequential Column Percent TCE Removal vs. Time

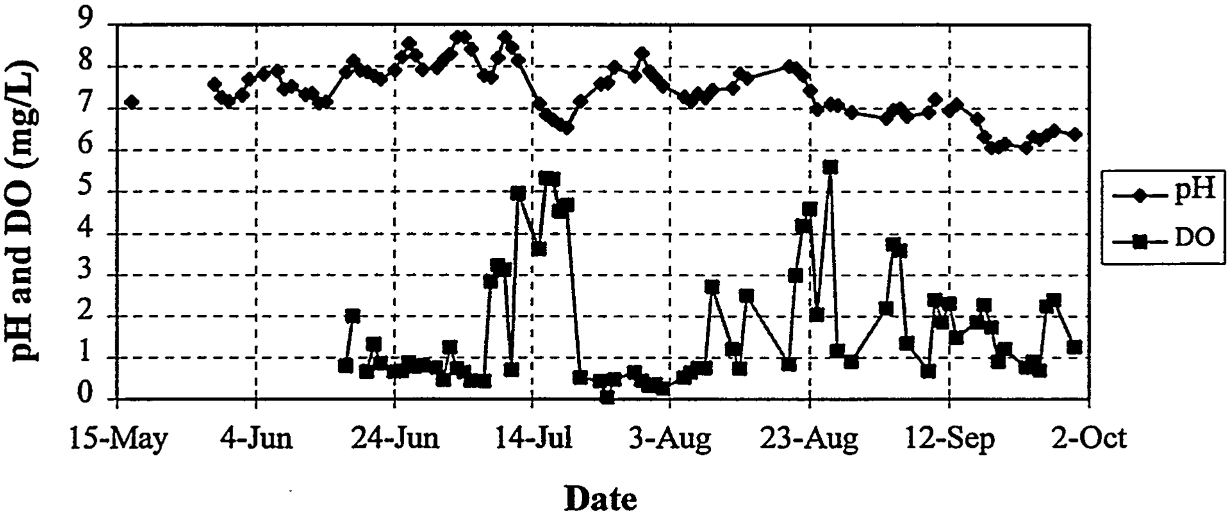


Figure 5. Sequential Column Effluent pH and Dissolved Oxygen

Table 6. Average ORP Values in mV for the Sequential Columns

	7/8-7/14	7/15-7/21	7/22-7/28	8/5-8/11	8/19-8/25	8/26-9/1	9/2-9/8	9/9-9/15	9/16-9/22	9/23-9/30
Aerobic Top	29	114	-8	-20	-49	-56	-78	-95	-17	93
Aerobic Bottom	-13	85	40	24	-92	-71	-22	-75	20	54
Anaerobic Top	-83	-80	-75	30	-170	-88	-29	-30	5	-33
Anaerobic Bottom	-142	-99	-74	80	-63	-80	-45	-12	-29	-70

Cis-, trans-, and 1,1-DCE, and VC may be produced from biological transformation of TCE. Concentrations of these daughter products have been measured during the column study and used to evaluate the performance of the anaerobic and aerobic columns. Table 7 shows the concentrations of these daughter products in the effluent of anaerobic and aerobic columns for various time periods in this quarter. Low concentrations of these daughter products in the effluents indicate that most of these compounds were degraded in the columns.

Table 7. Daughter Product Concentrations (ug/L) in the Sequential Columns (5/1-6/30)

	7/1-7/3		7/4-7/9		7/10-7/11		7/12-7/18	
Ports	Aerobic Top	Anaerobic Top	Aerobic Top	Anaerobic Top	Aerobic Top	Anaerobic Top	Aerobic Top	Anaerobic Top
VC	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5
1,1-DCE	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5
trans-DCE	9	20	9	14	16	19	8	19
cis-DCE	6	14	4.5	19	12	7	4	16

	7/19-7/28		7/29-8/5		8/6-8/11	
Ports	Aerobic Top	Anaerobic Top	Aerobic Top	Anaerobic Top	Aerobic Top	Anaerobic Top
VC	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5
1,1-DCE	<2.5	<2.5	<2.5	<2.5	<2.5	<2.5
trans-DCE	<2.5	17	8	16	8	19
cis-DCE	3	<2.5	<2.5	12	4	16

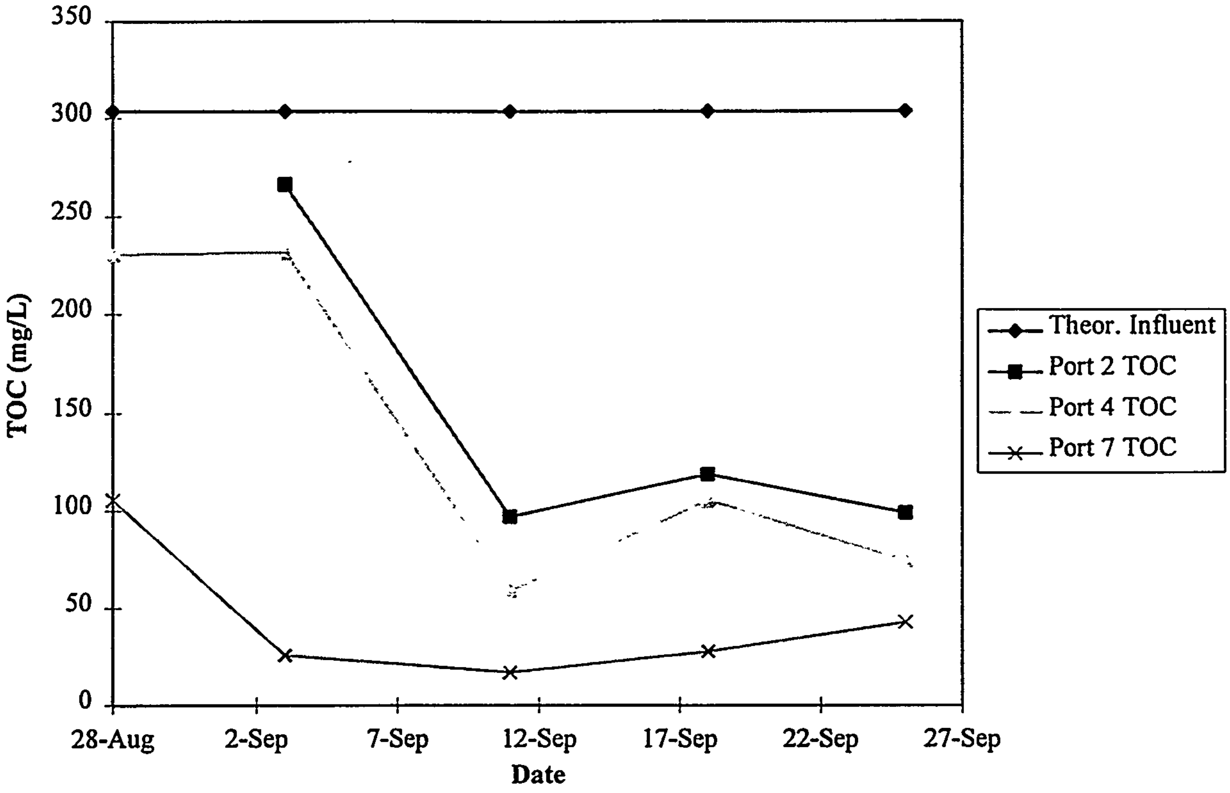


Figure 6. Sequential Column TOC Results

6.2 Simultaneous Column

A schematic diagram of the simultaneous setup is given Figure 6. The simultaneous column can be divided into three reaction zones, anaerobic, facultative and aerobic. Tables 8, 9 and 10 show the substrate and inorganic concentrations entering the anaerobic, facultative and aerobic zone, respectively, for various time periods in this quarter.

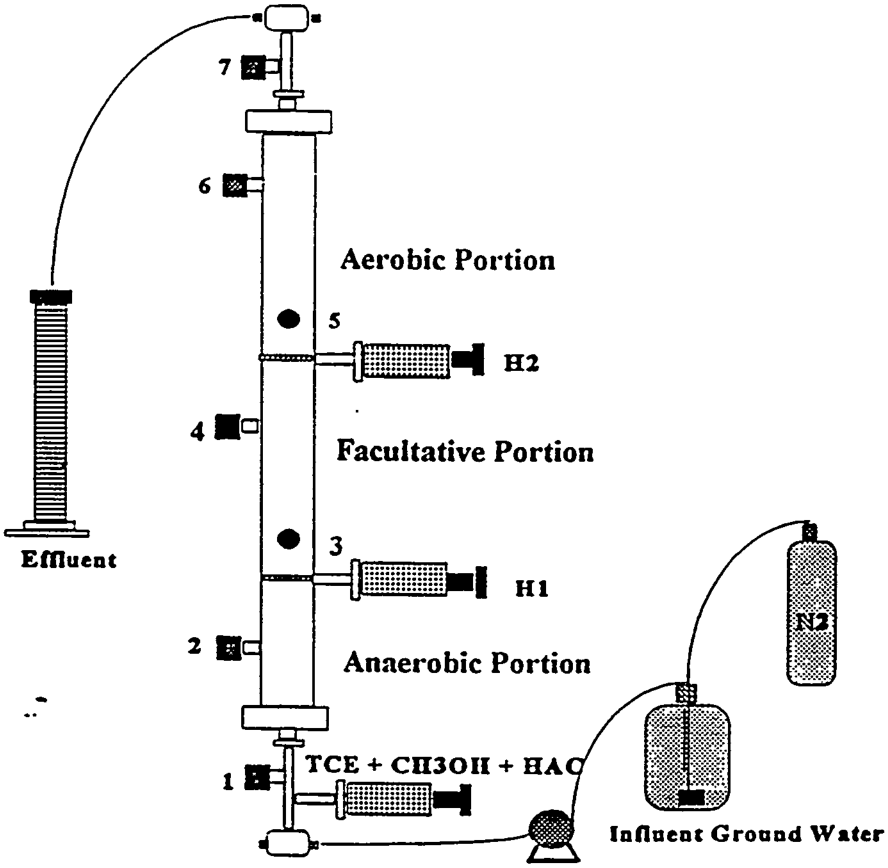


Figure 7. Schematic Diagram of the Simultaneous Column Setup

Table 8. Simultaneous Column Anaerobic Zone Operational Conditions (Concentrations, mg/L)

Chemical	7/1-7/14	7/15-7/28	7/29-8/4	8/5-9/30
NH_4Cl	28.35	28.35	28.35	28.35
K_2HPO_4	2.92	2.92	2.92	6.05[3]
KH_2PO_4	2.11	2.11	2.11	4.48[3]
$MgCl_2.6H_2O$	5.76	5.76	5.76	5.76
$MnCl_2.4H_2O$	1.92	1.92	1.92	1.92
$CoCl_2.6H_2O$	3.84	3.84	3.84	3.84
$ZnCl_2$	1.92	1.92	1.92	1.92
H_2SeO_3	0	0.08[1]	0.08	0.08
$CaCl_2.2H_2O$	6.34	6.34	6.34	6.34
H_3BO_3	0.58	0.58	0.58	0.58
$NiCl_2.6H_2O$	1.92	1.92	1.92	1.92
$Na_2MoO_4.2H_2O$	0.768	0.768	0.768	0.768
L-Cysteine	24.0	24.0	24.0	24.0
$Na_2S.9H_2O$	24.0	24.0	24.0	24.0
$NaHCO_3$	76.8	76.8	76.8	76.8
Yeast Extract	5.0	5.0	5.0	5.0
$FeCl_2•4H_2O$	6.0	6.0	6.0	6.0
Vitamin B-12	4.81	4.81	4.81	4.81
Methanol	800	800	800	800
H_2CO_3	528	528	0[2]	0
TCE	20	20	20	20
HRT, hrs	16	16	16	16

[1] To provide additional trace metals based on literature review.
[2] To return pH to acceptable range.
[3] To provide additional pH buffering.

Table 9. Simultaneous Column Facultative Zone Operational Conditions

Chemical	7/1 -7/15	7/16 - 7/23	7/24 - 9/30
H_2O_2, mg/L	110	100[1]	110[2]
CH_3OH, mg/L	57	57	57
HRT, hours	8	16	16

1 Evaluate toxic effects on microorganisms
2 To re-establish positive effluent DO (1-2 mg/L)

Table 10. Simultaneous Column Aerobic Zone Operational Conditions

Chemical	7/1 -7/15	7/16 - 7/23	7/24 - 9/30
H_2O_2, mg/L	332	306[1]	337[2]
CH_3OH, mg/L	60	60	60
HRT, hours	8	16	16

1 Evaluate toxic effects on microorganisms
2 To re-establish positive effluent DO (1-2 mg/L)

The simultaneous column has continued to out perform the sequential column yielding higher percent destruction for TCE. The following tables summarize the column concentrations with overall TCE removal rate ranging from 68-89%, as shown in Table 11. Changes in the operation of the simultaneous column have been limited to replacing sodium carbonate with phosphate buffer as with the sequential column, and determining if the peroxide injection concentrations were to high. It became apparent that after reducing the peroxide concentrations no improvement, in fact an increase in remaining Dps (Table 12), resulted so initial peroxide levels were reinstated. Once again do to GC failures no additional work was possible for the simultaneous column. Despite the lack of good GC analysis TOC's were made to determine the overall health of the column. Good TOC destruction was recorded through out the column (Figure 10) toward the end of this quarter (after pH had been stabilized). hanging the ground water flow to provide a 48 hr HRT, and a 10% increase in the peroxide concentration in the aerobic zone.

Table 11. Percent Removal throughout the Simultaneous Column.

	7/1-7/3	7/3-7/9	7/10-7/12	7/13-7/18	7/19-7/26	7/27-7/28	7/29-8/5	8/6-8/11	8/12-8/19
%TCE Removal Anaerobic Zone	83	63	74	82	70	59	75	69	70
%TCE Removal Facultative Zone	4	35	12	29	-3	16	9	-16	72
%TCE Removal Aerobic Zone	28	-11	21	-24	32	12	-3	-12	-54
Overall % Removal	88	73	82	84	79	69	77	60	87

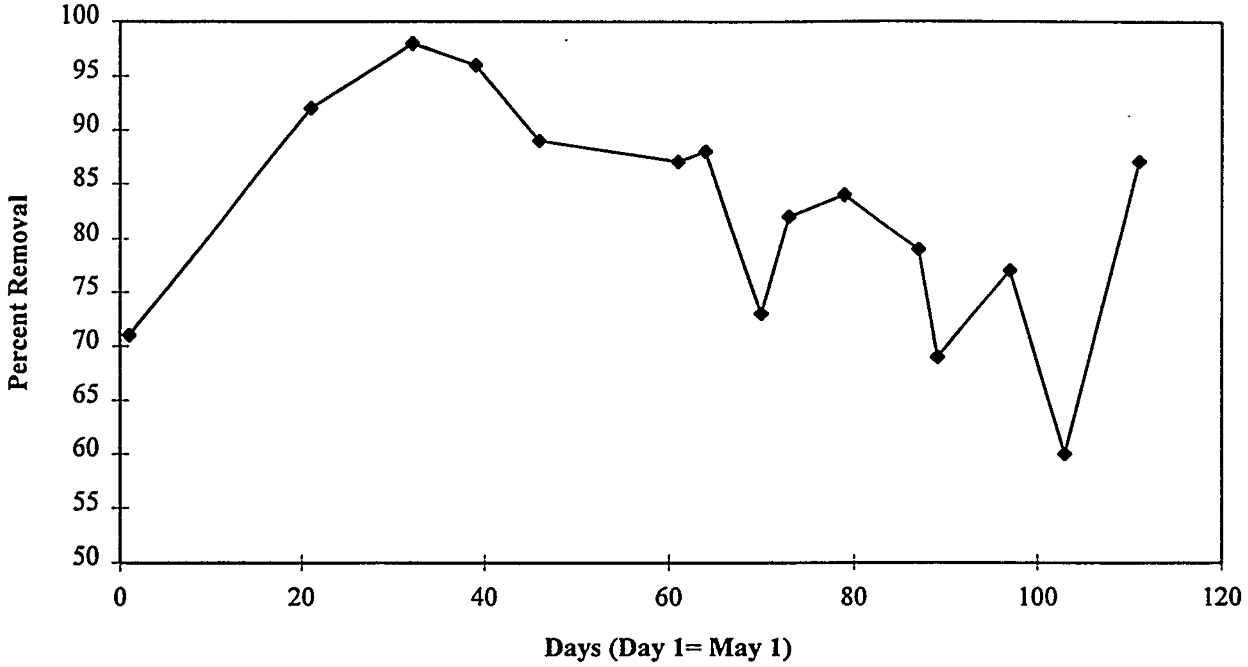


Figure 8. Simultaneous Column Percent TCE Removal

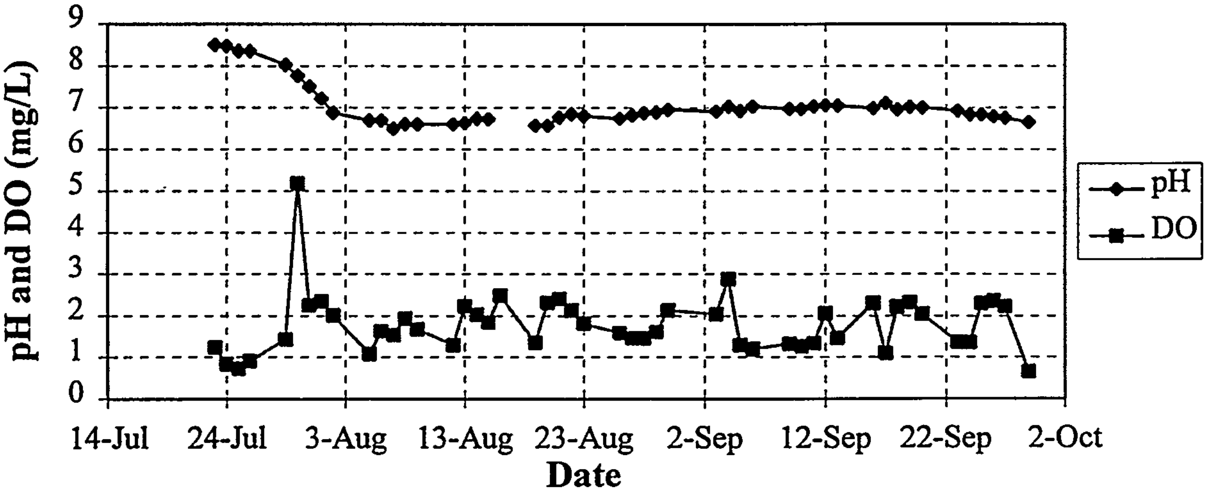


Figure 9. Simultaneous Column Effluent pH and Dissolved Oxygen

DEVELOPMENT AND IMPLEMENTATION OF A DECISION SUPPORT SYSTEM FOR MANAGEMENT OF THE EM50 TECHNOLOGY DEVELOPMENT PROGRAM
METC SUBTASK 1.7A

Quarterly Technical Progress Report
Reporting Period: July 1, 1996-September 30, 1996

Work Performed Under Contract
No.: DE-FC21-92MC29467

For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By:
Waste Policy Institute
Virginia Tech

DISCLAIMER

ABSTRACT

It has become apparent that there is a need for a means to organize and catalog various activities in the Office of Science and Technology Program in such a way that decision makers can take full advantage of the portfolio of R&D projects to meet the needs of the DOE Weapons Complex cleanup. An effort has begun to provide such a means and has proceeded to the point where a database system has been designed, but not fully implemented. The purpose of the present effort is to take a relational database system (implemented in Microsoft Access) that has an application front end written in Visual Basic and provide a useable product to the Department of Energy by October 15, 1996. The final product will include the database system with its front end application fully populated with data from one of the focus areas (Subsurface Contaminants). A decision tool process will be provided to assist in analysis and prioritization of the technology development portfolio being sponsored by the DOE EM-50 program.

To that end, the current effort involves three major tasks: 1. examine the data set that exists for the Subsurface Contaminants Focus Area and correct and complete information pertaining to all projects; 2. make a critical assessment of the coding for the application front end to the database; and 3. assess, recommend, and implement decision tools that provide the rigorous analyses needed to build justifiable, documented group decisions about the R&D portfolio.

Follow-on activity will include the further population of the database with the other 4 focus areas' data sets.

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5.0 WORK PERFORMED THIS PERIOD

The WPI Team continued efforts with Virginia Tech and West Virginia Universities, and DOE Savannah River personnel to develop a prototype system to support decision-making efforts for the Subsurface Contaminants Focus Area (SFCA). The three groups concentrated on: (1) collecting and entering data sets into the database, (2) refining database development, and (3) developing Decision Support System (DSS) tools.

At the United States Air Force's request, the WPI Team presented a prototype demonstration of the data base and proposed DSS tools to Human Systems Center (HSC) personnel from Brooks AFB, TX, on July, 24, 1996, at Virginia Tech University in Blacksburg, VA. As the result of this meeting, the WPI Team met with Air Force and Morgantown Energy Technology Center (METC) personnel on August 14, 1996, in Morgantown. The Air Force explained its current Technology Needs Assessment database system and its needs for a "next generation" system. They would like to continue to track development of the database and DSS tools to determine if the system can meet Air Force "next generation" needs.

WPI Team personnel in Aiken presented a prototype database demonstration to DOE Savannah River on August 13, 1996. DOE personnel were pleased with progress on the system, and surfaced some new issues, such as Internet access and database maintenance, which may be considered for follow-on work.

Another meeting was held in Blacksburg on August 30, 1996, to discuss the application of DSS tools, data linkages, and potential follow-on customer requirements. The importance of building reports and queries into the system to support decision making was discussed. This functionality is necessary to give the client the flexibility to use the system for specific decision-making analyses without support of the Analytical Hierarchy Process (AHP) DSS tools. Aiken personnel will ensure that the right reports and formats are generated to meet client needs.

During the week of September 8, WPI Team (Aiken) personnel presented an updated prototype demonstration to DOE Savannah River which included more extensive and complete data sets. DOE personnel were again pleased with progress and were awaiting delivery of the prototype software and documentation on September 23.

WPI Team personnel delivered the prototype software and system documentation to DOE Savannah River on September 23, 1996. WPI Team (Aiken) personnel presented a demonstration of the system at a DOE staff meeting on September 23 and at a Program Manager's Conference on September 26. DOE personnel were impressed with the system capabilities and plan to demonstrate the system to Headquarters DOE in Washington, DC. WPI Team (Aiken) personnel will present a more formal training session to DOE Savannah River after October 1, 1996.

Specific work performed by task during this reporting period follows:

Task 1 - Subsurface Contaminant Data Validation

Data from the current SFCA program information was provided to Virginia Tech University personnel during the last reporting period. Since two Focus Areas (Plumes and Landfills) were combined to form the Subsurface Contaminants Focus Area, efforts continued to ensure that all data had been collected and validated.

Task 2 - Data Assembly

Virginia Tech and WPI Team (Aiken) devoted significant efforts to assembling data into appropriate data sets and providing the proper linkages between the data sets to conform to the "Requirements Analysis and Design for the EM-50 Decision Support System" report developed and published by Virginia Tech on June 24, 1996. An Entity-Relationship model and relational model were developed to generate and link required data. Several iterations of data relationships evolved to ensure required linkages were available to generate needed reports and queries.

Task 3 - Data Entry

After concurrence on the models described in Task 2, WPI Team (Aiken) personnel began to provide data to Virginia Tech to populate the data base. Significant efforts were made to ensure all data tables were populated with current and correct data.

Task 4 - Existing Database Application

As noted on the last quarterly report, Virginia Tech personnel conducted an extensive analysis of the TechInvest system. Their report, " Documentation of TechInvest Prototype 1.0b (Beta)" documents their findings.

Task 5 - Database Development

Based on the specifications developed in the "Requirements Analysis and Design for the EM-50 Decision Support System" report published by Virginia Tech on June 24, 1996, Virginia Tech personnel embarked on full scale development of the new database. Virginia Tech focused the relational database to store information related to the following:

- waste management and environmental restoration problems,
- technology needs classified by different categories,
- technologies available from industry,
- technology needs unsatisfied by currently available technologies (technology gaps),
- technology development programs to meet unsatisfied technology needs, and
- relationships that exist between all of the above data.

Database development also considered who the users of the system were and what type of decisions needed to be made. Four categories of users for the system were identified:

- DOE - expected to fully utilize all components of the system,
- DOE site operators - expected to utilize the reporting and data entry capabilities of the system,
- DOE contractors - expected to use the reporting capabilities of the system, and
- Stakeholders/Public - expected to use the reporting capabilities of the system.

Extensive efforts were made to collect proper data define data relationships to ensure needed reports and queries could be generated by the users.

Task 6 - Database Demonstration

WPI Team personnel presented a prototype demonstration of the database to DOE Savannah River on August 13, 1996. DOE personnel were pleased with the progress and focus of the system.

Task 7 - Final Prototype Development

The prototype database software and system documentation were delivered to DOE Savannah River on September 23. WPI Team (Aiken) personnel delivered two prototype demonstrations to DOE personnel during the week of September 23. Aiken personnel will present a formal training session to DOE Savannah after October 1. Based on the feedback from this session, Virginia Tech will make final modifications to the database and documentation. The WPI Team will deliver the final prototype products on October 15.

Task 8 - Decision Support System (DSS) Development

In concert with database development, Virginia Tech personnel began developing a decision model that:

- matched available technologies to problems,
- aided in determining which technology development programs would be most beneficial in terms of cost, expediency, safety, and effectiveness, and
- generated reports and provided query capabilities consistent with the objectives of the system.

The DSS considered the same four categories of users as the database. Decisions related to the system would be made primarily by the DOE group of users. The primary decisions made by DOE personnel include:

- Deciding which technologies available apply to a particular problem. The applicable technologies are ranked base on criteria that include Worker Health and

Safety, Public Health and Safety, Environmental Protection, Regulatory Compliance, and Cost.

- Deciding where there are technology gaps and which technology development programs match the gaps based on the five criteria above. Additional criteria could be developed if desired or appropriate.

Task 9 - DSS Prototype Development and Demonstration

A meeting was held in Blacksburg on August 30 to discuss the application of DSS tools. Prototype development proceeded on schedule and was demonstrated to DOE Savannah River personnel during the week of September 23. DOE personnel were impressed with the system capabilities and plan to demonstrate the system to DOE Headquarters in Washington, DC.

WPI Team (Aiken) personnel will conduct a system training session for DOE Savannah River after October 1.

Task 10 - Prototype Modifications

After the October 1 training mentioned in Task 9, Virginia Tech will modify the prototype system to meet customer requirements.

Task 11 - DSS Full Database Extension

Based on customer feedback from the October 1 training session, Virginia Tech will modify the database to provide the requested DSS capabilities.

Task 12 - DSS/Database Test & Demonstration

The October 1 training session will include a DSS/Database Test & Demonstration. Based on requested system modifications, Virginia Tech will provide another demonstration if requested by the customer.

6.0 RESULTS AND DISCUSSION

Interim prototype demonstrations provided in August and September indicated that the DSS provides the functionality and flexibility requested by the customer. The October 1 training session will provide system access to a larger audience of users. Requests for system modifications may result as more users test the capabilities of the system.

7.0 CONCLUSIONS

The WPI Team expects to deliver the final system on October 15, on time and within budget.

8.0 WORK PLANNED FOR NEXT PERIOD

The only work remaining is the formal training session to be conducted by WPI Team (Aiken) personnel on October 1. If any modifications are requested to the system, Virginia Tech will make these modifications. The WPI Team will deliver the final product on October 15.

9.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES

The WPI Team is experiencing no outstanding technical or administrative difficulties at this time.

DEVELOPMENT OF A PROTOTYPE DATABASE AND DECISION SUPPORT SYSTEM FOR MANAGEMENT OF THE EM-50 TECHNOLOGY DEVELOPMENT PROGRAM

METC TASK 1.7B

Quarterly Technical Progress Report
Reporting Period: July 1 - September 30, 1996

Work Performed Under Contract
No.: DE-FC21-92MC29467

For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By:
James W. Denton
College of Business and Economics
West Virginia University
Morgantown, West Virginia

ABSTRACT

The prototype system to be developed in this project will provide a database and a decision support system to assist decision makers in optimizing the portfolio of R&D projects relating to the clean-up of subsurface contaminants at DOE sites. The database will provide a means of organizing and cataloging pertinent information on specific clean-up problems, as well as information on existing and potential technologies that may be utilized to solve those problems. The decision support system will consist of a collection of quantitative tools to be used to suggest and evaluate possible solutions, subject to the decision maker's assessment of such factors as the importance of relevant information, the magnitude of associated risks, and the probabilities of rewards to be realized.

The work of this project will build on previous work to build an initial database and existing software to provide access to the database. A team of professors and graduate students from Virginia Tech and one professor from West Virginia University will carry out the work of this project.

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EXECUTIVE SUMMARY

A domain knowledge help system, to be run on-line on PC was developed to aid database designers in constructing the EM50-DSS prototype database. Acronyms help screens and glossary help screens were included. This document contains summaries of the help screens that are available in the system. An additional help system is being developed to provide on-line context sensitive help for users of the EM50-DSS database.

1.0 Introduction

The Department of Energy has initiated a project, under the name of TechInvest, to analyze, prioritize and select techologies to be funded and applied to waste clean-up problems at various DOE sites. Some initial work on the TechInvest project has been completed, and this project has been initiated to complete that work, providing a working prototype decision support system by September 30, 1996 for the Subsurface Contamination focus area. Work will be carried out by personnel at Virginia Tech (Professors Laurence Moore, Saifur Rahman, and Tarun Sen with graduate student assistance, as well as Professor James Denton at West Virginia University.

2.0 PURPOSE

This project will complete the work on the initial TechInvest project by validating and entering data to the database where required. In addition, decision support tools will be added to aid in the process of prioritizing and selecting technologies to be applied to surface contamination problems. The criteria used for decision making should be consistent with the "gate" criteria used to evaluate projects for advancement to succeeding development phases, and consider costs, benefits, and risks from the standpoint of the stakeholder.

The finished product will provide DOE with the capability to analyze funding decisions and justify their programs using sound analytical reasoning, minimizing decisions that may appear to be arbitrary or made on an ad-hoc basis.

3.0 BACKGROUND

DOE has identified the need for a means of assuring that investment made in research and development in the Environmental Management program will result in the optimal benefit toward cleaning-up the department's sites. The key to achieving this goal is through the development of a decision-making process for technology management that is customer-oriented, and can link the most appropriate technology development activities with site clean-up operations. The Technology Investment Decision Model [PALADINO] has been designed to assure that funding decisions are made at all stages of technology development in a manner consistent with technical and nontechnical criteria for performance and acceptance by the customer.

A relational database has been designed using Microsoft Access and partially populated with data on surface contamination problems and applicable technologies. A user interface, written in Visual Basic, has also been designed to allow decision makers to enter, edit, and add data, as required, to the existing database. There are currently 5 separate databases in TechInvest, with partial data from the Subsurface Contaminants focus area. The data require validation, with updating required in certain cases, and missing data provided in other cases. There are some operational difficulties (bugs) with the Visual Basic interface which require fixing so that accurate reports may be generated and new data may be entered..

4.0 METHODOLOGY

Preliminary activities will consist of examining the existing data set, revising data where applicable, and adding data to incomplete sections. Appropriate relational links are needed to complete the logic structure of the database. The user interface, which allows the database to be queried, provides access to enter and revise data, and generates reports, needs to be checked for coding errors and debugged. The result of this preliminary effort will be a database system populated with a significant subset of site-specific data and technology data from the Subsurface Contaminants focus area.

The database will be used in conjunction with decision support tools to allow a decision-maker to select the most appropriate technology development portfolio to deal with subsurface contamination problems in the DOE EM-50 program. The decision support tools will allow decision makers to analyze alternate technologies, prioritize alternatives, and provide justification for funding decisions.

Research will be undertaken to select appropriate decision support tools for this task. First, there must be an understanding of the current decision-making process, as well as perceived needs and desires for improvements in the current decision-making process. The most appropriate methodology or methodologies will be selected from available decision support techniques. Eventually, all focus areas will be represented in the database.

The performance of the DSS will be assessed at two intermediate prototype stages prior to project completion. The initial prototype will demonstrate the interface between the database and the user, but will not utilize the full database. Based on client input, specific functions and features will be added to the DSS to produce an intermediate prototype. The intermediate prototype will then be demonstrated to and evaluated by the client. Based on client feedback, the prototype will be further refined and modified. In addition to the suggested modifications, the final product will include linkage to the full database. The client will therefore be intimately involved during the development process to assure that the resulting DSS provides the required functionality.

5.0 WORK PERFORMED THIS PERIOD

Two on-line help systems were developed during this period. The first system, which is complete was designed to provide database developers with domain knowledge about the Department of Energy and the Environmental Management program. The second system, which is currently available as a prototype, was designed to provide users of the EM-50 database with online context-sensitive help.

The first system consists of three executable files, running under Microsoft Windows 3.1 or Microsoft Windows 95. The first of these was developed in order to provide domain expertise to database developers working at Virginia Tech. There are three executable files in this system: the base file (technvst.hlp), a glossary (glossary.hlp), and an acronym file (acronym.hlp). The glossary and acronyms are available from within the base help file as pop-up windows.

This system looks and operates like other help systems that are routinely provided for Microsoft Windows software products. Standard control buttons are provided to close the window or reduce the window to an icon. Drop-down menus are also provided. Other features include:

- Hypertext links to related topics
- User annotations
- User bookmarks
- Navigation through a history list
- Defined browse sequences
- Keyword search capability
- Graphics with hotspot links

The help system contains information on EM organization, EM planning documents, the technology investment decision model (stage/gate process), technology development activities and procedures, DOE sites and facilities, a glossary of terms, and a list of acronyms.

6.0 RESULTS AND DISCUSSION

The following pages contain a summary of topics and their contents from the online help system. Double underlines indicate links to topics, and single underlines indicate a pop-up glossary or acronym window.

Table of Contents

Office of Science and Technology [See Organizational Structure]

The Office of Science and Technology (EM-50),formerly called the Office of Technology Development, has the overall responsibility to develop technologies to meet DOE's 30-year goal for environmental restoration. The office works closely with EM-30, EM-40, and EM-60 identifying, developing, and implementing innovative and cost-effective technologies. Activities within EM-50 include:

- Applied research and development (EM-53)
- Demonstration, testing, and evaluation (DT&E) of new and existing technologies (EM-54)
- Technology integration and program support (EM-52)

Office of Environmental Management [See Organizational Structure]

The Office of Environmental Management (EM) is responsible for managing the cleanup of DOE wastes from past nuclear weapons production and current operations. The EM mission is to minimize risks to the environment, human health, and safety posed by the generation, handling, treatment, storage, transportation, and disposal of DOE waste, and to bring DOE sites into compliance with all environmental regulations by the year 2019. The EM organization was established to provide visibility, focus, and accountability for DOE's waste management and remediation efforts.

Major offices within EM include:

- EM-30: The Office of Waste Management
- EM-40: The Office of Environmental Restoration
- EM-50: The Office of Science and Technology
- EM-60: The Office of Facility Transition and Management

Office of Waste Management

The Office of Waste Management (EM-30) has program responsibilities for waste management at all DOE sites generated during weapons processing and manufacturing, research activities, and site cleanup activities, including the treatment, storage, and disposal of several types of waste: transuranic, low-level radioactive, mixed, and solid sanitary wastes. EM-30 is also responsible for the storage, treatment, and processing of defense high-level radioactive waste, waste minimization efforts, and corrective activities at waste management facilities.

Idaho Sites

Installation	**Location**
Component Development & Integration Facility	Butte, Montana
Idaho Chemical Processing Plant	Idaho Falls, Idaho
Idaho National Engineering Laboratory	Idaho Falls, Idaho
West Valley Demonstration Project	West Valley, New York

Fernald Sites

Installation	**Location**
Fernald Environmental Management Project	Fernald, Ohio

Chicago Sites

Installation	Location
Ames Laboratory	Ames, Iowa
Argonne National Laboratory-East	Chicago, Illinois
Argonne National Laboratory-West	Idaho Falls, Idaho
Battelle Columbus Labs Decom. Project	Columbus, Ohio
Brookhaven National Laboratory	Upton, New York
Fermi National Accelerator Laboratory	Batavia, Illinois
Hallam Nuclear Power Facility	Lincoln, Nebraska
Piqua Nuclear Power Facility	Piqua, Ohio
Princeton Plasma Physics Laboratory	Princeton, New Jersey
Site A/Plot M	Cook County, Illinois

Albuquerque Sites

Installation	Location
Grand Junction Projects Office	Grand Junction, Colorado
Inhalation Toxicology Research Institute	Albuquerque, New Mexico
Kansas City Plant	Kansas City, Missouri
Kauai Test Facility	Kauai, Hawaii
Los Alamos National Laboratry	Los Alamos, New Mexico
Mound Plant	Miamisburg, Ohio
Pantex Plant	Amarillo, Texas
Pinellas Plant	Largo, Florida
Sandia National Laboratories-Albuquerque	Albuquerque, New Mexico
Sandia National Laboratories-Livermore	Livermore, California
Sandia National Laboratories-Tonopah	Tonopah, Nevada
South Valley Site	Albuquerque, New Mexico
Waste Isolation Pilot Plant	Carlesbad, New Mexico

Savannah River Sites

Installation	Location
Savannah River Site	Aiken, South Carolina

Oakland Sites

Installation	Location
General Atomics	La Jolla, California
General Electric Vallecitos Nuclear Center	Pleasanton, California
Laboratory for Energy-Related Health Research	Davis, California
Lawrence Berkeley Laboratory	Berkeley, California
Lawrence Livermore National Laboratory	Livermore, California
Energy Technology Engineering Center	Canoga Park, California
Stanford Linear Accelerator Center	Palo Alto, California

Rocky Flats Sites

Installation	Location
Rocky Flats Plant	Golden, Colorado

Richland Sites

Installation	Location
Hanford Reservation	Richland, Washington

Oak Ridge Sites

Installation	Location
Oak Ridge K-25 Site	Oak Ridge, Tennessee
Oak Ridge National Laboratory	Oak Ridge, Tennessee
Oak Ridge Y-12 Plant	Oak Ridge, Tennessee
Paducah Gaseous Diffusion Plant	Paducah, Kentucky
Portsmouth Gaseous Diffusion Plant	Portsmouth, Ohio
Weldon Spring Site Remedial Action Project	St. Louis, Missouri
Center for Energy and Environmental Research	Puerto Rico

Nevada Sites

Installation	**Location**
Nevada Test Site	South Central Nevada
Nevada Off-Site Locations:	
• Amchitka Island	Amchitka Island, Alaska
• Central Nevada Test Site	Central Nevada
• Gasbuggy Site	Farmington, New Mexico
• Gnome-Coach Site	Carlsbad, New Mexico
• Rio Blanco Site	Rifle, Colorado
• Rulison Site	Rifle, Colorado
• Shoal Site	Fallon, Nevada
• Tatum Dome Test Site	Hattiesburg, Mississippi
Tonopah Test Range	Nellis Air Force Base, Nevada

Sites/Facilities

Sites/Facilities Where Environmental Restoration and Waste Management Activities Are Being Conducted

Field Offices

- Albuquerque
- Chicago
- Fernald
- Idaho
- Nevada
- Oak Ridge
- Richland
- Rocky Flats
- San Francisco
- Savannah River

Other Sites

- FUSRAP Sites

UMTRA Sites

Strategic Plan

The EM Strategic Plan develops EM-wide strategic objectives, establishes EM long-term vision and mission, and outlines strategies for addressing issues and meeting goals. It describes the framework for subsequent planning efforts at the Headquarters and installation levels. It is prepared annually with input from Headquarters, Field Offices, and other Program Secretarial Offices.

The EM Strategic Plan sets the overarching policy framework for all EM planning and is responsive to DOE-wide planning requirements set by SEN-25A, the Strategic Planning Initiative. It provides direction for preparation of the Five-Year Plan. Roadmaps constitute a critical input to the annually-updated EM Strategic Plan.

Site-Specific Plans

SSPs provide details on activities, milestones, accomplishments, and funding requirements for each site. They focus on execution year activities, and are prepared annually by the Field Offices, with Headquarters review.

SSPs are linked to the Five-Year Plan, Activity Data Sheets, and the Progress Tracking System. The EM Integrated Planning Process will tie SSPs to Strategic Plans.

Secretary of Energy Notice 25-A

SEN 25A integrates planning, programming, and budgeting in a functional manner. It mandates six products that collectively underpin the yearly planning process: strategic plans, guidance to the field, multi-year program plans, crosscut plans (as assigned), program issues, and program planning proposals.

The Strategic Planning Initiative provides policy framework for all EM planning activities. The EM Five-Year Plan satisfies SEN 25A planning requirements.

Safety and Health Five-Year Plan

The EM Safety and Health Five-Year Plan identifies and prioritizes EM-wide Safety and Health activities. Combined with similar plans from other programs, it forms the DOE-wide Safety and Health Five-Year Plan. For EM, safety and health data is a subset of the EM Activity Data Sheets.

Remedial Action Assessment System

The purpose of RAAS is to develop and test a computer-based methodology for identifying, linking, and evaluating technologies for DOE sites. The methodology is being developed to standardize and compress the remedial investigation / feasibility study process. Once fully developed, RAAS would be a key tool in the development of the Environmental Restoration Program baselines and project plans.

Roadmaps

Roadmaps help managers respond to critical environmental, health, and safety issues at DOE sites by identifying issues or obstacles that might impede EM progress and developing strategies for their resolution. Their scope ranges from site-specific waste stream issues to crosscutting policy questions.

Roadmaps set the context for planning activities and feed national program planning, such as technical development, transportation, or waste-stream planning. Issues and strategies identified by roadmaps are incorporated into the Five-Year Plan.

Progress Tracking System

The purpose of the PTS is to maintain a system for reporting EM cost, schedule, and technical progress in a way that can be linked to planned activities as presented in the Five-Year Plan. All activities funded by EM are reported through the PTS. It is the primary EM system for monthly status reporting. Reports are submitted monthly at the Activity Data Sheet or Technical Task Plan level through the Field Offices. They contain information on planned costs, planned milestones, and their status.

The PTS reports on activities described in the Activity Data Sheets, which back up the Five-Year Plan for Environmental Restoration and Waste Management Programs. Budgets for these activities are revised through the IRB and OMB process and set through the Approved Funding Plan. For the Technology Development Program, the Technical Activity Data Sheets presented in the Five-Year Plan are further defined into component Technical Task Plans during the budgeting year.

Programmatic Environmental Impact Statement

The PEIS will analyze reasonable alternatives to siting DOE waste management facilities and will describe related environmental impacts. Based on this analysis, changes may be made to future site projects and activities. Public input is provided through scoping meetings and workshops. An EM Advisory Commitee will review the draft PEIS.

Results from the PEIS process will directly affect scope of Five-Year Plan as well as scope of activities at specific DOE installations.

Program Management Plans

These documents establish requirements, procedures, and systems by which program missions will be accomplished. They include activities of Headquarters program staff and support contractors, and serve as implementation documents for site-level activities.

EM/EH Memorandum of Understanding

The purpose of the MOU between the Office of Environmental Management and the Office of Environmental Safety and Health is to delineate the respective roles of the two organizations and to specify areas of interrelationship. This document is currently under development. EH is working on a draft version to be used as a source of negotiation. When completed, it will specify clear lines of authority, and reconcile overlapping planning activities in EM and EH.

Five-Year Plan

The EM Five-Year Plan is the primary planning tool for regulatory compliance, waste management, environmental cleanup, and technology development activities at DOE sites. It provides a detailed description of the EM program for internal and external goals, objectives, strategies, plans, and accomplishments.

The Five-Year Plan is produced annually. It presents EM's Strategic Plan; describes Environmental Restoration, Waste Management, Technology Development, Facility Transition, and Transportation program plans and accomplishments; and summarizes EM activities at the installation level. The plan presents financial data compiled from the Activity Data Sheets for the execution, budget and planning year, and for the four following years.

The five-Year Plan is supported by Activity Data Sheets, which are evaluated by independent cost reviews. Roadmaps at the installation level form the general framework for more detailed five-year planning. Program baselining and prioritization activities feed into the plan. It acts as a basis for Site-Specific Plans, which give further details for each installation. The Five-Year Plan receives extensive input from a number of sources: internal EM and DOEDOE@Acronym.hlp programs, State and Federal regulators, OMB, Congress, the general public, and other organizations and affected parties.

Final Report on DOE Nuclear Facilities

The purpose of this report is to assess the DOE nuclear complex and present recommendations to the Secretary on critical DOE-wide issues. The Committee highlighted nine issue areas: Risk Analysis, Research Reactors, Process Facilities, Environmental Cleanup, Radiation Protection, Rocky Flats Plant, Waste Isolation Pilot Plant, Safety Policy, and Other Activities.

This report contributes to the development of land-use planning and risk management initiatives articulated in the FY 1994-1998 Five-Year Plan.

DOE Orders

These orders are issued by the Secretary of Energy. They establish policies and guidelines for management of DOE nuclear facilities, including cost and scheduling criteria and requirements for outlay program acquisitions. They drive ADS and Five-Year Plan estimates and activity descriptions, as well as other management initiatives discussed in the plan.

Defense Authorization Act

Section 3135 mandates a Five-Year Plan for Environmental Restoration and Waste Management activities of DOE nuclear facilities. This act drives the Five-Year Plan content and production schedule. It also requires production of EM preliminary and final versions with formal public comment periods.

Budget Activities

The purpose of these documents is to prepare the EM program budgets for DOE, OMB, and Congressional review. Budgets are prepared annually in a two-step process. First, an IRB is prepared for internal DOE review in the summer. Then, it undergoes an external OMB review in the fall. The budget is then submitted to Congress by the President the following January.

Activity Data Sheet preparation for the Five-Year Plan forms the starting point for the IRB preparation.

Activity Data Sheet

The purpose of the ADS is to provide basic planning data that describe EM activities by site. Used in development of Five Year Plan and EM Budget. Also forms reporting basis in the Progress Tracking System. ADSs include scope of work, costs, schedules for each activity, regulatory drivers and milestones.

ADSs are developed annually by Field Offices and finalized by joint Headquarter/Field review process. All activities identified for funding under the EM program are described by ADSs. State and Federal regulators, Office of Management and Budget, Congress, general public, and other interested parties review completed ADSs.

ADSs form the basis for the Five-Year Plan, Site-Specific Plans, and other aspects of EM planning.

EM Planning Documents

Activity Data Sheet (ADS)
Budget Activities [Internal Review Budget (IRB) and Office of Management and Budget (OMB) budget process)
Defense Authorization Act
DOE Orders 2250.1C, 4700.1, 5820.2A
Final Report on DOE Nuclear Facilities by the Advisory Committee on Nuclear Facility Safety (Ahearne Committee)
EM Five-Year Plan
EM/EH Memorandum of Understanding
Program Management Plans
Programmatic Environmental Impact Statement (PEIS)
Progress Tracking System (PTS)
Roadmaps
Remedial Action Assessment System (RAAS)
EM Safety and Health Five-Year Plan
Secretary of Energy Notice 25-A "Strategy Planning Initiative"
Site-Specific Plans (SSPs)
EM Strategic Plan

Robotics

The Robotics Technology Development Program (RTDP) has a three-fold mission: 1) maximize worker safety in hazardous environments through remote operation and control of equipment; 2) increase productivity through enhanced capabilities and automation; and 3) reduce costs by developing more productive systems which expedite completion of remediation operations and decrease life cycle costs.

RTDP is organized around five technical application areas which develop a broad range of robotics technologies. The technical areas and their major projects are listed below.

- Tank Waste Retrieval (Advanced Long Reach Arm; Dextrous Manipulators; 3-D Workspace Mapping System)
- Contaminant Analysis Automation (Automated Laboratory)
- Mixed Waste Operations (Stored Waste Drum Inspection; AC SwingFree Crane Control; Automated Workcell Model Updating; Container Opening, Sorting, and Transportation

System)
- Decontamination and Dismantlement (Mobile Characterization System; Selective Equipment Removal; Small Pipe Characterization; Internal Duct Characterization)
- Crosscutting and Advanced Technology (Generic System Controls; Sensors; Mini-lab)

Innovative and Crosscutting Programs

Innovative and crosscutting programs include the following:

- Characterization, Monitoring, and Sensor Technology
- Efficient Separations Processing
- Robotics

Decontamination and Decommissioning Facilities

The aging of the DOE Complex's facilities, along with the reduction in nuclear weapons production, have resulted in 1,200 facilities being shut down at multiple field locations that require deactivation, decommissioning, and disposition. The inventory of permanent structures that require deactivation is projected to grow to about 20,000 permanent and temporary buildings and structures. Current cleanup processes tend to expose workers to radioactive and hazardous substances. They are labor-intensive, and expensive, producing an unacceptably large volume of secondary waste. Additionally, in the near term, high costs for shutdown, stabilization, surveillance, and maintenance will absorb the majority of dollars budgeted for decommissioning. Similar problems exist for the commercial nuclear power industry, other government facilities, and commercial facilities.

The Decontamination and Decommissioning (D&D) of Facilities Integrated Demonstration was initiated to develop and demonstrate improved processes for:

- Facility shutdown and stabilization
- Surveillance and maintenance
- Sampling, imaging, and characterization
- Decontamination and dismantlement
- Recycling and disposition

The primary objectives of the program are to reduce the costs of D&D to affordable levels and to ensure effective, long-term technical solutions.

Rocky Flats Compliance Program (RFCP)

The Rocky Flats Environmental Technology Site (RFETS), is the site of a former nuclear weapons components manufacturing. The treatment and disposal of mixed waste resulting from these manufacturing activities is driven by agreements between DOE, the EPA, and the Colorado Department of Health. Under these agreements, DOE will treat MLLW, and set specific milestones to develop new technology to treat the RFETS mixed waste.

Incineration and cementation techniques are the baseline processes used to treat hazardous waste. RFCP is investigating a wide variety of alternative treatment systems which produce better waste forms, improve process efficiency, achieve cost savings, and respond to regulator and public concerns to bring the RFETS into compliance with the regulatory agreements. In addition to developing treatment technologies, RFETS is demonstrating, testing, and evaluating systems to reduce the amount of mixed waste requiring disposal.

Mixed Waste Treatment

Approximately 169,000 cubic meters of mixed LLW is stored at DOE sites. Generation of mixed waste continues at the rate of 56,000 cubic meters per year. The problem is significant because definitive treatment standards have not been established and disposal facilities designated for mixed waste are not available. In addition, treatment capabilities and capacities are very limited. Therefore, DOE sites must store mixed waste for future disposal.

Applicable state and federal regulations for hazardous and radioactive waste are often inconsistent, complicating the development of treatment and disposal methods. Despite the lack of clear disposal criteria, compliance agreements specify deadlines for remediation. This regulatory quandary forms a challenging framework for developing and implementing a mixed waste technology RDDT&E program.

The Mixed Waste Integrated Program (MWIP) is linked to environmental restoration and waste management needs to ensure the development of treatment capabilities. MWIP addresses the requirements of RCRA for EM-30 projects, and the requirements of CERCLA for EM-40 projects. EM-50 conducts R&D to develop a suite of technologies for treating mixed waste to acceptable criteria and to provide design and reliability data to EM-30 and EM-40 within the required schedule.

Over 1.400 mixed waste streams have been classified into categories requiring similar processing steps. Baseline treatment schemes for each waste category have been organized to reflect the major steps in mixed waste processing: front-end handling, physical/chemical treatment, waste destruction, off-gas treatment, and final forms. Rather than developing isolated technologies, technologies are being devised to operate as coordinated systems, covering all of the steps.

Buried Waste

DOE and its predecessor agencies have operated numerous facilities throughout the United States for nuclear and non-nuclear research, development, and production. As a result of these operations, numerous sites throughout the DOE Complex have waste buried in the ground or stored for future disposal. Early disposal practices allowed the co-mingling of various types of waste. Much of this buried waste includes TRU waste, LLW, hazardous waste, mixed TRU waste, and MLLW. In addition, many of the original containers have degraded significantly, resulting in contamination of adjacent soil.

In order to identify technology deficiencies, and to develop as well as demonstrate appropriate technologies, the Buried Waste Integrated Demonstration (BWID) has been established at INEL. INEL was selected because about 30% ot the DOE Complex's buried TRU waste is located there.

The waste is made up of construction and demolition materials (lumber, concrete blocks, steel plates, etc.), laboratory equipment (hoods, desks, tubing, glassware, etc.), process equipment (heat exchangers, HEPA filters, valves, ion exchange resins, etc.), maintenance equipment (hand tools, cranes, oils and greases, etc.), and decontamination materials (paper, rags, plastic bags, etc.). Disposal containers included steel drums, cardboard cartons, and wooden boxes of various sizes. Since this is generally representative of other DOE buried-waste sites, remediation technology demonstrations performed here have significant potential for applications elsewhere.

Mixed Waste Landfill

Throughout the United States, and particularly in the Southwest, DOE used landfills to dispose hazardous and mixed wastes. The Mixed Waste Landfill Integrated Demonstration (MWLID) is evaluating new technologies to cleanup these chemical and mixed waste landfills. This is being accomplished by emphasizing in situ technologies that minimize landfill disturbance. These in situ technologies consist of:

- characterization technologies

- innovative extraction technologies
- containment and isolation technologies
- stabilization technologies
- chemical transformation technologies
- monitoring technologies

The demonstrated technologies will be evaluated against the baseline of conventional technologies and systems. The comparison will include the cost, efficiency, risk, and feasibility of using these innovative technologies at other sites. Key goals and measures of success of the MWLID are commercialization of these technologies to the private sector and routine use of these technologies by environmental restoration groups throughout the DOE complex.

MAWS

DOE has 16 sites listed on the EPA's National Priorities List. These sites have tank wastes, sludge pits, evaporation ponds, injection wells, and rubble pits contaminated with hazardous constituents and radioactivity that require remediation. The associated large amounts of contaminated soil and groundwater in the surrounding areas and other miscellaneous waste streams (fly ash, asbestos, transite, resins) also need to be remediated. Cementation (placing waste in cement) is largely ineffective over the long term. Vitrification, which makes waste into glass, is a technically sound alternative to cementation. However, the quantity of these waste streams is so high that the conventional energy-intensive vitrification approach, using 75% or more additives, would be very expensive.

Minimum Additive Waste Stabilization (MAWS) is an innovative approach to vitrification that provides an environmentally sound cleanup alternative for the large amounts of low-level radioactive/mixed waste that exists within the DOE Complex. At the heart of the typical MAWS process is vitrification, which incorporates all primary and secondary waste streams into a final, long-term, stabilized glass waste form using little or no additives. Additional technologies, such as thermal pretreatment, soil washing, gas scrubbing/filtration, and ion exchange may be required depending on the characteristics of the waste streams being treated.

Technical development of MAWS is proceeding in five main areas:

- Compositional envelope development for vitrified waste forms
- Development of processing technologies and system integration strategies
- Demonstration of the technologies at various DOE sites
- Life-cycle cost analysis to document savings
- Waste form performance assessment

Uranium in Soils

Past operations of uranium production have resulted in local radionuclide and/or toxic metal contamination of some surface and subsurface soils. The objective of the Uranium in Soils Integrated Demonstration is to develop optimal remediation methods for soils contaminated with radionuclides, principally uranium, at DOE sites. The cleanup phases will include characterization, excavation, decontamination, disposal, performance assessment, and regulation compliance.

Resource Recovery

Dissolved heavy metals and minerals in water pose a serious threat to aquatic systems and to human health. Some metals such as lead, mercury, and cadmium are neurotoxicants, while others form potential carcinogenic organometallics. One source of heavy metal contamination is acid mine drainage.

The Resource Recovery Project (RRP) develops technologies for reclaiming surface and groundwater, as well as metals and minerals from dilute heavy-metal aqueous solutions. The goal of the project is to complete this work and minimize waste byproducts by emphasizing technologies that recover valuable resources including water, precious metals, and industrial minerals, and then recycling them. .Industrial, commercial, agricultural, municipal, and recreational uses of these resources will be addressed.

Cost/benefit analyses will be an integral part of the RRP technology evaluation. These analyses will consider potential revenues generated from the sale of water and mineral resources. Pilot-scale demonstrations will collect data to determine the most efficient and cost-effective techniques for recovering resources and remediating contaminated waters. This will help to select the most appropriate reclamation technologies for specific DOE sites and other areas in the U.S. with heavy metal contaminated surface water and groundwater. In addition, these technologies will have application to hundreds of thousands of inactive or abandoned private mine sites throughout the U.S. The data will be applied to non-DOE sites through technology transfer.

Heavy Metals Contaminated Soils

Many DOE sites have metal-contaminated surface soils that present significant environmental problems. The cost of disposing large volumes of contaminated soil in land disposal facilities is high. Conventional technologies often produce waste volumes several times larger than in-place contaminated volumes. Without new technologies, projected costs could increase ten-fold. New cost-effective technologies are needed for heavy metal contaminated soils that address soil and vegetation removal, volume reduction, and waste disposal.

The Heavy Metals Contaminated Soils Project (HMCSP) will evaluate seven off-the-shelf technologies, routinely used by the mining industry, to conduct treatability studies on soils from DOE sites. These technologies include:

- Knelson Centrifugal Concentrator
- Carrier-Assisted Flotation
- Air-Sparged Hydrocyclone
- Campbell Centrifugal Jig
- U.S. Naval Academy Air Classification
- High Gradient Magnetic Separation System
- Dissolved Air Flotation (Denver Cell)

In Situ Remediation

Many environmental and waste management problems are located below ground, including buried waste, contaminated soils, contaminated groundwater, containerized hazardous and radioactive wastes, and underground detonation sites. Contaminants include volatile or non-volatile organics like chlorinated solvents, radionuclides, heavy metals, nitrates, or highly explosive materials. The preferred treatment of such contaminated media is in situ to prevent contaminant from spreading into the environment.

The In Situ Remediation Integrated Program (ISRIP) was established in recognition that in situ remediation could significantly reduce the cost of cleanup and minimize worker exposure by eliminating or minimizing waste excavation, transportation, and disposal. Additionally, in situ remediation allows for the cleanup of inaccessible sites such as deep subsurfaces and areas beneath structures.

The ISRIP is conducting R&D in three major program areas. The containment subprogram seeks to develop in situ technologies for pollution containment by barriers and absorbers/neutralizers. This will provide short-term containment while the source plume is being remediated, or long-term containment for sites that present no immediate health/environmental risk or that require development of new remediation methods. The treatment subprogram develops and evaluates in situ technologies for destruction, enhanced removal, extraction, and immobilization of groundwater/soil contaminants. The subsurface manipulation subprogram evaluates physical control systems for avoiding dispersal of contaminants, or assisting dispersal of treatment agents during in situ remediation. Technologies may include electrokinetic migration of contaminants, hydraulic isolation, auger or jet mixing, hydro- or cryofracturing, pneumatic fracturing, and vacuum-vaporizer well systems.

VOCs in Non-Arid Soils

More than 15 percent of the U.S. drinking water supplies are contaminated with chlorinated hydrocarbons. The major organic contaminants of waste sites at DOE facilities are volatile chlorinated solvents that were used to clean and to degrease nuclear weapons components during the production process. Current baseline technologies for remediating soils and groundwater contaminated with VOCs are inefficient and expensive. Pump and treat groundwater remediation, while effective for hydraulic plume control and removal of significant quantities of contaminant, is neither cost-effective nor efficient for cleaning up groundwater to drinking water standards. Excavation techniques are very expensive and involve workers' exposure to toxic vapors.

The objectives of the VOC-Non-Arid ID are to test the integrated demonstration concept, to demonstrate and to evaluate innovative technologies/systems for remediating VOC contamination in soils and groundwater, and to transfer technologies and systems to internal and external customers for use in full-scale remediation programs. In situ remediation is emphasized because it has tremendous advantages over above-ground treatment.

In situ remediation technology has the potential to be more effective in less time at a reduced cost and also has the benefit of minimizing worker exposure. Three in situ remediation systems were demonstrated: air stripping and sparging, bioremediation, and radio frequency heating.

VOCs in Arid Soils

VOC contamination of soils and groundwater is one of the most common environmental problems in the United States. When VOCs are released into the soil, they rapidly migrate forming large plumes that contaminate groundwater. No permanent solution currently exists to solve this problem.

VOC-Arid ID focuses on technologies to characterize/remediate VOCs as well as associated contaminants in soil and groundwater at arid sites. There are three phases of implementation: soil vapor extraction of VOCs from the vadose (unsaturated) zone, demonstration of enhanced techniques to control the transport of VOC vadose zone contaminants, and development and demonstration of technologies for containment, mobilizing for recovery, and/or immobilizing metals/radionuclides in both soil and groundwater. The primary focus will be on in situ treatment or enhanced recovery techniques for americium and plutonium in soils, and uranium and chromium in groundwater. These contaminants represent common VOC co-contaminants at DOE sites and are significant problems at the host site.

Focus Areas [See Focus Area Structure]

To focus Department-wide activities on DOE's most pressing environmental restoration and waste management problems, the Assistant Secretary for EM established a Working Group in August 1993 to develop and implement a new approach to environmental research and technology development. This has resulted in the identification of four major remediation and waste management problem areas within the DOE Complex. These problems have been targeted for action on the basis of risk, prevalence, or need for technology development to comply with environmental requirements and regulations.

Focus areas include:

- Subsurface Contaminants
- High-Level Waste Tank Remediation
- Mixed Waste Characterization, Treatment, & Disposition
- Decontamination and Decommissioning

UMTRA Sites (Uranium Mill Tailings Remedial Action)

Monument Valley, Arizona
Tuba City, Arizona
Durango, Colorado
Grand Junction, Colorado
Gunnison, Colorado
Maybell, Colorado
Naturita, Colorado
Rifle, Colorado
Slick Rock, Colorado
Lowman, Idaho
Ambrosia Lake, New Mexico

Shiprock, New Mexico
Belfield, North Dakota
Bowman, North Dakota
Lakeview, Oregon
Canonsburg, Pennsylvania
Edgemont, South Dakota
Falls City, Texas
Green River, Utah
Mexican Hat, Utah
Salt Lake City, Utah
Riverton, Wyoming
Spook, Wyoming

Decontamination and Decommissioning Focus Area

Problems in the Decontamination and Decommisioning Focus Area include:

- Decontamination and Decommissioning Facilities

Mixed Waste Characterization, Treatment, and Disposition Focus Area

Problems in the Mixed Waste Characterization, Treatment, and Disposion Focus Area include:

- Mixed Waste Treatment
- Supercritical Water Oxidation
- Rocky Flats Compliance Program

FUSRAP Sites (Formerly Utilized Sites Remedial Action Program)

* Indicates that remedial actions have been accomplished

Installation	Location
University of California*	Berkely, California
National Guard Armory*	Chicago, Illinois
University of Chicago*	Chicago, Illinois
W.R. Grace & Company	Curtis Bay, Maryland
General Motors	Adrian, Michigan
Latty Avenue Properties	Hazelwood, Missouri
St. Louis Airport Site	St. Louis, Missouri
St. Louis Airport Storage Site Vicinity Properties	St. Louis, Missouri
St. Louis Downtown Site	St. Louis, Missouri
Acid/Pueblo Canyon*	Los Alamos, New Mexico
Bayo Canyon*	Los Alamos, New Mexico
Chupadera Mesa*	White Sands, New Mexico
Niagara Falls Storage Site*	Lewiston, New York
Ashland 1/Ashland 2	Tonawanda, New York
Linde Air Products	Tonawanda, New York
Seaway	Tonawanda, New York
Albany Research Center*	Albany, Oregon
Aliquippa Forge	Aliquippa, Pennsylvania
Middlesex Municipal Landfill*	Middlesex, New Jersey
Middlesex Sampling Plant	Middlesex, NewJersey
Baker & Williams Warehouses	New York, New York
Elza Gate*	Oak Ridge, Tennessee
New Brunswick Laboratory	New Brunswick, New Jersey
Du Pont & Company	Deepwater, New Jersey
Maywood	Maywood, New Jersey
Kellex/Pierpont*	Jersey City, New Jersey
Wayne/Pequannock	Wayne, New Jersey
Colonie	Colonie, New York
Seymour Specialty Wire	Seymour, Connecticut
Shpack Landfill	Norton, Massachusetts
Ventron	Beverly, Massachusetts

Stages and Gates [See Gate Information Requirements Diagram]

Basic Research Stage
Gate 1

- There are no criteria associated with passing Gate 1 (Going from Basic research to Applied Research)

Applied Research Stage
Gate 2

- Address Priority DOE Need?
- Indicate Complementary or Redundant Efforts?
- Intellectual Property Protection?

Exploratory Development Stage
Gate 3

- Address Focus Area Need?
- Product and/or System Integration Requirements?
- Identify/Address Feasibility Issues
- User and Regulatory Needs & Wants Identified?
- Identify DOE Users, Windows of Opportunity, Market Size, Uncertainties?
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Gate 6

- EM-30/40 Procures Technology?
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- NEPA Documentation & Permits for DOE Deployment?

Implementation Stage

Pollution Prevention

Pollution Prevention activities involve integrating new technologies and technology systems into processes to reduce the toxicity or amount of hazardous and/or radioactive materials at the source, thus decreasing the toxicity, or quantity of waste for recycling, treatment, or disposal.

EM is responsible for the waste produced in dismantling approximately 2,000 weapons per year, including treatment of approximately 303 cubic yards of weapons components yearly. DOE estimates that a ten-fold reduction in waste volume is possible through advanced processes and technologies resulting in an annual storage cost savings of about 95%. In addition, EM has determined that innovative resource recovery processes could provide approximately $10 million per year through the resale of precious metals such as silver, gold, and platinum.

EM, in cooperation with other DOE programs, federal agencies, and industry, is developing technological solutions to address common waste stream problems--such as chlorinated solvents, toxic metals from finishing operations, VOCs from cleaning operations, and waste acid recycle.

Waste Retrieval and Processing

Waste retrieval and Processing activities focus on technologies for retrieving/processing hazardous, radioactive, and mixed waste and for treating buried and stored waste from current DOE D&D Complex operations, and facilities no longer in operation.

This represents one of the largest DOE problems. Within the DOE complex, large quantities of high level, low level , and transuranic waste have been burried or stored which need retrieval as well as treatment. Before 1970, most low-level and transuranic wastes were burried in common shallow land burial grounds. Most high level waste was stored in underground storage tanks. Over the years, many of the older disposal containers have developed leaks resulting in contamination of the adjacent soil.

Effective May 8, 1992, all DOE mixed waste streams fell under EPA's land disposal restrictions. They no longer can be disposed of without prior treatment to destroy, separate, or immobilize the hazardous component. Most hazardous components of mixed wastes have not been characterized; but from past knowledge, they represent the entire gamut of organic and inorganic hazardous waste. To solve many of the problems at hand, emerging technology must be developed.

Groundwater and Soils Cleanup

Groundwater and Soils Cleanup activities develop technology systems to clean up and contain hazardous/radioactive contaminants in groundwatef and soils. Sources of soil and groundwater contamination include: previous disposal of waste in ponds, seepage pits, trenches, and shallow land burial sites; spills and leakage from waste transport, storage facilities, and underground tanks; and discharges to the air and surface waters.

Volatile Organic Compound (VOC) and heavy metal contamination represent some of the highest priority problems for which innovative technologies are sought, due to their high concentrations, high mobility, high toxicity, and long-term persistence in the environment. Technologies are also under development for treatment of non-volatile organics, dense non-aqueous phase liquids (DNAPLES), radionuclides, nitrates, and explosive materials. In-situ methods are preferred, if feasible, because they minimize harm to the environment, lessen public exposure, and avoid the risks and costs of handling.

Problem Areas

Groundwater and Soils Cleanup
Waste Retrieval and Processing
Pollution Prevention

Integrated Programs/Integrated Demonstrations

In 1989, the Office of Science and Technology introduced the concepts of Integrated Programs and Integrated Demonstrations as an approach to more effectively manage RDDT&E activities. Integrated Programs (IPs) conduct applied research activities in key application areas required in each stage of the remediation process (e.g. characterization, treatment, and disposal). Technologies developed within IPs can be applied to environmental problems at many DOE sites, in any problem area.

An Integrated Demonstration (ID) is a cost-effective mechanism that assembles a group of related and synergistic technologies to evaluate their performance in a system context to solve waste management and environmental problems from cradle to grave. The Office of Science and Technology's programs are executed in three phases: Innovative Technology; R&DR_D@Acronym.hlp; and DT&EDT_E@Acronym.hlp. The Innovative Technology phase supports the transition of technology from concept to proof-of-concept. The goal is to prepare a technology for the applied R&D phase within an Integrated Program or Integrated Demonstration to address a distinct environmental problem in one problem area.

Waste Categories

High-Level Waste (HLW)

The highly radioactive waste material that results from the reprocessing of spent nuclear fuel, including liquid waste produced directly in reprocessing and any solid waste derived from the liquid, that contains a combination of transuranic waste and fission products in concentrations requiring permanent isolation. This waste is so highly radioactive that it generates heat on its own. Some of its elements will remain radioactive for thousands of years. It must be handled from behind heavy protective shielding.

Transuranic (TRU) Waste

Waste that is contaminated with alpha-emitting radionuclides with an atomic number greater than 92 (heavier than uranium), half-lives greater than 30 years, and concentrations greater than 100 nanocuries per gram of waste. It is produced during reactor fuel assembly, nuclear weapons production, and fuel reprocessing. This waste is not as intensely radioactive as HLW, but the slow decay of its radioactive components requires long term isolation.

Low-Level Waste (LLW)

Radioactive waste not classified as high-level waste, transuranic waste, spent nuclear fuel, or by-product material. It typically has small amounts of radioactivity in large amounts of material. LLW is generated in every process involving radioactive materials, including cleanup projects. Most radioactivity in LLW will decay to harmless levels in a few years.

Hazardous Waste

As defined in RCRA, a solid waste or combination of solid wastes that, because of its quantity, concentration, or physical, chemical, or infectious characteristics, may cause or significantly contribute to an increase in mortality or an increase in serious, irreversible, or incapacitating reversible illness or pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed of, or otherwise managed.

Mixed Waste

Waste containing both radioactive and hazardous components, as defined by the Atomic Energy Act and RCRA.

Uranium Mill Tailings

A category of nuclear waste consisting of the by-products of uranium mining and milling, consisting of naturally radioactive rock and soil. It exhibits low levels of radiation from the small amount of radium that decays, and emits radon, a radioactive gas.

Sanitary Waste

Wastes such as garbage, that are generated by normal housekeeping activities and that are not hazardous or radioactive.

Efficient Separations Processing

Separations technologies can be used to remove dilute, harmful constituents, such as radionuclides, from the host material, generating a small volume of concentrated waste for subsequent immobilization/disposal. The remaining larger volume, now less toxic and easier to handle, can be treated and disposed of less expensively. The Effluent Separations and Processing Integrated Program (ESPIP) was created to identify, develop, and perfect separations processes to treat wastes as well as to address environmental problems throughout the DOE Complex.

ESPIP funds R&D ranging from computer-aided design of new selective separation agents to extraction process design. ESPIP also develops separation processes to extract high-value materials and non-radioactive hazardous components from DOE waste. Many of these separation methods also have potential for solving a variety of waste and environmental problems in commercial markets outside the DOE Complex.

Characterization, Monitoring, and Sensor Technology

The DOE emphasis on nuclear weapons production over the past several decades has resulted in soil and groundwater contamination by hazardous, radiochemical, and mixed wastes at sites nationwide. DOE is required to characterize approximately 3,700 contaminated sites before remediation, treatment, and decommissioning/deactivation activities commence.

In order to design an effective method for site cleanup, an engineer must know the type, location, and concentration of contaminants, and the hydrogeology and subsurface soil properties of a site, During remediation, treatment, and site closure, monitoring technologies are needed to ensure worker safety and effective cleanup. Characterization, monitoring, and sensor (CMS) technologies have numerous and diverse applications within site cleanup and waste management operations.

The Characterization, Monitoring, and Sensor Technology Integrated Program (CMSTIP) was established to bring new, effective CMS technologies in support of remediation and waste management activities. CMSTIP matches and adapts available technologies to help solve EM's immediate environmental problems. In the long term, the program also stimulates, coordinates, and sponsors CMS research and development to promote emerging CMS technologies.

CMSTIP activities are grouped into the following areas:

- Characterization and monitoring in high-level waste tanks and hot cells

- Characterization and monitoring of mixed waste, waste stream processes, and effluents
- Characterization technologies for D&Dd_d@acronym.hlp activities
- Field-deployable, surface-based, geophysical and chemical site characterization technologies
- Small prototype sensors for in situ characterization and monitoring of subsurface properties

Supercritical Water Oxidation

DOE has large inventories of mixed waste stored at its sites and facilities which fall under EPA's Land Disposal Restrictions. This waste cannot be stored indefinitely, nor can it be disposed of without prior treatment to destroy, separate, or immobilize the hazardous components. DOE sites and facilities will continue to generate significant quantities of mixed and hazardous wastehazardous_waste@glossary.hlp as part of ongoing operations including cleanup.

Supercritical Water Oxidation (SCWO) technology holds promise for treating a portion of DOE's mixed waste. The process involves bringing together organic waste, water, and an oxidant (such as air, oxygen, etc.) to temperatures and pressures above the critical point of water (374° C, 22.1 MPa). Under these conditions, organics in the waste are destroyed to levels of over 99.99%. The resulting effluents, which consist primarily of water and carbon dioxide, are relatively benign.

The SCWO Program will construct and demonstrate hazardous and mixed waste pilot-scale SCWO units. The program will be conducted in two phases; Phase 1 is the Hazardous Waste Pilot Plant (HWPP) Demonstration, and Phase 2 is the Mixed Waste Pilot Plant (MWPP) Demonstration.

The SCWO Program works closely with private industry to ensure the commercialization of this technology with stakeholders and regulators to ensure the technology's acceptability. In addition, the program coordinates very closely with other federal agencies, such as DoD, U.S, Navy, U.S. Air Force, U.S. Army, EPA, and the Advanced Research Projects Agency to leverage federal resources in the development of the technology.

Underground Storage Tanks

Across the DOE Complex, 332 USTs have been used to capture, process, and store radioactive and hazardous wastes generated from the production and processing of nuclear weapons material since the 1940s. Problems associated with UST waste remediation are complex. First, many tank safety issues need to be resolved, including hydrogen gas and heat generation, accumulation of hazardous substances such as organics and potentially explosive ferrocyanides, and tank leakage. Second, the chemical composition of the tank waste varies from tank to tank. Most of the waste is alkaline and is rich in sodium and nitrite, but various hydrated metal oxides, organics, inorganics, and radionuclides including fission products and transuranics are present. Also, a high level of radioactivity exists in some of the UST waste. The heterogeneity of the tank waste -- both physically and chemically -- across the DOE Complex dictates that different retrieval and processing technologies be developed.

The UST-ID has focused its technology development on areas where currently available technology cannot meet the technical and safety requirements for tank waste remediation. The fundamental problem areas shared by most of the participating sites have been designated as UST-ID subprogram elements: waste characterization, waste retrieval and conveyance, waste separation and pretreatment, LLW treatment and disposal, and site closure. Examples of technologies being developed include in situ characterization, using laser spectroscopies, and robotics systems for tank waste retrieval.

High-Level Waste Tank Remediation Focus Area

Problems in the High-Level Waste Tank Remediation Focus Area include:

- Underground Storage Tanks
- Buried Waste

Subsurface Contaminants Focus Area

Problems in the Subsurface Contaminants Focus Area include:

- VOCs in Arid Soils
- VOCs in Non-Arid Soils
- In Situ Remediation
- Resource Recovery
- Heavy Metals Contaminated Soils
- Uranium in Soils
- MAWS

- Mixed Waste Landfill

Technology Investment Decision Model*

A Technology Investment Decision Model serves as a framework for technology management. The model incorporates decision points (gates) between each stage of the research and development of a new technology. At each gate, a go/no go decision must be made to determine whether or not the technology will continue to be developed and receive funding. Projects that do not continue through the development stages may be terminated, held for future consideration, or revised to more completely address relevant issues. The goal of the model is to choose the most beneficial technology development projects for funding. Seven stages and six gates have been identified in the technology development process.

*Source: Joseph Paladino and Paul Longworth, *A Decision Model for Technology Development in the Department of Energy's Environmental Clean-up program,* U.S. Department of Energy Office of Environmental Management, Office of Technology Development Report.

Office of Environmental Restoration

The Office of Environmental Restoration (EM-40) has program responsibilities for assessment and clean-up of inactive hazardous and radioactive facilities and waste sites at all DOE installations and some non-DOE sites, including remedial actions and facility decontamination and decommissioning (D&D) activities. EM-40 oversees program activities to reduce or eliminate risks to human health and the environment.

Office of Facility Transition and Management

The Office of Facility Transition and Management (EM-60) has the responsibility to ensure that shut-down facilities are brought to a deactivated state, are properly maintained, and are eventually decontaminated and/or decommissioned or released for other uses.

Office of Technology Transfer and Program Integration

The Office of Technology Transfer and Program Integration (EM-52) provides management, financial, and internal program support to the line organizations that comprise EM-50. It also provides efforts to encourage and to facilitate the infusion and diffusion of innovative environmental technologies for international and domestic applications through collaborative partnerships with U.S. and foreign industry and organizations, the national laboratories, other federal agencies, and universities.

Office of Research and Development

The Office of Research and Development (EM-53) is responsible for establishing applied research and development (R&D) programs at DOE sites nationwide. Programs are designed to identify operational needs in environmental restoration, waste operations, and corrective activities, and to provide solutions to key technical issues that, if not solved in a timely manner, would adversely affect DOE's ability to meet its cleanup goal.

Office of Demonstration, Testing, and Evaluation

The Office of Demonstration, Testing, and Evaluation (EM-54) is responsible for identifying environmental restoration and waste management technologies in the R&D stage that are ready for transition to the demonstration arena. Those technologies are complete systems to demonstrate a solution to a specific problem area. Programs are conducted to advance selected technologies so they can be utilized by DOE to meet its cleanup goal in a cost-effective manner.

Site Technology Coordinating Groups

Mission
Objectives
Scope
Organizational Structure
Interfaces

STCG Mission

The mission of the STCGs is to ensure that major obstacles and needs of their site are captured and communicated to the Focus Area Groups, and that newly developed technological solutions are implemented by their site operations in order to improve the performance of environmental programs in site remediation and waste management.

STCG Objectives

- Define and prioritize the Corporate needs of the site.
- Translate and communicate a common set of site problems and planned activities.
- Assure that Focus Area programs meet site needs.
- Implement new technology in site-wide environmental programs to improve site performance.
- Define the responsibility for demonstrating all new technology at the site (TPO).

STCG Scope

- Identification of planned EM programs/projects over a five-year period. This is to be updated annually, or more often if major shifts are planned or anticipated.
- Identification of major challenges, problems, and opportunities for improvement through the introduction of new technology.
- Review and concurrence that the Focus Areas have accurately understood the Site's needs and priorities, and that the technology development activities of the Focus Areas are responsive to those needs and priorities.
- Coordination of all technology development activities conducted at their site.
- Development and implementation of processes that ensure early deployment of operational schedules that reflect decision points for potential deployment of new technologies.

STCG Organizational Structure

The STCGs will structured at the discretion of the DOE Site Manager, to whom the STCG Chairperson will report. It is recommended that the Chair of the STCG be the Deputy

Manager at each site. At minimum, the STCG should include members from: R&D, DP, EM-30, EM-40, EM-60, M&Os and Laboratories. Additional members, at the discretion of the Site Manager, could include federal, state, and local regulators, regional stakeholders, and representatives of Indian tribes.

The functional requirements of the organization are to provide a single point of contact for Focus Area Groups. They must also provide a corporate position for the site on priorities, activities, etc. Finally, they must be committed to the process of the new approach to quickly infuse new technology into the site operations.

STCG Interfaces

The chairs of the STCGs report to their respective site manager. He/she is responisble for the interfaces to the regional stakeholders (SSAB), Indian tribes, and the focus area management and implementation teams.

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7.0 CONCLUSIONS

The online help system described above was utilized by the database design team at Virginia Tech during their design of the EM-50 database. It provided domain knowledge concerning the organization, structure, and procedures followed in the Office of Environmental Management, and helped the designers to better understand how to structure the design of their database.

8.0 WORK PLANNED FOR NEXT PERIOD

Work for the next period will consist of designing an on-line help system to provide context-sensitive help for users of the EM-50 database. This will include descriptions of all screens that are used for data input, data tables used in the database, and explanations of the Analytic Hierarchy Process (AHP). AHP is being used to aid decision-makers in evaluating and prioritizing projects to be funded in the technology development effort.

A GIS-BASED INFRASTRUCTURE FOR SITE CHARACTERIZATION AND REMEDIATION

METC Task No. 2.1

Quarterly Technical Progress Report
Reporting Period:
July 1,1996 - September 30, 1996

Work Performed Under Contract
No.: DE-FC21-92MC29467

For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By:
Marshall University
Center for Environmental, Geotechnical, and Applied Sciences
Huntington, West Virginia 25755
(304) 696-5453 (304) 696-5454 Fax

ABSTRACT

The overall goal of this effort is to determine an effective geographic information system (GIS)-based infrastructure for describing, characterizing, and remediating contaminated sites. The work is being carried out with the remediation needs of the U.S. Army Corps of Engineers Huntington, WV district office as a basis, providing the benefit of real needs of on-going remediation projects. Working closely with METC and the Corps of Engineers, during 1996 we will (a) achieve actual on-site application of the system to a Corps project in Mason County, WV, (b) assess and augment the infrastructure for use in decontamination and decommissioning (D&D) efforts, and (c) develop a prototype interactive tutorial system to effectively transfer infrastructure concepts and skills to prospective users and managers.

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EXECUTIVE SUMMARY

This document constitutes the required Quarterly Report for the third quarter of 1996, for Task No. 2.1 under the DOE/METC/WVU Cooperative Agreement, contract no. DE-FC21-92MC29467. It documents technical accomplishments and provides cost status as of September 30, 1996.

Of the three primary tasks (2, 3 and 4), we are progressing on schedule for tasks 3 and 4. For task 2, we will extend the duration to end of December 1996 to map and possibly model using GIS, the effect of pump and treat of an underground aquifer on the plant life around the wetlands of a section of the contaminated area at the WV Ordnance site, now called the McClintic Wildlife Station. This extension is due to the fact that the pump and treat process did not begin in August 1996 as anticipated, but will begin in early November 1996.

For task 3, we will satisfy the intent of the task, but with a more general focus. This is because of the difficulty in arranging for a specific contaminated site relative to a specific technology. We are using the GIS technology infrastructure for other projects with state and federal agencies, and will assess and augment the infrastructure based on these projects.

For Task 4, we continue building the prototype interactive tutorial system using Director 5.0. We have built a framework for the chapters and lessons, and have built three lessons based on the framework. We have included a few screen shots from a lesson with this report.

In summary, we are in an excellent position to successfully complete all tasks during the calendar year.

1.0 INTRODUCTION

Under the 1995 METC contract, the Marshall University (MU) Environmental Center determined and set up an effective geographic information system (GIS) based infrastructure for describing, characterizing and remediating contaminated sites. The 1995 effort resulted in (1) a detailed description of hardware and software comprising a system proven effective through experimentation and evaluation, (2) operating procedures for effective system use, (3) educational/training materials for technology transfer, and (4) recommendations for follow-on system implementation. In carrying out this effort, we worked with the U.S. Army Corps of Engineers, Huntington, WV District, using their needs for characterization and remediation of contaminated sites as a realistic basis for determining system requirements. In this multi-year effort, we are strongly emphasizing the need to integrate this resulting technology into activities of interest and benefit to DOE. The working relationship with the Corps of Engineers has been closely coordinated with METC.

2.0 PURPOSE

This follow-on work for calendar year 1996 continues to address the DOE focus area Cross Cutting Innovative Technologies, Characterization sub-area. The results of this year's effort will be (1) actual on-site application of the GIS-Based Infrastructure in an ongoing project of the Corps of Engineers in Mason County, West Virginia, (2) assessment and augmentation of the Infrastructure for use in decontamination and decommissioning (D&D) efforts, and (3) development and evaluation of a prototype interactive tutorial system to effectively transfer the Infrastructure technology and underlying GIS concepts and skills to prospective users and managers.

3.0 BACKGROUND

Geographic Information Systems (GISs) are increasing rapidly in numbers and areas of application. No doubt the expansion is influenced strongly by the growing national interest in environmental protection and restoration. Numerous GIS systems are available--including commercial (e.g., ARC/INFO, Intergraph MGE, MapInfo), government-sponsored (GRASS), and university research based (IDRISI, ICASE/GRACE). The desired applicability of GISs is extremely broad (e.g., air quality monitoring, groundwater contamination, areas in a city accessible by public transport). Thus the GIS systems must be very generic in their capabilities. This genericness is attractive in providing versatility of application breadth, but of necessity limits the depth of features for a given application area.

The nation faces a massive problem relative to contaminated sites. The U.S. Department of Energy, the U.S. Army Corps of Engineers, and many other government agencies are faced with requirements to remediate contaminated sites under their purview. The Huntington District of the U.S. Army Corps of Engineers (USACE) has undertaken to assemble computer-based tools to help them in describing, characterizing, and remediating contaminated sites. They have obtained the Integrated Computer Assisted Site Evaluator / Graphical Remedial Assessment and Cost Evaluation (ICASE/GRACE) system, developed at the University of Cincinnati, for evaluation. They have also obtained and installed Intergraph's MGE system, and anticipate obtaining Intergraph's ERMA (for Environmental Resource Management Application) system, intended to support site assessment, modeling and remediation. Under an earlier METC task (1994), Marshall University evaluated ICASE/GRACE for use by the Huntington USACE District.

4.0 METHODOLOGY

Following are descriptions of the 1996 tasks to be conducted, and associated schedules.

Task 1: *Information Required for the National Environmental Policy Act.*

Schedule: Within 60 days of contract award.

Task 2: *Application of the GIS-Based Infrastructure at the West Virginia Ordnance Site.*

Personnel from Marshall University will work as a team with Corps of Engineers personnel to transfer the GIS-Based Infrastructure technology to actual use on a project managed by the HTRW Section of the Huntington District office. The project will include obtaining/ developing digitized maps of the WV Ordnance area, and associating on-line with the maps data characterizing contaminated sites (actual data when available, or modeled) in various forms from many sources. We will work with Corps personnel in all aspects of the project, including translating data for compatibility, entering data into the GIS database, and associating it with corresponding map elements.

Since the area of this site is very large (about 3000 acres), it will present many challenging issues such as organization of database tables, interaction of multiple contaminants from various locations on the site, handling of incomplete and/or inconsistent data, and extraction of the effect of individual contaminants.

Once the database is established, it will be possible to model various contamination scenarios. Based on such scenarios, we will model and assess remediation strategies for some of the contaminants, in collaboration with the Corps of Engineers. The Environmental Resource Management Application (ERMA) package, which is an Intergraph product, will be used for this purpose.

Schedule: January 1 through September 30, 1996 (now extended through December 31, 1996).

Task 3: *Assessment and Augmentation of the Infrastructure for D&D Use.*

In concert with METC personnel, we will make use of available characterization of contaminated sites within WV Ordnance to assess strategies for D&D. The GIS-Based Infrastructure, with its open software architecture, accommodates incorporation of additional models. The WV Ordnance site data resulting from Task 2 will provide a diversity of contaminants and geological characteristics for evaluating D&D remedial technologies and operational strategies. We will coordinate the selection of specific sites and technologies with METC, prior to undertaking. We will support METC in evaluating remediation approaches relative to specific D&D contaminated sites, by means of models operating on site data. The Intergraph ERMA remediation software will be considered for applicability in this effort, as well as models available to METC and the Corps of Engineers.

Schedule: August 1 through December 31, 1996.

Task 4: *Prototype Development and Evaluation of an Interactive Tutorial System.*

By use of commercially available courseware authoring tools, we will develop a prototype interactive tutorial system to transfer the GIS-based technology concepts and skills to users and managers. The tutorial sessions will provide step by step lessons incorporating real data. The approach will be based on sound pedagogical principles, and will be practically oriented to maximize use on the job. The tutorials will mimic the interface (menus, buttons and windows) of the actual system. We will incorporate selectable modes by degree of user sophistication, so that more or less guidance is provided based on need. We will also develop tutorial materials of varying abstraction/detail based on the needs of various personnel--ranging from managers to the every-day user of GIS technology. In these lessons we will cover representative facets of the Intergraph MGE system, and additional models and features incorporated into our GIS-Based Infrastructure. A significant benefit of the resulting approach will be the availability of the tutorials for use and reuse after an employee begins to use the skills on the job.

We will conduct a limited field test of the prototype using a representative range of personnel in order to ensure its effectiveness and make any needed adjustments. The result of this effort will be a working prototype incorporating proven concepts which can later serve as the basis for developing an operational tutorial system.

Schedule: January 1 through December 31, 1996.

5.0 WORK PERFORMED THIS PERIOD

5.1 Activities

During this period, we continued our efforts on Task 2 (Application of the GIS-Based Infrastructure at the West Virginia Ordnance Site) and Task 4 (Prototype Development and Evaluation of an Interactive Tutorial System). We also began work on Task 3 (Assessment and Augmentation of the Infrastructure for D&D Use). The following paragraphs give more details on each task.

Task 2: *Application of the GIS-Based Infrastructure at the West Virginia Ordnance (WVO) Site.*

As mentioned in the second quarterly report, we have been unable to acquire contamination data from the Corps of Engineers for the West Virginia Ordnance site, now renamed to McClintic Wildlife Station (MWS). However, the Huntington District Corps personnel are involved in a project to pump and treat an aquifer in an area under the MWS site which has been contaminated by surface contaminants. The pump and treat operation, scheduled to begin in August of 1996, will now begin in early November, 1996. It is envisioned that the pump and treat process may affect the water level, and hence the plant life in and around the wetlands on the site. Thus, the Corps is required to establish a monitoring plan to document the possible effects of the pump and treat process. We have had several discussions with the Corps personnel for using GIS technology for the monitoring process. Marshall University Environmental Center and the College of Science are anticipating a contract with the Huntington District Corps, for "before" and "after" monitoring of the water level and plant life of the wetlands in the area. Under the auspices of this METC contract, we will provide mapping, GIS, and groundwater modeling (using MGE ERMA) support. The monitoring process plan has been presented to the WV Division of Environmental Protection (DEP), WV Division of Natural Resources (DNR) and the community.

Due to the delay by the Corps in beginning the pump and treat process, we will extend the duration of this task to the end of December 1996 to provide the mapping/GIS support for the early phases of the pump and treat process.

We purchased MGE Mapping Office, MGE Voxel Analyst, and Microstation 95 to assist us in gathering and analysis of the data from the pump and treat process.

Task 3: *Assessment and Augmentation of the Infrastructure for D&D Use.*

Due to the difficulty in arranging for a specific contaminated site for a specific technology, we have generalized the focus of this task to assess and augment the infrastructure at Marshall for other uses, including D&D use. In the past year, MU Environmental Center has been involved in several GIS related efforts which were executed using the hardware, software and personnel infrastructure built during this and the previous year METC contract. The experience gained through these contracts has enabled us to work closely with several local, state, and federal agencies and provide them effective mapping/GIS support. In the paragraphs below, we describe a few additional mapping/GIS contracts we expect in the near future.

The Huntington Corps is involved with a project at Dolly Sods, WV which is an unexploded ordnance site, now under the jurisdiction of the U.S. Forestry Service. Corps personnel have approached us with regards to use of GIS technology, and conversion of data of the site maintained in MGE format to ARC/Info format. We are negotiating to provide the GIS and quality assurance support, and are expecting a contract within a week.

Marshall University (MU) Environmental Center personnel, along with personnel from the MU School of Medicine recently supported the International Union of Operating Engineers (IUOE) National Hazmat Program, Beaver, WV in the assessment of the Advanced Worker Protection System developed by Oceaneering Space Systems (OSS), Houston Texas. Development of the protective suit, to be used primarily for DOE D&D purposes, and the assessment were funded by DOE METC. Negotiations are currently underway for MU to provide long-term support to IUOE testing activities, including human subject review board (HSRB), development of interactive training materials, and statistical analysis of test results.

Thus our previous successful efforts with the GIS infrastructure, coupled with our current and forthcoming efforts, provide the basis for successful completion of the intent of the task.

Task 4: *Prototype Development and Evaluation of an Interactive Tutorial System.*

We have made excellent progress on the prototype interactive tutorial system using the Director multimedia software. Based on the framework built during the last quarter, we have developed three lessons. We have included a few screens from a lesson (Appendix B).

During the second quarter, we purchased a soundcard and additional memory for the machine used for creating the multimedia tutorial. This purchase was invoiced this quarter. Additionally, we purchased a 4 GB hard drive to store the tutorial during development, and we upgraded our software from Director 4.0 to Director 5.0.

5.2 Costs

The following is an unofficial summary of costs incurred during this quarter; Marshall University Research Corporation is the agent for formal contract and financial transactions.

Salaries	$ 19,210.90
Benefits	$ 3,945.97
Students	$ 4,455.80
Supplies	$ 322.28
Travel	$ 616.27
Equipment	
< $1000	$ 2,665.96
> $ 1000	$ 0.00
Total Direct Costs	$ 31,217.18
Indirect Cost (less Equipment)	$ 12,174.70
Total	$ 43,391.88

6.0 RESULTS AND DISCUSSION (CURRENT PERIOD)

Due to the delay in the pump and treat process at the McClintic Wildlife Station (formerly known as the West Virginia Ordnance Site), the duration of Task 2 will be extended to end of December 1996 to achieve the GIS support required in Task 2. We are satisfying the intent of Task 3 but generalizing the focus. We are making good progress on Task 4, the development of the tutorial system.

7.0 CONCLUSIONS (CURRENT PERIOD)

We will extend the duration of Task 2 until end of December 1996, by which time we will have data from the pump and treat process at the McClintic site. We are on schedule with the other tasks and are well positioned to achieve successful completion of all tasks during this year.

8.0 WORK PLANNED FOR NEXT PERIOD

8.1 Activities

We will continue our efforts on Task 2 and Task 4. For Task 3, we will assess and augment our infrastructure based on previous and forthcoming GIS related efforts. One specific project, whose duration is approximately two months, is the Dolly Sods project with the Huntington District Corps. We anticipate a contract with the Corps with regards to use of GIS technology, and conversion of data of the site maintained in MGE format to ARC/Info format. This work is also of interest to the U.S. Forestry Service.

8.2 Costs

The following table summarizes anticipated costs for the next quarter.

Salaries	$ 18,216.42
Benefits	$ 3,573.89
Students	$ 2,118.93
Supplies	$ 160.61
Travel	$ 2,050.44
Equipment	
< $1000	$ 0.00
> $1000	$ 0.00
Total Direct Costs	$ 26,120.29
39 % Indirect Cost (less Hardware)	$ 10,186.91
Total	$ 36,307.20

9.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES

We anticipated the pump and treat process to begin in August 1996, but it will not begin until November 1996. We have extended the duration of Task 2 until end of December 1996 to facilitate the use of GIS technology for effective monitoring and modeling purposes. With the adjustment in schedule, we expect to fully complete Task 2, and all other tasks, by the end of the year.

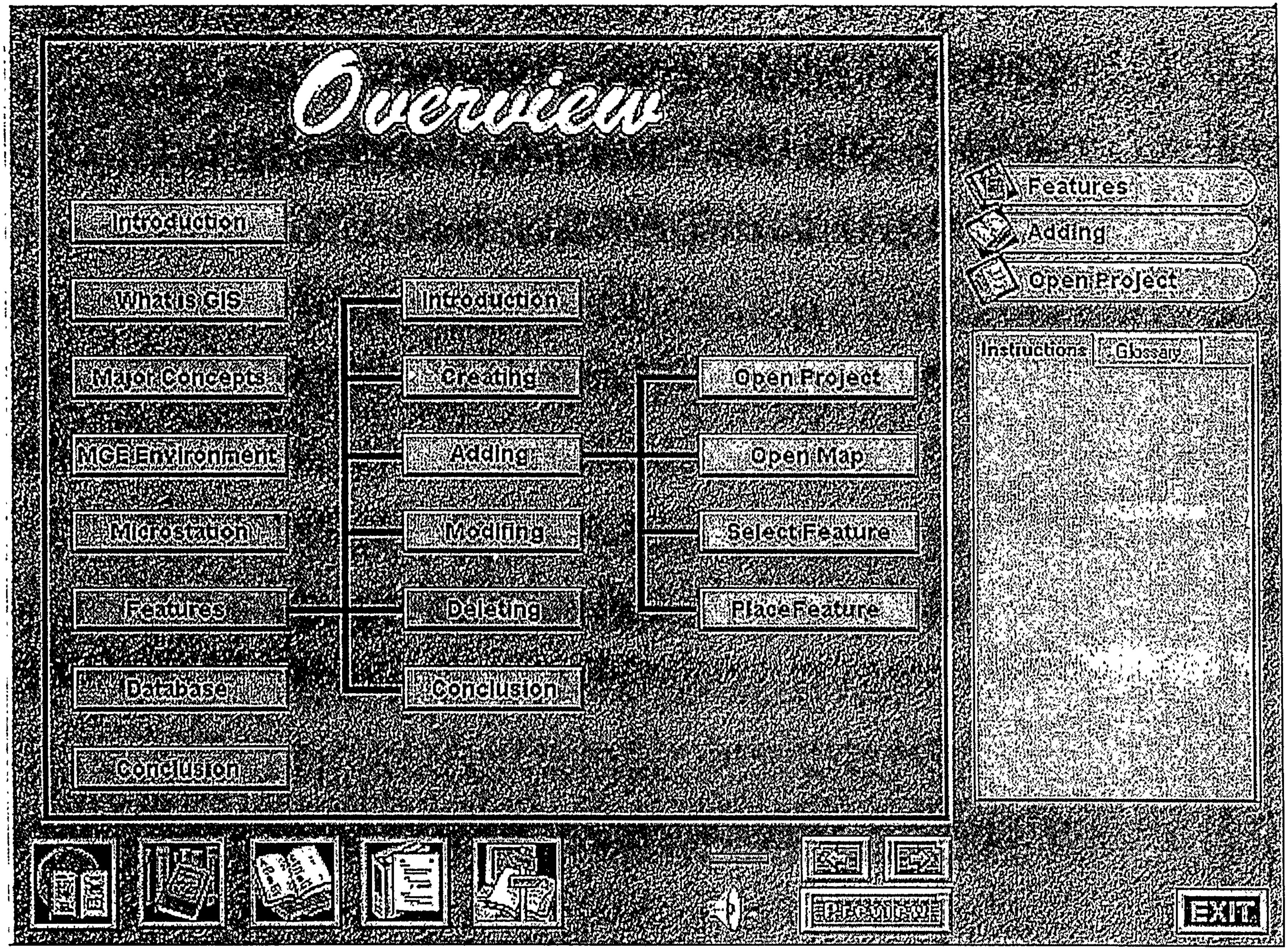
APPENDICES

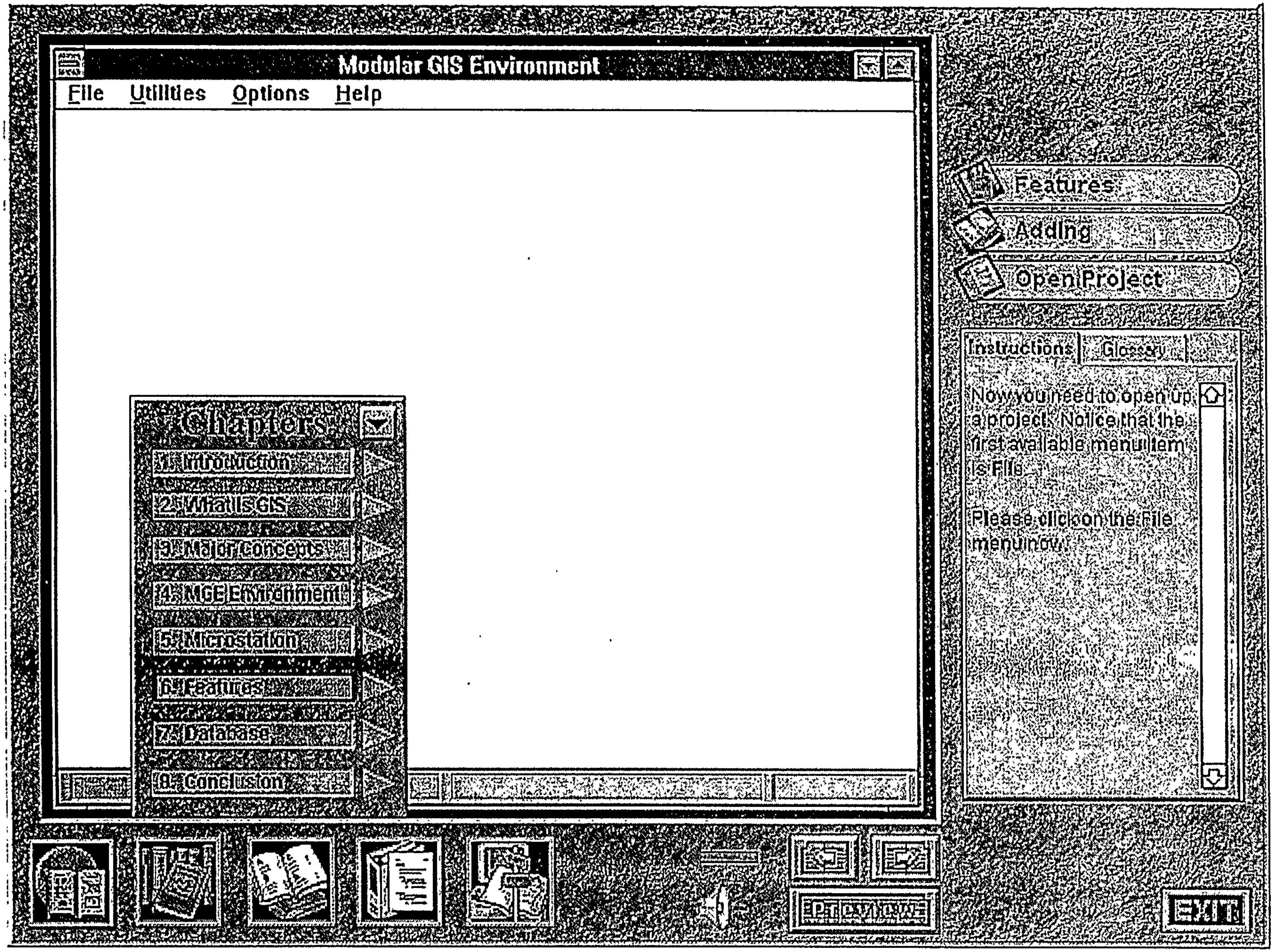
A. LIST OF ACRONYMS AND ABBREVIATIONS

ASCII	American Standard Code for Information Interchange
CD-ROM	compact disk read only memory
COTS	commercial off-the-shelf
D&D	decontamination and decommissioning
DOS	disk operating system
ERMA	Environmental Resource Management Application (an Intergraph product)
GB	gigabyte; approximately 1,000 MB
GIS	geographic information system
HP	Hewlett Packard
HTRW	hazardous, toxic & radioactive waste
ICASE/GRACE	Integrated Computer Assisted Site Evaluator/Graphical Remedial Assessment and Cost Evaluator
IP	Internet protocol
LCD	liquid crystal display
MB	megabyte; approximately one million bytes
MGE	Modular GIS Environment (an Intergraph product)
MHZ	megahertz
MS	Microsoft
MU	Marshall University
NT	New Technology (as part of Windows NT)
PC	Personal Computer
PCMCIA	Personal Computer Microchannel Interface Adaptor
RAM	random access memory
RIS	relational interface system
SCSI	small computer systems interface
SQL	system query language
SVGA	super video graphics adapter
TD 3	Technical Desktop 3
TD 4	Technical Desktop 4
UPS	un-interruptible power supply
USACE	United States Army Corps of Engineers

Appendix B

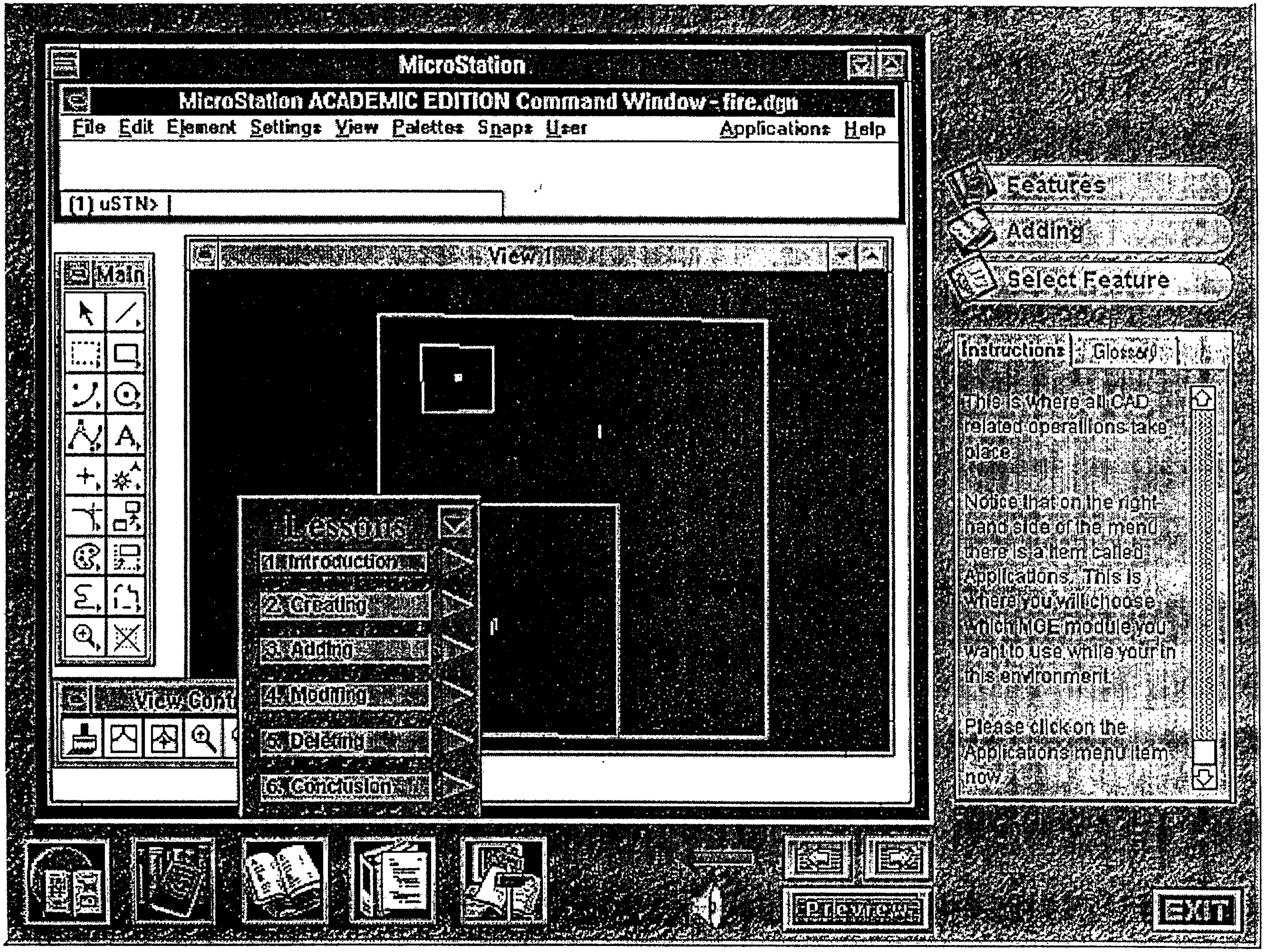
Screen shots from Prototype Tutorial


Overview
Introduction
What is GIS
Major Concepts
MGE Environment
Microstation
Features
Database
Conclusion
Introduction
Creating
Adding
Modifing
Deleting
Conclusion
Open Project
Open Map
Select Feature
Place Feature
Features
Adding
Open Project
Instructions
Glossary
EXIT



Now you need to open up a project. Notice that the first available menu item is File.

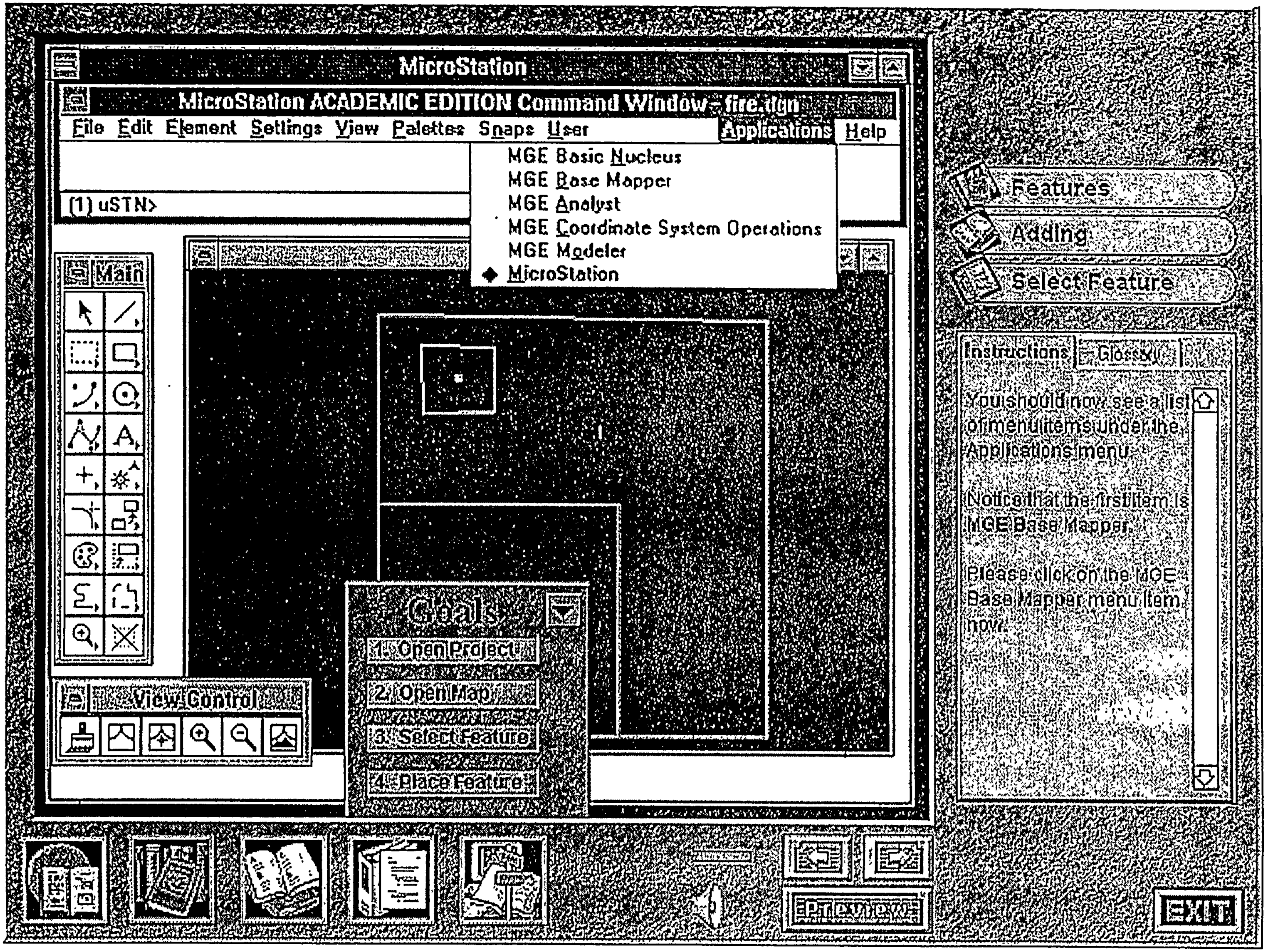
Please click on the File menu now.



This is where all CAD related operations take place.

Notice that on the right-hand side of the menu there is a item called Applications. This is where you will choose which MGE module you want to use while your in this environment.

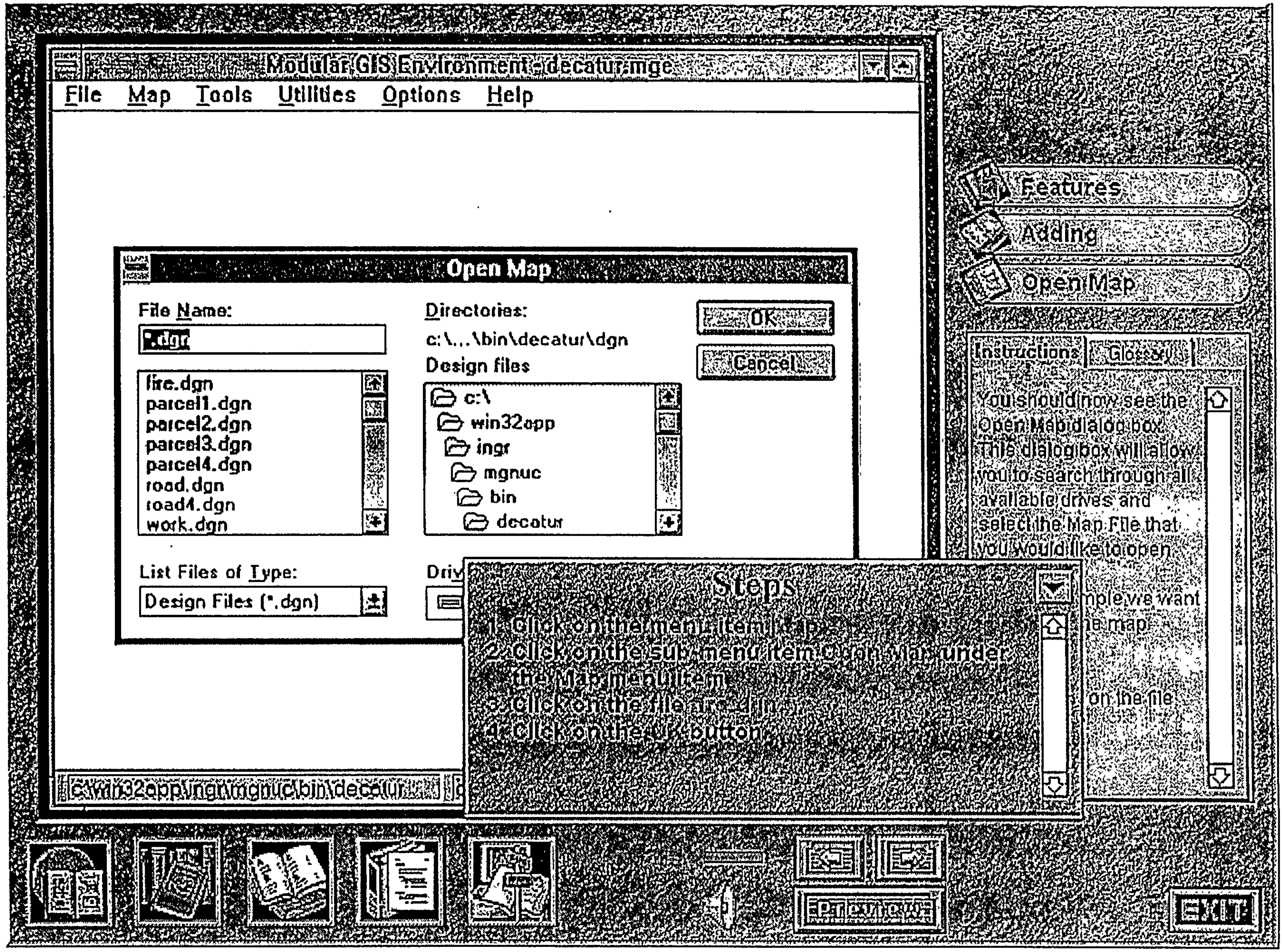
Please click on the Applications menu item now.



You should now see a list of menu items under the Applications menu.

Notice that the first item is MGE Base Mapper.

Please click on the MGE Base Mapper menu item now.



File Name:
*.dgn

fire.dgn
parcel1.dgn
parcel2.dgn
parcel3.dgn
parcel4.dgn
road.dgn
road4.dgn
work.dgn

List Files of Type:
Design Files (*.dgn)

Directories:
c:\...\bin\decatur\dgn

Design files

c:\
win32app
ingr
mgnuc
bin
decatur

Driv

c:\win32app\ingr\mgnuc\bin\decatur

Steps

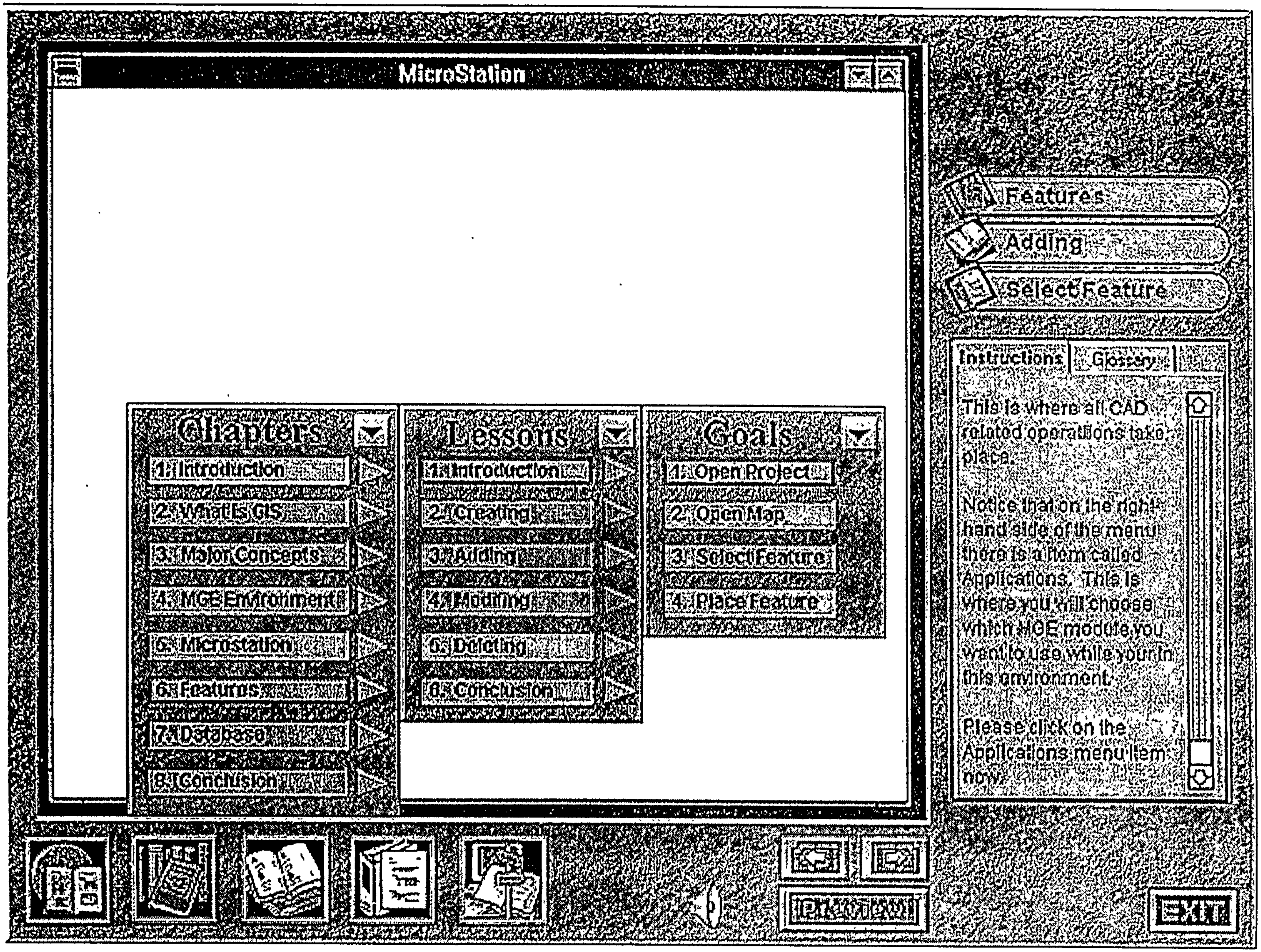
1 Click on the menu item Map.
2 Click on the sub-menu item Open Map under the Map menu item.
3 Click on the file fire.dgn.
4 Click on the OK button.

You should now see the Open Map dialog box. This dialog box will allow you to search through all available drives and select the Map File that you would like to open.

nple we want

e map

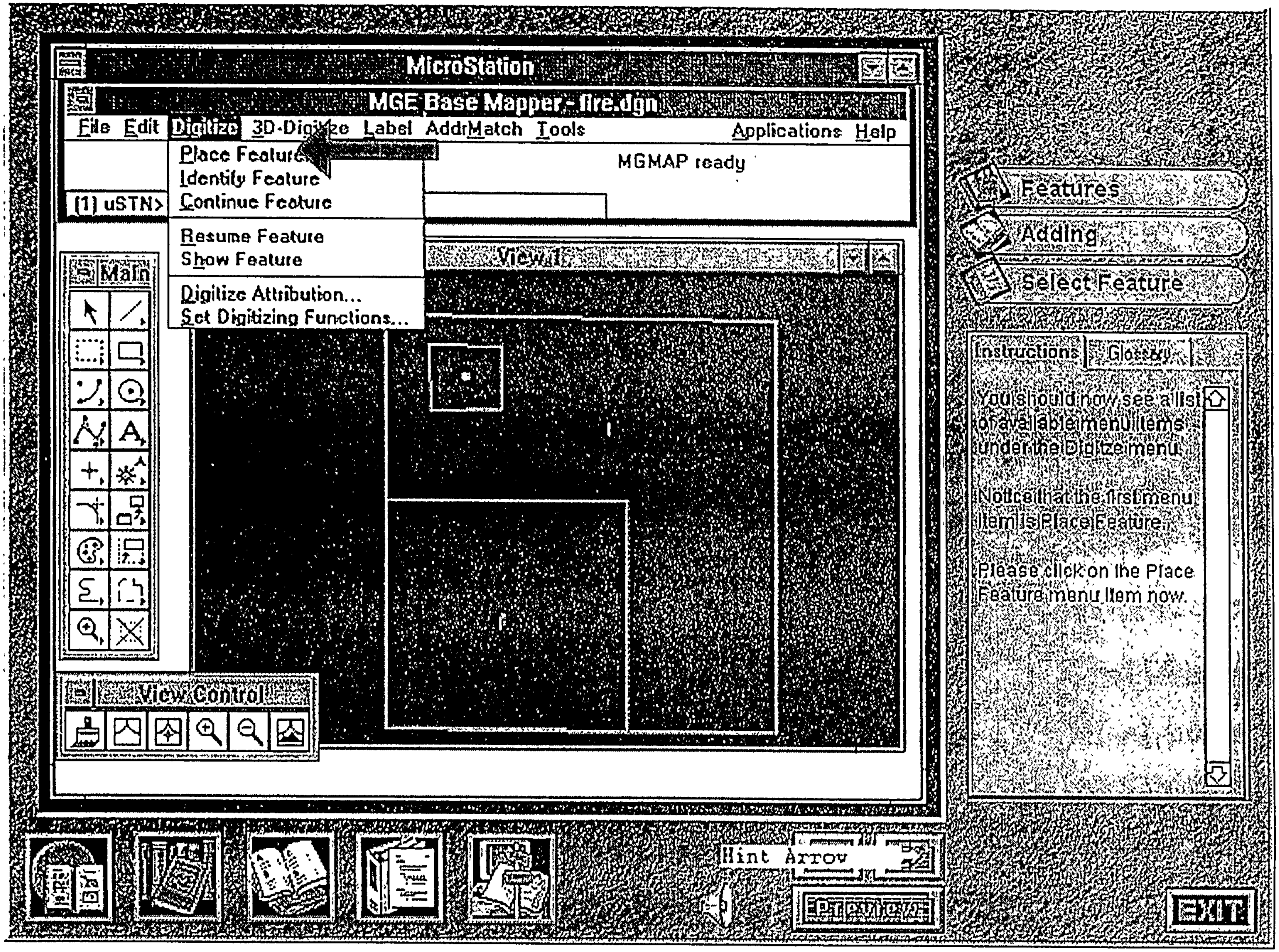
on the file



This is where all CAD related operations take place.

Notice that on the right hand side of the menu there is a item called Applications. This is where you will choose which MGE module you want to use while your in this environment.

Please click on the Applications menu item now.



You should now see a list of available menu items under the Digitize menu.

Notice that the first menu item is Place Feature.

Please click on the Place Feature menu item now.

Treatment of Mixed Wastes via Steam Reforming

METC Task No.2.2

Quarterly Technical Progress Report
Reporting Period: July 1 - September 30, 1996

Work Performed Under Contract
No.: DE-FC21-92MC29467

For:
U.S.Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By:
Dr. Richard Turton
Department of Chemical Engineering
West Virginia University
Morgantown, WV 26506-6102

DISCLAIMER

Abstract

This work addresses the evaluation of the destruction efficiency of RCRA organic components and surrogates in a steam reforming atmosphere. The benefit of using a steam atmosphere to destroy RCRA organics, e.g. dichlorobenzene, lies in the virtual elimination of dioxins and furans in the product gases. This technology has been proven by ThermoChem Inc., Columbia, MD, which currently is developing a two-stage fluidized bed unit to dispose of mixed wastes. The work outlined in this proposal addresses the quantification of the destruction efficiency of key RCRA organics and surrogates in the second stage of the ThermoChem process. A series of experimental runs will be performed in a 6.5 cm internal diameter reactor. The purpose of these runs is to evaluate the efficiency of destruction of organic materials as a function of residence time, gas composition and reaction temperature. In addition, the efficacy of using catalysts to improve the destruction efficiency will be determined.

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Executive Summary (Current Work)

During the current reporting period the destruction efficiencies of toluene and dichlorobenzene were evaluated. Two experimental runs using toluene were completed. Each run consisted of evaluating the destruction efficiency or conversion of the toluene at different space times and at a series of different temperatures. For temperatures of 1065°C (1950°F) and space times of approximately 2 seconds, the destruction efficiency of toluene in a steam reforming environment were measured to be 0.9993, for one sample, and below the detectable limit (>0.9999) for two other samples.

One experimental run was performed using dichlorobenzene (DCB). For the range of conditions used in the experimental run, a maximum destruction efficiency of 0.9983 was measured. This occurred at 965°C and a space time of approximately 1.7 seconds. At higher temperatures the destruction of DCB was found to decrease slightly, this anomalous result may be due to loading of DCB in the GC column and is currently being investigated.

1.0 Introduction

The treatment and destruction of mixed wastes is becoming an increasingly important problem in the environmental arena. Many of the problems associated with mixed wastes are due to the wide variety in the type, content and form of the waste itself. For example, clothing contaminated with small quantities of radioactive elements and sludges from fuel storage tanks would both be considered mixed wastes. The challenge, therefore, in dealing with this type of waste is to create processes which are flexible enough to handle a wide variety of feeds and to render them harmless to the environment. This research is aimed at determining the destruction efficiencies of RCRA organics in a variety of reforming atmospheres and at different temperatures. The work is essentially supporting an on going effort by ThermoChem Inc., Columbia, MD to develop a two stage process for the treatment of mixed wastes.

2.0 Purpose

There have been several reports regarding the destruction of organic materials at elevated temperatures in a variety of non-oxidative environments (1,2,3). However, the results are generally applicable to specific equipment and cannot be easily extrapolated to different operating conditions. The purpose, therefore, of this work is to systematically evaluate the destruction efficiencies of a variety of organic materials at different conditions and to establish correlations to support the design of new equipment.

This work relates directly to an existing process developed by ThermoChem Inc., Columbia, MD, and will be performed in close association with ThermoChem Inc.

3.0 Background

The ThermoChem Process utilizes a two stage fluidized bed system to volatilize and subsequently destroy organic materials. The process uses the first stage fluid bed to vaporize organic material from shredded mixed wastes and to concentrate the inorganic material in the remaining solids. The inorganic portion of the waste, containing heavy metal compounds etc., is eventually vitrified with glass frit in a separate unit. The gas from the first stage is sent to a second stage, operating at elevated temperatures (1600 - 1800°F), where the organics are reformed in a steam environment. In addition to steam, the ThermoChem process may have some hydrogen and carbon monoxide in the gas entering the second stage. The use of hydrogen and carbon monoxide as additional reforming gases and the possibility of using a catalyst to promote the reforming reaction are of interest in this process and will be investigated in this research.

It is envisioned that the results of this work will help to define the optimum conditions for the steam reforming process and to determine the efficacy of using a catalyst to promote destruction efficiency of RCRA organics and surrogates in such a process.

4.0 Methodology

The work proposed is a continuation of a current project under the WVU/METC cooperative agreement. The scope of the current project was modified to enable a closer working relationship with ThermoChem Inc. which currently is developing a process to destroy mixed wastes via steam reforming. At present, the test equipment consists of a 6.5 cm inside diameter stainless steel reactor which is expected to operate in the temperature range of 850 - 930°C (approx. 1600 - 1800°F). The bed is mounted in a vertical, 3-zone tube-furnace. The feed gas, mixtures of hydrogen, carbon monoxide and dioxide and steam, are fed from gas bottles and are metered through valves and flowmeters to the reactor. Organic liquid material is fed from a pressurized tank via a flowmeter and vaporized prior to being sent into the reactor.

The object of the current work is to quantify the destruction efficiency of RCRA organics and surrogates in a steam reforming atmosphere for a variety of operating conditions. A gas chromatograph, fitted with a flame ionization detector, will be used to evaluate the destruction efficiency of the organic material down to the 99.99% level of destruction or better. The need for a catalyst to improve the destruction efficiency will be addressed if the homogeneous reaction rates are too low. The tasks for this project are outlined below.

Task 1: Information Required for the National Environmental Policy Act.

Task 2: Upgrading and Calibration of Gas Chromatograph.

The gas chromatograph currently in use in Dr. Turton's laboratory will be upgraded with a Flame Ionization Detector (FID) to enable destruction efficiencies of up to and greater than 99.99% to be measured accurately. Once the GC has been upgraded a series of tests will be performed to calibrate the unit for measuring the organic components used in this work. Calibrations will consist of running the test bed at low temperatures (<200°C) using an inert or non-inert carrier gas and injecting (spraying) the organic material at different flowrates into the bed. Samples will be taken after the gas cooler and used to calibrate the peaks obtained from the GC. A minimum of three concentrations points, for each organic material, will be used to calibrate the gas chromatograph. Due to the widely differing characteristics of the organic materials used in this study it may be difficult to identify all the components in a reasonable time using a single column in the GC. Since the destruction efficiency of the RCRA organics and aromatic surrogates (in this case ethylene glycol, di-chlorobenzene, toluene and naphthalene) are the most important measures of the process's effectiveness, these will be evaluated and used as the measure of the destruction efficiency.

Task3: Experimental determination of Destruction Efficiency of Organic Material without the use of Catalyst.

The experimental program to be used in this part of the work will consist of an experimental test matrix in which the following independent variables will be varied:

	Number of Levels
Temperature	3 (1800, 1700 and 1600°F)
Residence Time	2 (1 and 2 s)
Hydrogen Content of Gas	3 (0, 5% and 10% on a volume basis)
Carbon Monoxide Content of Gas	2 (0 and 5% on a volume basis)

This test matrix is consistent with the needs of ThermoChem Inc. and was discussed with Mr. G.Voelker and Dr. R.Chandran of ThermoChem Inc. The organic materials to be tested were also discussed with ThermoChem Personnel and the materials given in Task 2 above will be used in the preliminary tests.

The dependent variables will be the destruction efficiencies of the organic components fed to the bed. Currently the organic feed material of interest consists of the following components: Toluene, DiChlorobenzene, Ethylene Glycol, Naphthalene. These components may be substituted with other RCRA components or surrogates, if the destruction efficiencies of other organics are desired.

Task 4: Correlation of Experimental Results

Using the results from Task 3 the conversion (destruction) of the different organic species will be correlated with the independent variables listed above in Task 3. The experimental results and correlation will be compared to existing homogeneous kinetics data in the literature. Any limitations, due to equilibrium considerations, will be investigated.

Task 5: Experimental determination of Destruction Efficiency of Organic Material using Catalyst.

The investigation into the use of catalysts to promote the destruction of the organic materials will be investigated if the results of Task 3 indicate that this is necessary. This phase of the work will involve input from ThermoChem Inc. The test matrix for part of the work will include operating temperature, gas composition, and gas-solid contact time in the reactor using a fixed organic feed. The mode of operation of the reactor will be a packed or fluidized bed and the particle size will be chosen to obtain the required gas residence time with a bed height of 24-30" of catalyst.

Task 6: Preparation of Quarterly and Annual Reports

Three quarterly and one annual report will be prepared for this project.

5.0 Worked Performed in this Period

The work performed in this period focussed on the experimental program outlined in Task

3. The two components that were studied in this reporting period were toluene and dichlorobenzene. The destruction efficiencies of these compounds over a range of temperatures, residence times and gas compositions were evaluated. The experimental methods and major results for these experimental runs are described in the following sections.

5.1 Experimental Procedure

The experimental procedure, which was followed for each run, was straightforward and is described with reference to Figure 1. The three zone furnace was set to the desired temperature and the bed allowed to come to steady state, as determined by the measurements of three thermocouples inside the bed. When the temperatures indicated by the thermocouples in the heated zone have not changed over a five minute period the bed is deemed to have come to steady state. This usually takes 3-4 hours from the start of the experiment and around 1 hr when the bed temperature is changed from one temperature to the next. It is noted that the third thermocouple, T3, lies outside the heated section of the reactor and is not used to determine the steady state condition when changing the reactor temperature. The reading of this thermocouple does change slightly with time but simply reflects the slow temperature rise of the small amount of packing outside the heated zone. During the reactor preheat stage, the flow of water to the vaporizer and the flow of carrier gas are set to predetermined values and started. After 1 hour or so the liquid from the condenser is measured over a timed period to check the material balance. Typically 90-92% of the water charged to the reactor is collected from the condenser. Another 1-2 % is accounted for in the saturated carrier gas which is vented to the atmosphere. The remaining water is assumed to be lost through minor leaks, etc.

After the bed has reached steady state and the material balance has been successfully established, the flow of organic material is initiated. The flow is controlled by adjusting the setting of the peristaltic pump which transports the organic material from the storage vessel to the organic vaporizer. The vaporizer temperature is set above the normal boiling point of the organic to ensure vaporization. The vaporized organic is mixed with the steam and carrier gas prior to being fed to the base of the packed bed reactor. The bed is allowed to obtain steady state conditions prior to sampling the exit gas stream.

A second test is then made to ascertain the material balance. This is achieved by measuring the concentration of organic material in the reactor effluent stream at the lowest temperature used in the experiments. From previous experience, it has been determined that the destruction of the organic material at temperatures at and below 750°C is negligible. Therefore, by knowing the flows of all materials into the reactor, the concentration of organic in the reactor effluent can be both calculated and measured. Three samples are taken of the effluent gas (2 ml) and are injected into the Gas Chromatograph (SRI model 8100 with FID). Consistency between the samples and with the calculated concentration signifies that the material balance is correct. It should be pointed out that

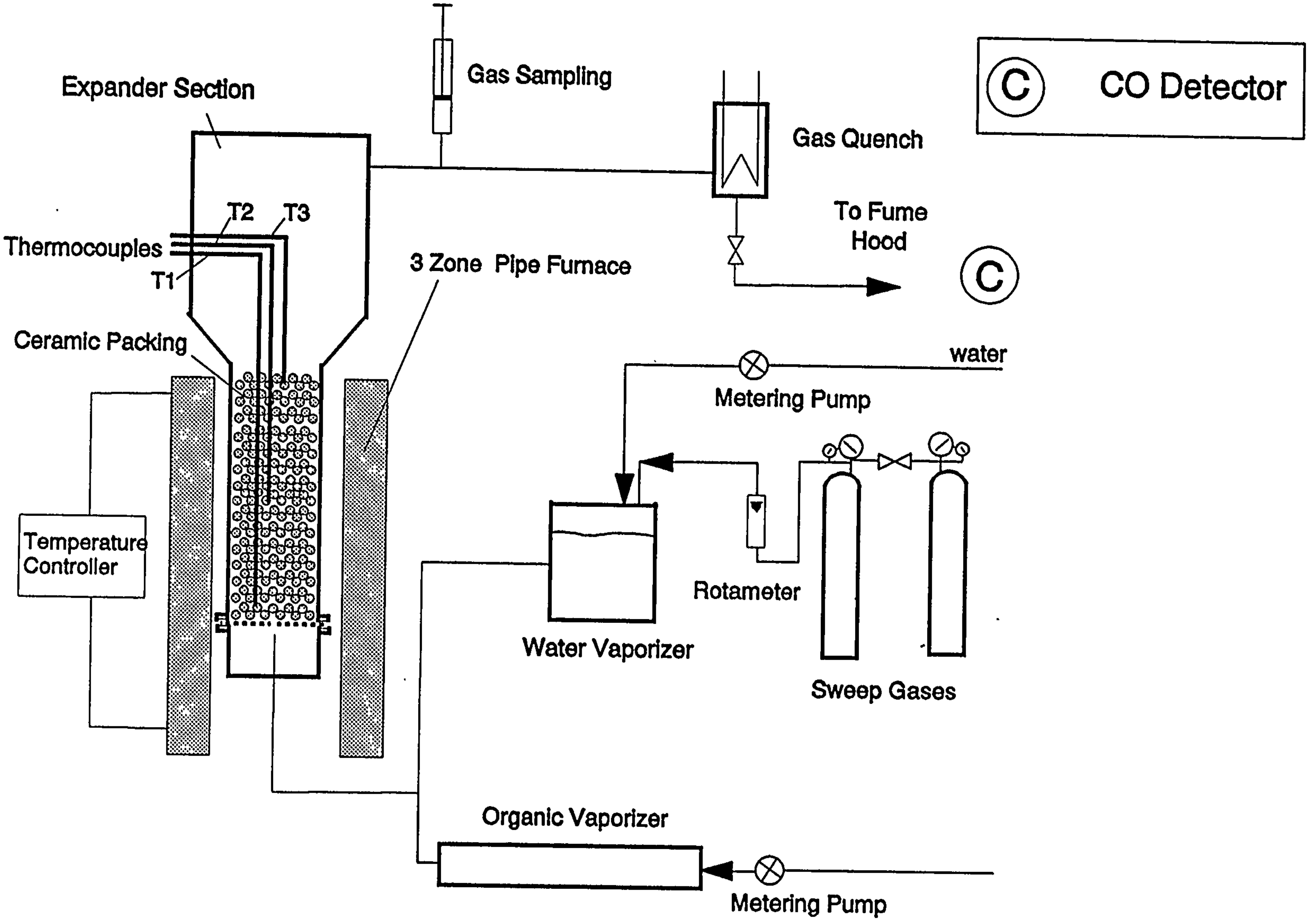

Figure 1: Experimental Set-Up for Steam Reforming of Organics

not until the material balance has been checked by both the methods described above, and shown to be consistent, is the experiment allowed to continue.

5.2. Data Analysis

In obtaining concentration data using the GC, it has been observed that considerable deviations in the area count from the chromatograms for samples taken at the same operating conditions can occur. Originally large changes in area counts for identical conditions were observed, but much of this was due to flow pulsations in the water vaporizing system and have consequently been eliminated. However, variation in the area counts from the GC still occur. For example, area counts of 14,513, 15,879 and 15,279 were recorded for the reactor effluent in one experiment using toluene at a reactor temperature of 750°C. These data show a mean value of 15,224 and a calculated standard error of 485 area units or about 3%. At a temperature of 1050°C, all other experimental conditions being kept the same, the average of the three readings was 71.2 with a standard error of about 20%. The larger scatter of experimental data at higher temperatures and hence high conversions (low concentrations) has been observed for all the test runs. This leads to a higher error bar at lower concentrations. Currently, it is not clear why this trend in the data is present. These errors are not due to limitations of the instrumentation, since area counts of down to 1 and less are reproducible for standard samples using the current GC. It is believed that the impact of these errors on the computed destruction efficiency are not great, except for very high conversions. Research into why these fluctuations in concentration are present will continue. It is also noted that until the concentration of organic material decreases appreciably the conversion or destruction is assumed to be negligible and set to zero (see Tables 1, 2, and 3). The computed destruction efficiencies are obtained by back calculating the conversion which would be necessary to give the area count measured by the GC.

5.3 Experimental Runs using Toluene

The conditions used in the experimental runs with toluene are summarized in Tables 1 and 2 and the destruction efficiencies are plotted as a function of temperature in Figure 2. The mole fraction of components in the inlet gas to the reactor are calculated based on the flowrates of the components in the feeds. The three reactor temperatures refer to the three thermocouples inserted into the packed bed. It should be noted that third thermocouple (T3) is placed at the very top of the packed section, outside of the heated zone, and takes a long time to reach a steady temperature. The determination of steady state is made with the two thermocouples (T1 and T2) which lie within the heated section of the reactor. These temperature readings more accurately reflect the conditions in the main part of the reactor.

Table 1: Experimental Conditions and Results for Toluene Experiments (Run T1)

Sample	Reactor Temperature (°C)			Mole Fraction in Inlet Gas			Area Count	%Destr	Space Time (s)
	T1	T2	T3	Toulne	Steam	Nitrogn			
1	606	603	560	0.013	0.746	0.241	18,034	0	1.86
2	606	603	565				16,716	0	1.86
3	606	603	570				15,050	0	1.86
1	740	730	617	0.013	0.746	0.241	14,513	0	1.59
2	750	760	637				15,879	0	1.59
3	750	760	657				15,279	0	1.59
1	855	856	735	0.013	0.766	0.221	7,500	50.12	1.43
2	862	866	745				8,767	40.86	1.43
3	865	864	757				7,836	44.22	1.43
1	950	960	810	0.013	0.746	0.241[1]	1,093	92.17	1.33
2	958	962	820				1,629	88.68	1.33
3	960	960	832				1,060	91.63	1.33
1	1052	1050	854	0.013	0.746	0.241	93.5	99.31	1.23
2	1068	1065	875				57.3	99.64	1.23
3	1068	1067	889				62.7	99.53	1.23

[1] Carbon Dioxide was used instead of nitrogen for experiments at a reactor temperature of 850°C

Table 2: Experimental Conditions and Results for Toluene Experiments (Run T2)

Sample	Reactor Temperature (°C)			Mole Fraction in Inlet Gas			Area Count	%Destr	Space Time (s)
	T1	T2	T3	Toulne	Steam	Nitrogn			
1	766	761	610	0.0105	0.7691	0.2202	12,907	0	2.42
2	765	762	620				15,628	0	2.42
3	765	764	638				14,839	0	2.42
1	865	861	675	0.0107	0.7655	0.2238	11,249	21.88	2.20
2	865	862	672				9,867	31.12	2.20
3	865	862	688				8,356	41.05	2.20
1	962	962	750	0.0105	0.7693	0.2201	167	98.72	2.02
2	965	962	760				154	98.85	2.02
3	964	961	782				126	99.08	2.02
1	1065	1063	785	0.0105	0.7693	0.2201	9.68	99.93	1.87
2	1065	1062	788				BDL	BDL	2.10
3	1065	1064	780				BDL	BDL	2.40

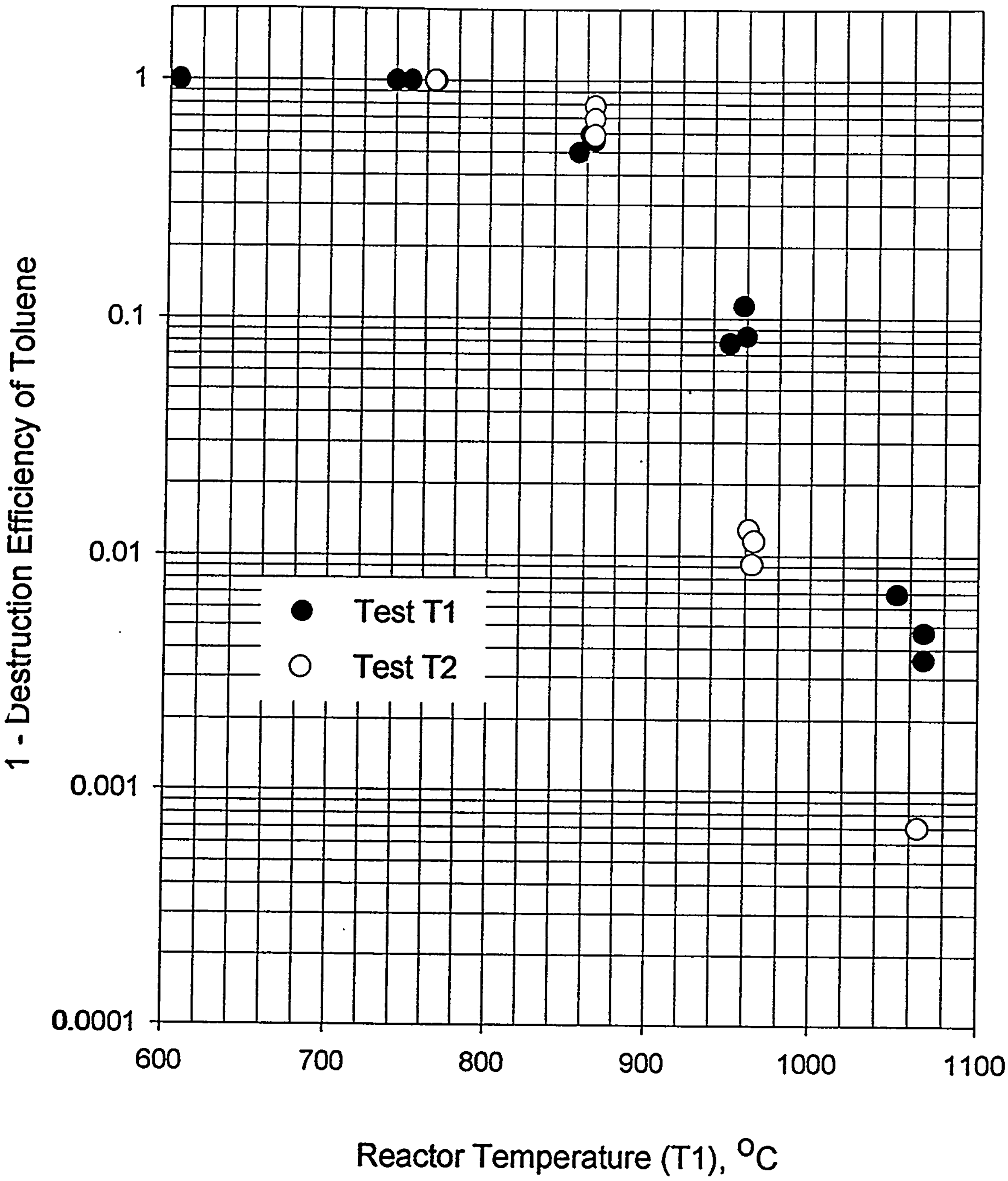

Figure 2: Results for Experimental Runs Using Toluene

5.4 Experimental Runs using Dichlorobenzene

The conditions used in the experimental runs with dichlorobenzene are summarized in Table 3 and the destruction efficiencies are plotted as a function of temperature in Figure 3. Similar comments to those made for toluene, regarding the placement of the thermocouples and estimation of inlet composition, also apply to this set of data.

6.0 Results and Discussion for Current Reporting Period

The experimental program, to investigate the destruction efficiency (conversion) of organics in a steam reforming atmosphere, was continued in this period. Destruction efficiencies of 99.93% and greater were obtained for the highest temperature runs (1050 °C) using toluene. The main differences between the two toluene runs was the use of lower space times in the T1 data set. This is believed to be the primary reason for the lower destruction efficiencies (conversions) in this run compared to the T2 data set. It is important to note, that a kinetic model cannot be reliably calculated from this data due to the fact that the space time changes for each temperature. As more data is collected, multivariable regression techniques will be used to back calculate kinetic model parameters and the appropriate form of the kinetic model.

The destruction efficiencies for the dichlorobenzene run (DC1) show a maximum value of around 99.8% at reactor temperatures of approximately 960 °C. At higher temperatures the destruction efficiency appears to go down slightly. Since doing this run it has been observed that the GC column is sensitive to dichlorobenzene loading. Consequently, after several consecutive runs the column starts to retain dichlorobenzene. If the column is not conditioned at this point subsequent measurements are found to be falsely high. It is believed that this is the reason for the observed decrease in destruction efficiency observed at the highest temperature in the DC1 data set. A new protocol is currently being developed to alleviate this problem in the future.

7.0 Conclusions for Current Period

The experimental results show that high destruction efficiencies (99.93% and greater) are possible for toluene in a steam reforming atmosphere at temperatures of approximately 1065 °C (1950 °F). Lower destruction efficiencies were obtained for dichlorobenzene at similar conditions. However, the observed results for dichlorobenzene at 1065 °C may be in error due to the loading of the GC column. This data set will be repeated using a new GC column conditioning protocol.

Table 3: Experimental Conditions and Results for Dichlorobenzene Experiments (Run DC1)

Sample	Reactor Temperature (°C)			Mole Fraction in Inlet Gas			Area Count	%Destr	Space Time (s)
	T1	T2	T3	DCB	Steam	Carbon Dioxide			
1	650	650	590	0.0092	0.8105	0.1802	15,379	0	2.20
2	665	660	610				14,782	0	2.20
3	665	660	630				14,531	0	2.20
1	766	761	640	0.0092	0.8105	0.1802	15,525	0	1.99
2	765	762	650				14,871	0	1.99
1	865	861	680	0.0092	0.8105	0.1802	6,370	52.55	1.81
2	865	862	700				4,500	66.15	1.81
3	865	862	730				5,014	62.28	1.81
1	962	962	750	0.0092	0.8105	0.1802	81	99.36	1.66
2	965	962	760				32	99.75	1.66
3	964	961	782				21	99.83	1.66
1	1065	1063	785	0.0092	0.8105	0.1802	45	99.66	1.54

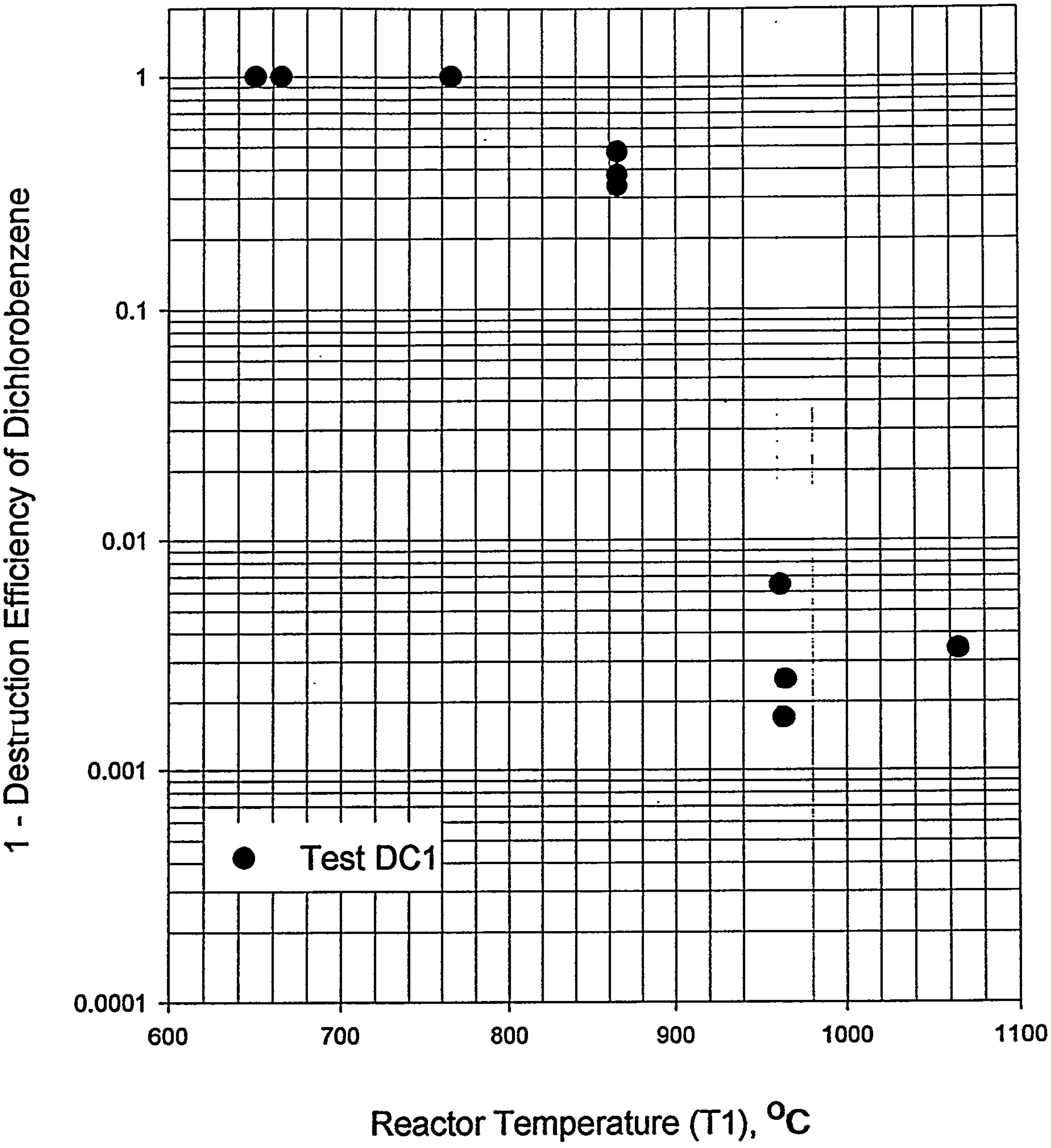

Figure 3: Results for Experimental Runs Using Dichlorobenzene

8.0 Work Planned for Next Period

The experimental program outlined in Task 3 will be continued during the next period. The two compounds used during this reporting period will be used for the remainder of the experimental work and once sufficient data has been collected, the parameters for a kinetic model will be evaluated. Currently there is no plan to evaluate the catalytic destruction of organics during the next reporting period. The results of the work to date suggest that thermal destruction will be sufficient to obtain destruction efficiencies of four nines or better.

9.0 Technical and Administrative Difficulties for Current Period

As described in Sections 5 and 6, there is significant variability in the data being collected which will impact the accuracy of the destruction efficiencies at the highest temperatures used in this work. A new protocol for conditioning the GC column will be developed to help alleviate some of these problems.

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Acronyms and Abbreviations

FID - Flame Ionization Detector
GC - Gas Chromatograph
RCRA - Resource Conservation and Recovery Act (1976)
SRI - Scientific Research Instruments, Inc.
DCB - Dichlorobenzene

USE OF CENTRIFUGAL MEMBRANE TECHNOLOGY WITH NOVEL MEMBRANES TO TREAT HAZARDOUS/RADIOACTIVE WASTES

METC Task No. 2.3

Quarterly Technical Progress Report
Reporting Period: 7/1/96 - 9/30/96

Work Performed Under Contract
No.: DE-FC21-92MC29467

For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Brian E. Reed, Associate Professor
Wei Lin, Research Assistant Professor
Department of Civil and Environmental Engineering, West Virginia University
PO Box 6103, Morgantown, WV 26506-6103
304.293.3031 ext. 613, 304.293.7109 (Fax)

DISCLAIMER

ABSTRACT

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks packs set on a hollow rotating shaft inside a cylindrical housing. The waste stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate is forced through the membrane and is collected in the hollow shaft and discharged. The concentrate exits at the edge of the membrane packs.

The centrifugal action continuously cleans the membrane surface decreasing cleaning chemical use and maintenance and increasing the volume of waste treated per area of membrane. Because of the reactive nature of most DOE wastewaters, the use of conventional membrane materials may not be viable. Thus, specialty membranes that can separate hazardous contaminants from the permeate stream and are chemically and radiologically stable will also be tested.

The objective of this research is to investigate the feasibility of using centrifugal membrane technology combined with innovative membranes for the of treatment hazardous/radiological wastes. The following tasks will be completed during this research: 1) perform extensive literature review on membrane technology, 2) identify and acquire waste and membranes, 3) modify, acquire, and be trained in the use of SpinTek technology, and 4) test and evaluate SpinTek technology and specialty membranes.

The following variables will be monitored as a function of run time: 1) contaminant concentration in concentrate and permeate, 2) membrane flux, 3) temperature and pressure, 4) conductivity and pH of all liquids. The following parameters will be used to judge the effectiveness of the process: 1) contaminant concentration in the permeate, 2) physical and chemical resistance of membranes, 3) volume and waste reduction factors, 4) quantity and quality of residuals, 5) energy and requirements, 6) ease of operation and likelihood of process upsets.

The result of this work will be a technology that can treat a variety of wastes such as contaminated groundwater, mixed-waste process water, and existing waste residual (*e.g.*, low-level waste). The process can be used for both inorganic (*e.g.* radionuclides and heavy metals) and organic contaminants. It is envisioned that the volume reduction factors for this technology will be high and a relatively a low amount of residual requiring additional treatment will be produced.

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EXECUTIVE SUMMARY

In the third quarter the effect of membrane type (polymeric versus ceramic) on the performance of the SpinTek technology was investigated. A total of six runs were conducted using an industrial coolant waste that had an oil content of approximately 5%. This type of waste represents one of the more difficult waste to treat using ultrafiltration thus its use tests the extreme performacne limits for both membrane types. Three runs were conducted using the polymeric membrane and three runs were conducted with the ceramic membrane. For the majority of the runs the system pressure was between 42 and 43 psi, the rotational speed was 1750 rpm, and the feed tank temperature was 110°F. For several runs, these system paramerters were changed for a relatively short period of time to ascertain their effect on process performance. The polymeric membrane (PV-100K) had a molecular weight cut-off of 100,000 and the average pore size of the ceramic membrane was 0.1 µm.

Based on data obtained during this portion of the research, it appears that the SpinTek centrifugal system equipped with a ceramic membrane is able to satisfactorly treat a concentrated oil/grease waste stream. Such a waste stream exists at the RMI facility in Astabula, Ohio.

The ceramic membrane is superior to the PV-100K polymeric membrane for the following reasons: 1) the ceramic membrane had a significantly higher flux, 2) the maximum operation temperature for the PV-100K membrane is 120°F, compared with >160°F for the ceramic membrane (it is advantageous to run the system at the highest temperature possible as this results in a larger flux), and 3) permanent fouling occurred in all three PV-100K runs indicating membranes would have to be installed frequently in full-scale operation. No permanent fouling was observed for the ceramic membrane.

For the PV-100K membrane, permeate quality deteriorated significantly during batchdown operation. For the ceramic membrane Runs 2 and 3, there was no difference in permeate quality (turbidity, O/G and TSS) between semibatch operation and batchdown. Turbidity appears to be a good real-time indicator of permeate TSS and O/G concentrations. Turbidity and permeate flux can be used to judge when the system should be cleaned.

Increasing the temperature from 110 to 140°F increased the average flux by about 45 percent (220 to 319 gal/ft^2-d). Only a portion (about 24 %) of the increase in flux could be attributed to the decrease in viscosity with increasing temperature. For the ceramic membrane, the maximum operational temperature of the full-scale centrifugal system will most likely be determined by other portions of the system (*e.g.*, fittings).

Decreasing the rotational speed decreased the permeate flux and the flux did not recover when the rotational speed was increased back to its maximum value (1750 rpm). It is hypothesized that when the rotational speed was initially decreased, the membrane became fouled and subsequent increases in the rotational speed were not able to clean the membrane effectively. Thus, in a full-scale system, the maximum rotational speed should be used. Finally, permeate water quality (turbidity O/G and TSS) were not affected by temperature or rotational speed excursions.

1.0 INTRODUCTION

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks packs set on a hollow rotating shaft inside a cylindrical housing. The waste stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate is forced through the membrane and is collected in the hollow shaft and discharged. The concentrate exits at the edge of the membrane packs. The centrifugal action continuously cleans the membrane surface decreasing cleaning chemical use and maintenance, increasing the volume of waste treated per area of membrane, and increasing the contaminant/solids concentration in the residual (*i.e.* increases volume reduction factor). SpinTek's centrifugal membrane technology represents an improvement over conventional ultrafiltration technology and increases the number of waste streams that can be treated using membrane treatment.

2.0 PURPOSE

The purpose of this research is to investigate the feasibility of using centrifugal membrane technology combined with innovative membranes for the treatment hazardous/radiological wastes. The result of this work will be a technology that can treat a variety of wastes such as waste from DOE decontamination/decommissioning efforts, biological wastes, and waste with a high solids content (*e.g.*, low level tank waste), contaminated groundwater, mixed-waste process water, and existing waste residual. The process can be used for both inorganic (*e.g.* radionuclides and heavy metals) and organic contaminants. It is envisioned that the volume reduction factors for this technology will be high and a relatively a low amount of residual requiring additional treatment will be produced.

3.0 BACKGROUND

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. A schematic of the SpinTek membrane vessel is presented in Figure 1. The SpinTek system uses a series of flat, round membrane disks packs set on a hollow rotating shaft inside a cylindrical housing. The fluid stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate ("clean" liquid) is forced through the membrane and is collected in the hollow shaft and is discharged. The concentrate (liquid containing the contaminants) exits at the edge of the membrane packs.

With time, the membrane surface will become fouled resulting in a decrease in the permeate flow rate. To reduce fouling in conventional membrane systems, a large portion of the concentrate is recycled back to the membrane unit producing large liquid velocities near the membrane surface. The large velocities increase turbulence which reduces the thickness of the fouling layer. In centrifugal membrane systems, the rotation of the membrane disk induces the turbulence required to minimize the thickness of the fouling layer. In conventional membrane systems, maximum surface velocities of about 15 ft/s are possible while with centrifugal systems surface velocities of 60 ft/s are typical. As the concentrate thickens with treatment time, conventional systems are not able to maintain the high velocities because of the difficulty in pumping viscous material at large flow rates. Because the centrifugal membrane system does not rely on pumping to produce the required surface velocities, extremely concentrated wastes can be treated.

Membrane systems are typically operated in either the semi-batch mode or the batch mode. In Figure 2, a schematic of the pilot-scale SpinTek unit is presented. In the semi-batch mode, fresh waste is added to the system at a rate equal to the permeate production rate. In the batch-mode, which normally occurs at the end of semi-batch operation, no fresh waste is added to the system and the concentrate remaining from semi-batch operation is concentrated further ("batch down"). During batch-down, large concentration factors are achieved (permeate is still being produced reducing the concentrate volume).

All membrane technologies are volume reduction technologies because contaminants are not degraded or destroyed. Thus, an important measure of the efficiency of membrane technology is the concentration factor (CF). The CF during semi-batch and batch-down operation are calculated using the following two equations:

Semi-Batch Operation

$$CF_{SB} = 1 + V_{perm}/V_{feed\ tank}$$

Batch-Down Operation

$$CF_{BD} = CF_{SB} \times [V_{feed\ tank}/(V_{feed\ tank} - V_{perm})]$$

where,

CF_{SB} = concentration factor during semi-batch operation.

V_{perm} = volume of permeate produced, gal.

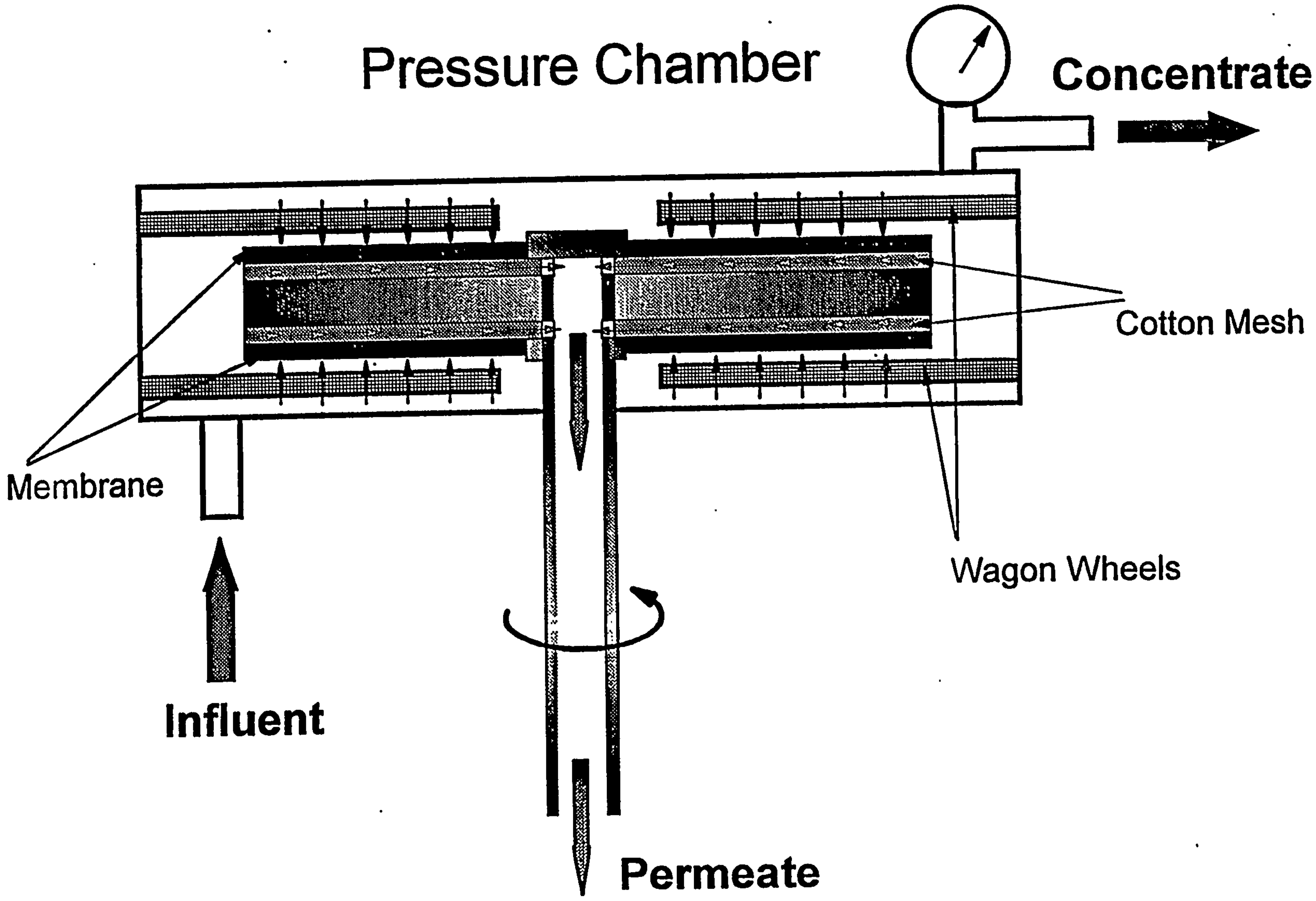

Figure 1. Schematic of SpinTek Membrane Vessel.

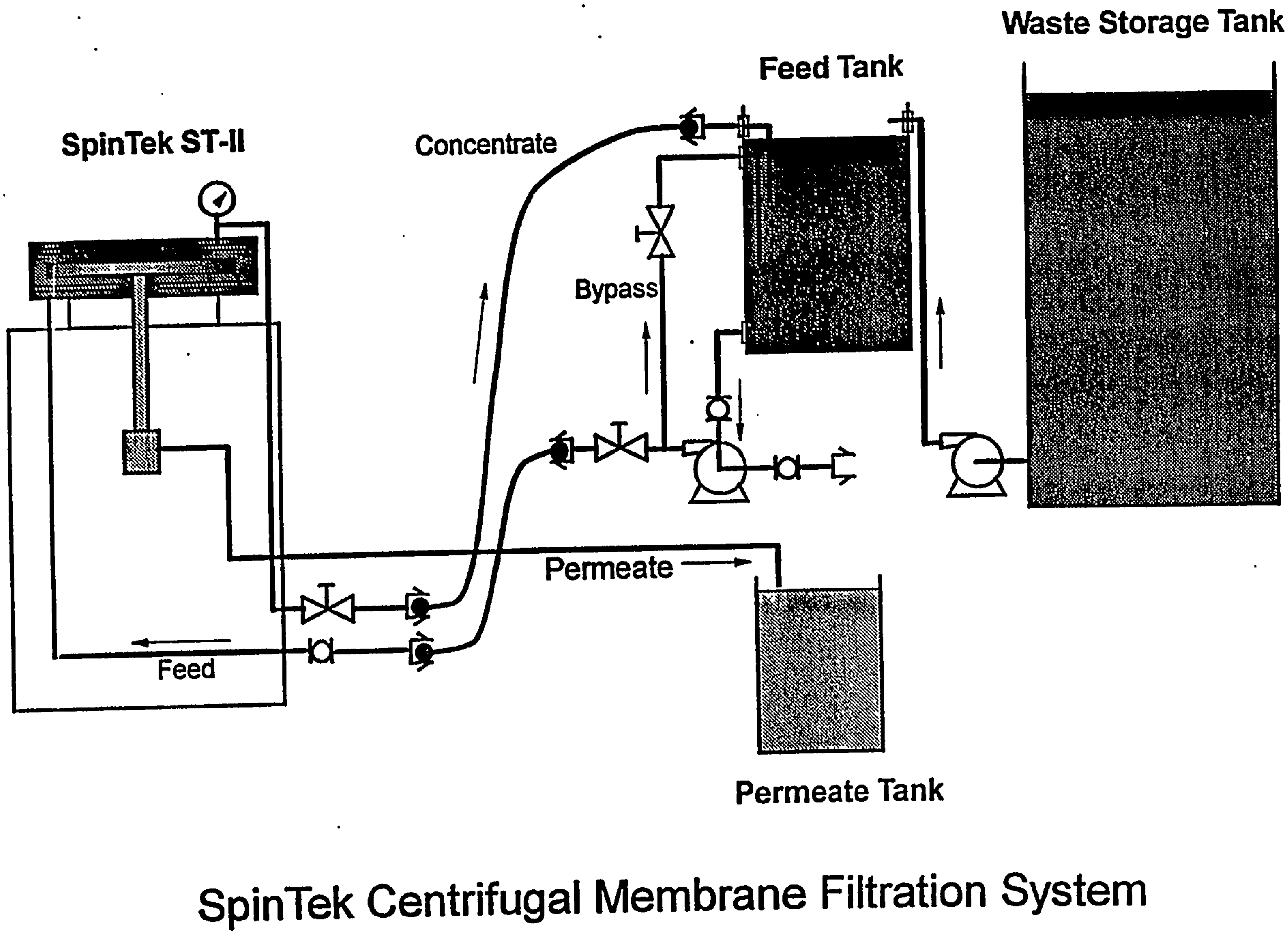

Figure 2. Schematic of the Pilot-Scale SpinTek Unit.

$V_{feed\ tank}$ = volume of the feed tank (15 gal), gal.
CF_{DB} = concentration factor during batch-down operation.
Concentration factors are expressed as 1X, 2X, *etc.*, and increase with treatment time. CFs in excess of 100X are obtainable with the membrane technology. Another measure of treatment efficiency is the permeate flux (volume of permeate/membrane area per time). The permeate flux for a given membrane is dependent on the waste's viscosity, solids content, and temperature. These waste characteristics can also change during treatment.

Membrane's that are used for ultrafiltration are characterized by the molecular weight of a molecule that is not allowed to pass through the membrane. For example, the membrane used in this quarter's research had a molecular weight cut-off (MWCO) of 100,000. In theory, compounds having a molecular weight greater than the MWCO would be retained by the membrane and compounds with molecular weights less than the MWCO would pass through the membrane and be contained in the permeate. It should be noted that the MWCO designation is somewhat misleading because a molecule having a molecular weight less than the membrane's MWCO may still be retained by the membrane because its unique geometry.

4.0 METHODOLOGY

Centrifugal membrane technology will be tested using several combinations of wastes and membranes. For each membrane type, the clean water flux (CWF) will be determined for the virgin membrane. The CWF serves as a baseline indicator to determine if the membrane is 1) permanently fouled and 2) sufficiently cleaned. For each waste-membrane combination, the SpinTek system will be operated first in the semi-batch mode and then at the end of the treatment run in the batch mode. The following variables will be monitored during technology testing:

1. Contaminant concentration in concentrate and permeate.
2. Membrane flux.
3. Temperature and pressure.
4. Conductivity and pH of all liquids.

Permeate flux, contaminant(s) concentration in the permeate, concentration factor, operational ease, and the membrane's physical/chemical resistance and cleanability will be used to judge the effectiveness of the process. Several runs will be conducted for each membrane-waste combination. Special attention will be placed on identifying the quantity and quality of any residuals produced. Quality assurance/quality control practices, as outlined in US EPA's Test Methods for Evaluating Solids Wastes, will be followed. The use of standard analytical methods (*i.e.*, ASTM, Standard Methods for the Measurement of Water and Wastewater, etc.) will also be followed. Details on how each run was conducted will be provided in Chapter 6 -Results and Discussion.

5.0 WORK PERFORMED DURING THIS PERIOD

The following work was conducted during the period 7/1/96 to 9/30/96: 1) continuation of literature search, 2) waste and membrane identification and acquisition, and 3) technology testing.

1.1 Literature Search

A comprehensive literature search on membrane technology was conducted in the first quarter of this research and over 100 articles and reports have been acquired to date. As new relevant documents come to light the material will be requested through West Virginia University's Interlibrary Loan program.

1.2 Waste and Membrane Identification and Acquisition

The identification of wastes and membranes that warrant further study will be on ongoing task for this research project. In this quarter a ceramic membrane having an average pore size of 0.1 μm was acquired. An industrial coolant having an (O/G) content of about 5% was also acquired. The use of actual waste as opposed to synthetic is more economical and provides a better opportunity to judge system performance. In addition, numerous researchers have investigated the treatment of (O/G) wastes using conventional ultrafiltration systems thus, a comparison between the SpinTek and conventional systems can be made. Also, because an O/G waste is one of the more difficult wastes to treat, the limits of the SpinTek system can be established.

1.3 Technology Evaluation

In the third quarter, the SpinTek technology with either a polymeric or ceramic membrane was used to treat a coolant waste having an oil content of about 5%. Results and discussion are presented in Section 6.0. A direct comparison of the performance of the system with a ceramic and a polymeric membrane will be presented.

6.0 RESULTS AND DISCUSSION

In this chapter results from the treatment of an industrial coolant using a polymeric membrane having an MWCO 0f 100,000 and a ceramic membrane having an average pore size of 0.1 µm will be presented and discussed. The industrial coolant had an oil content of about 5%. Tables and Figures are included at the end of the text portion of Section 6.0. Results will be presented for each run separately and then compared at the end this chapter. Results consist of a description of the waste aquisition, system preparation, system operation, and experimental data and discussion.

RUN 1 - POLYMERIC MEMBRANE

Waste Acquisition

One 55 gallon drum containing 50 gallons of coolant was received from a West Virginia industry.

System Preparation

Prior to the start of this run, a virgin polymeric membrane (PV-100K)was installed in the SpinTek system. Characteristics of the polymeric membrane are presented in Table 1. After installing the polymeric membrane in the centrifugal UF unit, the clean water flux (CWF) was measured using tap water at a temperature of 110°F. The feed tank was filled with 118°F tap water and then maintained at 110°F using a heat exchanger When the temperature had reached 110°F, the system was started and the system parameters set adjusted as follows: 1) vessel pressure = 42 psi, 2) rotational speed = 1750 rpm, 3) return flow = 1.2 gal/min, and 4) feed tank temperature = 110 ± 2°F . Permeate flux was measured every 5 minutes until it had stabilized for a 30 minute period. The average clean water flux was 118 gal/ft^2-d.

System Operation

Following the CWF determination, the system was drained and coolant was pumped from the drum through a 100 mesh sieve into the wastewater storage tank (see Figure 2). The total volume of coolant was 50 gallons. The coolant was then mixed and samples were taken for O/G, TSS and pH. O/G and TSS samples were preserved according to Standard Methods. pH was measured immediately.

Following initial sampling, 14.5 gallons of the coolant (V_{system}) were pumped from the storage tank to the feed tank. The system was then started and the temperature was allowed to increase to 110°F using heat/energy from the pump and the rotational device. After reaching 110°F, the temperature was maintained at 110 ± 2°F using a heat exchanger. The operation conditions were as follows: membrane rotational speed = 1750 rpm, vessel pressure = 40-43 psi, return flow rate = 1.2 gal/min, V_{system} = 14.5 gal (for semi-batch operation, feed tank volume decreased with time during batchdown). Permeate flow rate and turbidity, feed tank temperature, and vessel pressure were measured frequently. A calibrated 15 gallon carboy was used to collect the permeate. The hydraulically-based concentration factor was calculated by measuring the volume of permeate produced (V_{perm}) and the Equations 1 and 2.

In this run, the system was operated in semi-batch mode until a concentration factor of 3.45X was reached (35.5 gallons of permeate accumulated, 23.2 hours). At this point, batchdown operation began and was stopped at CF = 9.1X (5.5 gallons of concentrate left in the system, 8.6 hours). At predetermined values of the CF, the permeate was sampled for O/G, TSS, COD and pH.

At the conclusion of batchdown operation, all concentrate was drained from the system and its volume measured. The concentrate was saved and used in residual treatment experiments (to be described in a subsequent report). The system was then flushed using tap water at ≈135°F until the liquid from the return hose was clear. At this point the system was drained of all liquid and washed. The washing solution consisted of 181 grams of *Biz* detergent and ≈30 mL of *Dawn* dishwashing liquid in 5 gallons of hot tap water (≈135°F). The pH of the washing solution was 10.3. The washing solution was added to the feed tank and the system was operated for about 50 minutes at a temperature between 125 and 135°F. Following washing, the washing solution was drained and the system flushed again using tap water. It is important to remove all washing solution from the system because a small amount of soap can increase the subsequent clean water flux. In full-scale systems this is not critical and some membrane manufacturers suggest that the washing solution remaining in the membrane tubes not be emptied, as this improves the subsequent treatment flux. Following the second flush, the feed tank was filled with 10 gallons of 110°F tap water and the clean water flux was determined as described earlier. After determining the CWF, the membrane was visually inspected. If the CWF was not close to the CWF taken prior to the run, the cleaning step was repeated. If after visual observation it was determined that the membrane was permanently fouled, it was replaced.

Experimental Data and Discussion

In Table 2, a summary of the permeate results is presented. In Figure 3, the permeate flux and concentration factor versus time are presented. In Figure 4, the feed tank temperature versus operational time is presented. During the first 2.75 hours, the feed tank temperature increased from 74 to 110°F and the flux increased correspondingly. The importance of temperature on the permeate flux can be ascertained by comparing the initial portion of the permeate flux and temperature graphs. As the temperature increases, the viscosity of the water decreases, making it easier to push water through the membrane. The size of the full-scale system is related to the allowable operational temperature (higher temperature → higher permeate flux → smaller full-scale system). Based on previous work, the maximum operational temperature (excluding cleaning) for the polymeric membrane is about 120°F. Thus, for the polymeric membrane, the permeate flux is limited to a large extent by the membrane material. As will be discussed later, ceramic membranes offer the advantage of being able to operate effectively at much higher temperatures (≈160°F)

Disregarding the initial 2.75 hours, the flux was fairly stable (≈75 gal/ft^2-d) up to about 20 hours, after which it declined slightly to about 65 gal/ft^2-d. The average flux during semibatch operation (excluding the first 2.75 hours) was 73 ± 4.8 gal/ft^2-d. During batchdown, the flux decreased from 64 to 26 gal/ft^2-d. The average flux during batchdown operation was 50 ± 11.7 gal/ft^2-d. The average flux for the entire run (excluding the first 2.75 hours) was 64 ± 14 gal/ft^2-d.

The flux decreased more rapidly during batchdown operation because the O/G and TSS concentration in the feed tank increased sharply. The O/G and TSS accumulated on the membrane surface, causing membrane fouling and, a decrease in the flux. In Figure 5, the permeate flux versus the concentration factor is presented. Also presented in Figure 5 is a best fit linear regression to the data and the corresponding 95% confidence limits. The r^2 value for the regression fit was 0.96, indicating a good relationship between the CF and permeate flux. Thus, while it may appear from the results in Figure 3 that the decrease in flux at higher CFs would be greater than at lower CFs, the results in Figure 5 demonstrate that the flux-CF relationship is linear over a large CF range.

In Figure 6, permeate turbidity versus time is presented. Permeate turbidity during semibatch operation ranged from 8.6 to 35 NTU and averaged 20 ± 9.3 NTU. The decrease in turbidity after about the fifth hour may be due to the fouling layer acting like a second membrane. Turbidity increased sharply during batchdown, from 15 to 1,213 NTU, because O/G and TSS built up on the membrane surface increasing the amount of material passing through the membrane. The average turbidity during batchdown was 175 ± 267 NTU and for the entire run was 81 ± 182.

In Figure 7, permeate O/G and TSS are presented versus operational time. O/G and TSS followed the same trend as turbidity. Permeate O/G was stable during semibatch operation, ranging from 88 to 109 mg/L, and averaging 98 ± 10 mg/L. During the later stages of batchdown operation the permeate O/G increased from 106 to 1,190 mg/L. The average O/G concentration during batchdown was 289 ± 363 mg/L. The average O/G concentration for the entire run was 220 ± 300 mg/L. During semibatch operation, TSS decreased from about 95 to 24 mg/L. During batchdown, TSS increased in the same manner as O/G, going from about 10 mg/L at the start of batchdown to 250 mg/L at the end. The average TSS during semibatch operation was 53 ± 28 mg/L and during batchdown was 57 ± 74 mg/L. The average permeate TSS concentration for the entire run was 56 ± 63. Initial O/G and TSS concentrations were 17,800 mg/L and 7,700 mg/L, respectively. The overall O/G and TSS removal efficiencies were about 99 percent.

In Figure 8, permeate COD versus operational time is presented. The average COD during the course of the entire run 4,120 ± 725 mg/L. The majority of the COD was soluble thus, its removal by the membrane system was not expected.

Following the treatment run, the CWF was determined to be 118 gal/ft^2-d which was the same as the virgin membrane CWF. The membrane vessel was disassembled and the membrane was visually inspected. A ring of oil had accumulated on the outer one-half of the membrane. Since the oil was not removed during cleaning, a new membrane was installed for the next run.

RUN 2 - POLYMERIC MEMBRANE

Waste Acquisition

Two 55 gallon drums containing 89.5 gallons of coolant was received from a West Virginia industry

System Preparation

Prior to the start of this run, a virgin polymeric (PV-100K) membrane was installed in the SpinTek system. Characteristics of the polymeric membrane were presented previously in Table 1. After installing the polymeric membrane in the centrifugal UF unit, the clean water flux (CWF) was measured as described previously. The average clean water flux was 589 gal/ft^2-d.

System Operation

Following the CWF determination, the system was drained and coolant was pumped from the drums through a 100 mesh sieve into the wastewater storage tank. The final volume of coolant was 89.5 gallons. The coolant was then mixed and samples were taken for O/G, TSS, and pH. O/G and TSS samples were preserved according to Standard Methods. pH was measured immediately.

Following initial sampling, 14.5 gallons of the coolant (V_{system}) were pumped from the storage tank to the feed tank. The system was then started and the temperature was allowed to increase to 110°F using heat/energy from the pump and the rotational device. After reaching 110°F, the temperature was at 110 ± 2°F using a heat exchanger. The operation conditions were as follows: membrane rotational speed = 1750 rpm, vessel pressure = 40-42 psi, return flow rate = 1.1 gal/min, V_{system} = 14.5 gal (for semi-batch operation, feed tank volume decreased with time during batchdown). Permeate flow rate and turbidity, feed tank temperature, and vessel pressure were measured frequently. A calibrated 15 gallon carboy was used to collect the permeate. The hydraulically-based concentration factors were calculated by measuring the volume of permeate produced (V_{perm}) and the Equations 1 and 2.

In Run 2, the system was operated in semi-batch mode until a concentration factor of 6.2X was reached (75 gallons of permeate accumulated, 26.5 hours). During semibatch operation, at 21.5 hours, the system was cleaned as described previously (except a clean water flux was not taken) and then put back into semibatch operation. At the end of semibatch operation, batchdown operation began and was stopped at CF = 15.2X (5.9 gallons of concentrate left in the system, 7.3 hours). At predetermined values of the CF, the permeate was sampled for O/G, TSS, COD and pH.

At the conclusion of batchdown operation, all concentrate was drained from the system and its volume measured. The concentrate was saved and used in residual treatment experiments (to be described in a subsequent report). The system was then cleaned and the clean water flux determined as described earlier. After the CWF determination, the membrane was visually inspected.

Experimental Data and Discussion

In Table 3, a summary of the permeate results is presented. In Figure 9, the permeate flux and concentration factor versus time are presented. In Figure 10, the feed tank temperature versus operational time is presented. During the first 3 hours, the feed tank temperature increased from 74 to 110°F and the flux increased correspondingly. As mentioned previously, the importance of temperature on the permeate flux can be ascertained by comparing the initial portion of the permeate flux and temperature graphs.

Disregarding the initial 3 hours, the flux decreased from 172 gal/ft^2-d, at 3 hours, to 79 at 21.5 hours. At this point, because of high turbidity, the system was shut down and cleaned using the procedure described earlier except a CWF was not determined. After cleaning, the flux increased to 172 gal/ft^2-d then declined to 111 gal/ft^2-d at 26.5 hours (the end of semibatch operation). The average flux for the time period 3 hours to 21.5 was 125 ± 32 gal/ft^2-d and for 21.5 to 26.5 hours was 138 ± 26 gal/ft^2-d. The average flux for all of semibatch operation was 128 ± 31 gal/ft^2-d. During batchdown operation, the flux decreased from 111 to 21 gal/ft^2-d. The average flux during batchdown operation was 61 ± 31 gal/ft^2-d. The average flux for the entire run (except for the first 3 hours) was 104 ± 44 gal/ft^2-d.

In Figure 11, the permeate flux versus the concentration factor, best-fit linear regression, and the 95% confidence limits are presented. The regression line is fitted only to data (circles) generated prior to the cleaning at 4.6X (21.5 hours). The actual permeate flux after cleaning (triangles) is higher than what is predicted by the flux-CF regression line calculated using data prior to cleaning. Thus, for this run, the flux after cleaning cannot be predicted using data generated prior to cleaning.

In Figure 12, permeate turbidity versus time is presented. Permeate turbidity increased during semibatch operation from a low value of 1.4 NTU to 237 NTU at about 21.5 hours. At this point the system was shutdown and cleaned. Following cleaning, the turbidity decreased to 5.4 NTU but increased during the remainder of semibatch operation to 162 NTU (at 26.5 hours). The average turbidity for the time periods 3 hours to 21.5 was 95 ± 85 NTU and for 21 to 26.5 hours was 70 ± 65 NTU. The average turbidity for all of semibatch operation was 90 ± 80 NTU. During batchdown operation, the turbidity increased from 162 to 1509 NTU. The average turbidity during batchdown operation was 597 ± 459 NTU. The average permeate turbidity for the entire run (except for the first 3 hours) was 210 ± 313 NTU.

In Figure 13, permeate O/G and TSS versus operational time are presented. Both O/G and TSS followed the same trend as turbidity - increasing during the first part of semibatch operation, decreasing after cleaning, then increasing through the remainder of semibatch operation and batchdown. O/G increased during semibatch operation from a low value of 81 mg/L to 1538 mg/L at about 21.5 hours. Following cleaning, the O/G decreased to 146 mg/L but increased to 244 mg/L at 26.5 hours. The average O/G concentration for all of semibatch operation was 426 ± 494 mg/L. During batchdown operation, the O/G concentration increased from 244 to 4,390 mg/L. The average O/G concentration during batchdown operation was 1,481 ± 1,684 mg/L. The average permeate O/G concentration for the entire run (except for the first 3 hours) was 832 ± 1,172 mg/L. TSS increased during semibatch operation from a low value of 1 mg/L to 329 mg/L at about 21.5 hours. Following cleaning, the TSS decreased to about 24 mg/L at 26.5 hours. The average TSS concentration for all of semibatch operation was 82 ± 107 mg/L. During batchdown operation, the TSS concentration increased from 31 to 243 mg/L. The average TSS concentration during batchdown operation was 161 ± 107 mg/L. The average permeate TSS concentration for the entire run (except for the first 3 hours) was 120 ± 111 mg/L. Initial O/G and TSS concentrations were 15,900 mg/L and 11,520 mg/L, respectively. The overall O/G and TSS removal efficiencies were about 95 and percent, respectively.

In Figure 14, permeate COD versus operational time is presented. The average COD for the entire run (except for the first 3 hours) was 6250 ± 3492. COD increased during the course of the run and followed the same trend as turbidity, O/G, and TSS. During periods of low O/G and TSS, the majority of the COD was associated with the aqueous phase. When O/G and TSS were high, the majority of the COD was filterable. The membrane system was not expected to remove soluble COD.

After the second cleaning of Run 2 (after batchdown), the CWF was 172 gal/ft^2-d which was much lower than the virgin CWF (589 gal/ft^2-d). The membrane vessel was disassembled and the membrane visually inspected. A ring of oil had accumulated on the outer one-half of the membrane. The oil was not removed during cleaning and a new membrane was installed for the third run.

RUN 3 - POLYMERIC MEMBRANE

Waste Acquisition

Two 55 gallon drums, each containing 50 gallons of coolant were received from a West Virginia industry. One drum was used for this run, and the second drum was used for Run 1 - Ceramic Membrane.

System Preparation

Prior to the start of this run, a virgin PV-100K membrane was installed in the SpinTek system. After installing the polymeric membrane in the centrifugal UF unit, the clean water flux (CWF) was measured to be 718 gal/ft^2-d, using the method described previously.

System Operation

Following the CWF determination, the system was drained and coolant was pumped from the drum through a 100 mesh sieve into the wastewater storage tank (see Figure 1). The final volume of coolant was 50 gallons. The coolant was then mixed and samples were taken for O/G (duplicate), TSS (triplicate), and pH. O/G and TSS samples were preserved and analyzed according to Standard Methods. pH was measured immediately.

Following initial sampling, 10 gallons of the coolant (V_{system}) were pumped from the storage tank to the feed tank. The system was then started and the temperature was allowed to increase to 110°F using heat/energy from the pump and the rotational device. After reaching 110°F, the temperature was maintained at 110 ± 2°F using a heat exchanger. The operation conditions were as follows: membrane rotational speed = 1750 rpm, vessel pressure = 41 - 43 psi, return flow rate = 1.1 gal/min, V_{system} = 10 gal (for semi-batch operation, feed tank volume decreased with time during batchdown). Permeate flow rate and turbidity, feed tank temperature, and vessel pressure were measured frequently. A calibrated 15 gallon carboy was used to collect the permeate. The hydraulically-based concentration factor was calculated by measuring the volume of permeate produced and the Equations 1 and 2.

In this run, the system was operated in semi-batch mode until a concentration factor of 5.0X was reached (40 gallons of permeate accumulated, 23.75 hours). At this point the system was shutdown and cleaned as described earlier except no clean water flux was taken. Following cleaning, the concentrate from semibatch operation was placed back into the feed tank and batchdown operation began. Batchdown stopped at CF = 10X (5 gallons of concentrate left in the system, 4.9 hours). Permeate collected between changes in CF (*i.e.*, 1X to 2X, 2X to 3X, *etc.*) was collected in the 15 gallon carboy. At each CF, the composite permeate sample was sampled for O/G, TSS, and pH. At predetermined values of the CF, the concentrate was sampled for TSS.

At the conclusion of batchdown operation, all concentrate was drained from the system and its volume was measured. The concentrate was saved and used in residual treatment experiments (to be described in a subsequent report). The system was cleaned and the CWF determined as described earlier. After the CWF determination, the membrane was removed from the system and visually inspected.

Experimental Data and Discussion

In Table 4, a summary of the permeate results is presented. In Figure 15, the permeate flux and concentration factor versus time are presented. In Figure 16, the feed tank temperature versus operational time is presented. During the first 0.9 hours, the feed tank temperature increased from 99 to 110°F after which it was maintained at 110 ± 2°F. During the first 5 hours of operation, the permeate flux decreased from 102 to 83 gal/ft^2-d and then stabilized at about 80 gal/ft^2-d for the next 10 hours (5 to 15 hours). After hour 15 the flux decreased from 78 /ft^2-d to 60 gal/ft^2-d at the end of semibatch operation. The average flux during semibatch operation (not including the first 0.88 hours) was 78 ± 8.5 gal/ft^2-d. Cleaning the system at the end of semibatch operation did not appear to improved permeate production. The permeate flux decreased from 60 to 17 gal/ft^2-d during batchdown and averaged 45 ± 14.4 gal/ft^2-d. The average flux for the entire run (except for the first 0.88 hours) was 69 ± 17.5 gal/ft^2-d.

In Figure 17, the permeate flux versus the concentration factor, best-fit linear regression to the data, and the 95% confidence limits are presented. The r^2 value for the regression fit was 0.98. Unlike polymeric membrane - Run 2, cleaning the system did not alter the flux-CF relationship. The data points after cleaning (5.6X to 10X) followed the same trend as the points between 1X and 5X. The system may not have been cleaned effectively, but this is unlikely given the rigorous cleaning procedure (hot water flush, washing solution, hot water flush).

In Figure 18, permeate turbidity versus time is presented. Permeate turbidity during semibatch operation ranged from 0.6 to 24 NTU and averaged 4.3 ± 6.1 NTU. The system was shutdown and cleaned at the end of semibatch operation because turbidity was rapidly increasing. After cleaning, the turbidity decreased to 7 NTU, then increased to 1380 NTU over the course of batchdown operation. The average turbidity during batchdown operation was 595 ± 518 NTU.

In Figure 19, permeate O/G and TSS and concentrate TSS versus operational time are presented. Permeate O/G was stable during semibatch operation, ranging from 76 to 114 mg/L and averaging 89 ± 11 mg/L. During the later stages of batchdown operation the permeate O/G increased from 113 to 9,660 mg/L. The average O/G concentration during batchdown was 2,310 ± 4,123 mg/L. The average O/G concentration for the entire run was 945 ± 2,632 mg/L. Permeate TSS followed the same trend as permeate O/G for both semibatch and batchdown operation. During semibatch the permeate TSS ranged from < 1 to 11 mg/L and averaged 3.4 ± 3.4. During batchdown, TSS increased from 10 mg/L at the start of batchdown to 401 mg/L at the end. The average TSS during batchdown was 147 ± 151 mg/L and for the entire run was 59

± 114. Initial O/G and TSS concentrations were 53,430 mg/L and 11,940 mg/L, respectively. O/G and TSS removal efficiencies were 98 and 99 percent, respectively.

The concentrate TSS (Figure 19) did not adequately represent the behavior of the system with regards to the CF. The concentrate TSS should have increased with operational time in the same manner as the hydraulic-based CF. For example, the initial coolant had a TSS concentration of 11,940. The concentrate TSS at the end of semibatch operation (5X) and batchdown (10X) should have been on the order of 60,000 and 120,000 mg/L, respectively. The most probable cause for this discrepancy is the loss of solids, mostly oil, during the TSS test. In the TSS test, the filter samples are dried at 103°C until the filter paper has reached a steady weight. In a separate experiment, the filter weight was monitored versus drying time. The filter continually lost weight, resulting in the concentrate TSS being underestimated. In addition, the initial TSS was taken on a sample that was at room temperature while the concentrate TSS was taken at 110°F. The higher temperature may have allowed more oil to pass through the GF/C filter (0.8 μm). Since the majority of the TSS is associated with O/G, a lower than expected concentrate TSS would be observed. Thus, an accurate concentrate TSS cannot be obtained and the CF must be based on amount of permeate produced or a substance that does not evaporate/volatilize at 103°C.

Following the treatment run, the CWF was determined to be 115 gal/ft^2-d which was substantially lower than the virgin membrane CWF (718 gal/ft^2-d). The membrane vessel was disassembled and the membrane visually inspected. A ring of oil and accumulated on the outer one-half of the membrane and this oil was not removed during cleaning. If another polymeric run was to be attempted, a new membrane would have to be installed.

RUN 1 - CERAMIC MEMBRANE

Waste Acquisition.

Two 55 gallon drums, each containing 50 gallons of coolant were received from a West Virginia industry. The first drum was used for Run 3 - polymeric membrane and the second drum was used for this run.

System Preparation

After Run 3 of the polymeric membrane experiments, a ceramic membrane was obtained from SpinTek. Characteristics of the ceramic membrane are presented in Table 5. After installing the ceramic membrane in the centrifugal UF unit, the clean water flux (CWF) was measured to be 320 gal/ft^2-d, using the method described previously.

Following the CWF determination, the system was drained and coolant was pumped from the drum through a 100 mesh sieve into the wastewater storage tank (see Figure 1). The coolant in the storage tank was kept under quiescent conditions for 1 hour. Based on visual observations, an invert emulsion did not form but a thin oily layer on the surface was present. This layer was skimmed from the surface and the final volume of coolant was 50 gallons. The coolant was then mixed and samples were taken for O/G (duplicate), TSS (triplicate), and pH. O/G and TSS were preserved and analyzed according to Standard Methods. The pH was measured immediately.

Following initial sampling, 10 gallons of the coolant (V_{system}) were pumped from the storage tank to the feed tank and the contents of the feed tank were heated to about 100°F using the heat exchanger.. The system was then started and the temperature was allowed to increase to 110°F using energy from the pump and the rotational device. After reaching 110°F, the temperature was at 110 ± 2°F using the heat exchanger. The operation conditions were as follows: membrane rotational speed = 1750 rpm, vessel pressure = 41-43 psi, return flow rate = 1.1 gal/min, V_{system} = 10 gal (for semi-batch operation, feed tank volume decreased with time during batchdown). Permeate flow rate and turbidity, feed tank temperature, and vessel pressure were measured frequently. A calibrated 15 gallon carboy was used to collect the permeate. The hydraulically-based concentration factor was calculated by measuring the volume of permeate produced and the Equations 1 and 2.

In this run, the system was operated in the semibatch mode until a concentration factor of 5X was reached (40 gallons of permeate accumulated, 17 hours). At this point batchdown began and was stopped at CF = 9.1X (5.5 gallons of concentrate left in the system, 4.5 hours). Permeate collected between changes in CF (*i.e.*, 1X to 2X, 2X to 3X, *etc.*) was collected in the 15 gallon carboy and the composite sampled for O/G, TSS, and pH. At predetermined values of the CF, the concentrate was sampled for TSS.

At the conclusion of batchdown operation, all concentrate was drained from the system and its volume measured. The concentrate was saved and used in residual treatment experiments (to be described in a subsequent report). The system was cleaned and the CWF determined as described earlier.

Experimental Data and Discussion

In Table 6, a summary of the permeate results is presented. In Figure 20, the permeate flux and concentration factor versus time are presented. The permeate flux increased during the first 15 minutes to a maximum value of 128 gal/ft^2-d and then decreased gradually for the remainder of the semibatch portion of the run. At the start of batchdown the flux was 70 gal/ft^2-d and decreased to about 20 gal/ft^2-d at the end of the run. The flux decreased more rapidly during batchdown operation because the concentration in the feed tank increased sharply and O/G and TSS built up on the membrane surface causing the membrane to become increasingly fouled. In Figure 21, the permeate flux versus the concentration factor is presented. Also presented in Figure 21 is a best-fit linear regression to the data and the 95% confidence limits. The r^2 value for the regression fit was 0.99. Based on the results in Figure 21, the flux-CF relationship is linear over a large CF range.

In Figure 22, the feed tank temperature versus operational time is presented. For the majority of the run, the feed tank temperature was maintained between 108 and 112°F using a heat exchanger. Before operation began, the feed tank temperature was increased to about 100°F using the heat exchanger. During the first 15 minutes of operation the temperature increased to 109.6°F. During the last 1/2 hour of batchdown operation, the temperature was difficult to control because of the low level of concentrate in the feed tank (*i.e.*, a large portion of the heat exchanger coils were not submerged in the concentrate). The temperature increased to a maximum of 120°F toward the end of the run. Unlike the polymeric membrane, high temperatures will not degrade the ceramic membrane, although temperature will effect the permeate flux and quality. The higher temperatures at the end of batchdown may have caused the flux to be higher than expected but this was not readily apparent from the data presented in Figures 20 and 21.

In Figure 23, permeate turbidity versus time is presented. Turbidity remained relatively constant during semi-batch mode, averaging about 8 NTU, then increased sharply during batchdown. Reasons for the increase in turbidity include: 1) as the CF increased O/G and TSS built up on the membrane surface increasing the amount of material passing through the membrane and 2) the increased temperature allowed more oil and solids to "squeeze" through the membrane's pores.

In Figure 24, permeate TSS and O/G and concentrate TSS are presented versus operational time. Permeate O/G and TSS were stable during semibatch operation and increased during batchdown. Reasons cited previously for the increase in turbidity during batchdown operation also apply to the increase in permeate O/G and TSS. As expected, the concentrate TSS increased with time. The initial TSS coolant concentration was about 2,350 mg/L and was about 18,500 mg/L at the end of the run. This corresponded to a CF (based on solids) of about 7. As mentioned previously, the hydraulically based CF is more reliable than the TSS based CF because in the TSS analysis there can be a considerable loss of solids during the TSS experiment. As expected, permeate production and quality were much better during semibatch operation. Initial coolant O/G and TSS coolant concentrations were 31,310 mg/L and 2,350 mg/L, respectively. O/G and TSS removal efficiencies for the entire run were greater than 99 percent.

RUN 2 - CERAMIC MEMBRANE

Waste Acquisition

One 55 gallon drum containing 50 gallons of coolant was received from a West Virginia industry.

System Preparation

Prior to the start of this run, several runs involving activated sludge and kaolinite were conducted with the ceramic membrane from Run 1. After each of these experiments, the membrane was cleaned using the method described earlier. Prior to the start of this run, the CWF was measured to be 513 gal/ft^2-d.

System Operation

Following the CWF determination, the system was drained and coolant was pumped from the drum through a 100 mesh sieve into the wastewater storage tank (see Figure 1). Biocide was added (100 mL/50 gallons coolant) to prevent excessive biological activity. The coolant was kept under quiescent conditions overnight. Based on visual observation, an invert emulsion did not formed but an oily layer was present on the surface. This layer was skimmed from the surface and the final volume of coolant was 50 gallons. The coolant was then mixed and samples were taken for O/G (duplicate), TSS (triplicate), and pH. O/G and TSS samples were preserved and analyzed according to Standard Methods. pH was measured immediately.

Following initial sampling, 12 gallons of the coolant (V_{system}) were pumped from the storage tank to the feed tank. The system was then started and the temperature was allowed to increase to

110°F using heat/energy from the pump and the rotational device. After reaching 110°F, the temperature was maintained at 110 ± 2°F using a heat exchanger. The operational conditions were as follows: membrane rotational speed = 1750 rpm, vessel pressure = 41.5 - 42.5 psi, return flow rate = 0.8 - 1.1 gal/min, V_{system} = 12 gal (for semi-batch operation, feed tank volume decreased with time during batchdown). Permeate flow rate and turbidity, feed tank and permeate temperature, vessel pressure, and return flow rate were measured frequently. A calibrated 15 gallon carboy was used to collect the permeate. The hydraulically-based concentration factor was calculated by measuring the volume of permeate produced and the Equations 1 and 2.

In this run, the system was operated in the recycle mode (permeate routed back to the feed tank) for long periods of time during semibatch and batchdown operation. During semibatch operation, the concentration factor was increased to 4X in 1X increments. A total of 36 gallons of permeate were accumulated over 191 hours. During each CF increment, the system was run in recycle mode for 48 hours. The performance of the system could be determined as both as a function of concentration factor and operating time. During batchdown, the CF was increased from 4X to 6X (8 gallons of concentrate left in the system, 49 hours total). At CFs of 5X and 6X, the system was operated in recycle mode for 24 hours.

During recycle operation, TSS of the permeate was sampled periodically. Between changes in CF (*i.e.,* 1X to 2X, 2X to 3X, *etc.*), a composite sample of the permeate was collected from the 15 gallon carboy and sampled for O/G and TSS. Concentrate TSS was measured periodically during the recycle operation as well as during changes in CF. At the conclusion of batchdown, all concentrate was drained from the system and its volume was measured. Concentrate samples of TSS (duplicate) and O/G were taken. The remaining concentrate was saved and used in residual treatment experiments (to be described in a subsequent report). All samples were analyzed by WVU-CEE.

Following treatment, the system was flushed for 1/2 hour with 136°F tap water. The system was cleaned for about 2 hours using the washing solution that was described earlier. During washing, the flux decreased to zero. The system was emptied and flushed using 134°F tap water. However, the flux was not reestablished. The membrane vessel was opened and the membrane was visually inspected. A white substance was caked on the inner portion of the membrane. The "cake" was wiped off, a sample taken, and then the membrane was sponged with a ≈ 1% sulfuric acid solution. The membrane was rinsed with cold tap water then put back in the membrane casing. The clean water flux was determined to be 268 gal/ft^2-d. The system was re-washed using a mixture of 30 mL of *Dawn* dishwashing liquid and 5 gallons of 135°F tap water for 1 1/2 hours, flushed with 129°F tap water for 10 minutes, and then the CWF determined. The CWF was 431 gal/ft^2-d.

Experimental Data and Discussion

In Table 7, a summary of the permeate results is presented. In Figure 25, the permeate flux and concentration factor versus time are presented. In Figure 26, the feed tank temperature versus operational time is presented. During the first 0.3 hours, the feed tank temperature increased from 95 to 110°F and the flux increased correspondingly.

During the first 4.4 hours, the permeate flux increased from 111 to 158 gal/ft^2-d after which it increased slightly during the remainder of the 1X recycle operation. The average flux during each recycle (not including the first 0.3 hours or the data from the transition to each new CF) is presented in Table 7. The flux for the entire run ranged from 92 gal/ft^2-d to 174 gal/ft^2-d averaged 135 ± 23 gal/ft^2-d.

During each transition to a new CF (semi-batch mode from 1X to 4X and batchdown mode for 4X to 6X), the flux decreased but then recovered during the subsequent recycle operation. The decrease in flux during CF transition (*i.e.,* semibatch operation) was due to the increase in O/G and TSS concentrations in the feed tank. However, it is unclear why the flux during recycle at a constant CF increased. In Figure 27, the permeate flux versus the concentration factor, best-fit linear regression line and the 95% confidence limits are presented. The r^2 value for the regression fit was 0.98. Based on the results in Figure 27, the flux-CF relationship is linear over a large CF range.

In Figure 28, permeate turbidity versus time is presented. The permeate turbidity decreased from 9 NTU at 0.3 hours to 2.9 NTU at 1.3 hours, then remained relatively constant during the entire run. The permeate turbidity for the entire run ranged from 0.65 NTU to 9.0 NTU and averaged 1.2 ± 0.9 NTU. There was not a noticeable difference in permeate turbidity during semibatch operation and batchdown.

In Figure 29, permeate O/G and TSS and concentrate TSS are presented versus operational time. There was not a noticeable difference in permeate O/G and TSS during semibatch operation and batchdown. Permeate O/G was stable during the entire run, ranging from 32 to 42 mg/L, and averaging 37.4 ± 5.0 mg/L. Likewise, the permeate TSS remained stable during the entire run, ranging from <1 to 32 mg/L. The average permeate TSS during the entire run was 8.8 ± 8.3 mg/L. The concentrate TSS at the beginning and end of the run were 2,670 and 16,110 mg/L, respectively resulting in a TSS-based CF of 6.03. The hydraulic and TSS based CFs were in good agreement because care was taken to minimize the TSS drying time. The initial O/G concentration was 48,830 mg/L. O/G and TSS removal efficiencies were greater than 99 percent.

Following the treatment run, the system was flushed using 134°F tap water and then washed using the *Biz/Dawn* washing solution. As in Run 1 - ceramic membrane, the flux during washing approached zero. The membrane was visually inspected and the same white substance observed in Run 1 -ceramic membrane was present. The membrane was sponged cleaned using a 1% sulfuric acid solution, rinsed with tap water, placed back in the system, and the CWF was determined. The CWF was 268 gal/ft^2-d. The system was re-washed using 30 mL *Dawn* dishwashing liquid in 5 gallons of 135°F tap water for 1 1/2 hours, and then flushed with 129°F tap water for 10 minutes. The CWF after the second washing was 431 gal/ft^2-d. The inclusion of *Biz* detergent was discontinued after this run and a new washing solution (30 mL *Dawn*, pH = 12) was developed based on discussions with SpinTek.

RUN 3 - CERAMIC MEMBRANE

Waste Acquisition

One 55 gallon drum containing 40 gallons of coolant was received from a West Virginia industry.

System Preparation

Prior to the start of this run, a biosorbent run took place, after which the system was cleaned using 30 mL *Dawn* dishwashing liquid and 5 gallons of hot tap water. The ceramic membrane installed for Run 1 - Ceramic Membrane was used. After cleaning, the CWF was determined to be 431 gal/ft^2-d using the method described earlier.

System Operation

Following the CWF determination, the system was drained and coolant was pumped from the drum through a 100 mesh sieve into the wastewater storage tank (see Figure 1). Biocide was added (100 mL/50 gal) to prevent excessive biological activity. The coolant was mixed and samples were taken for O/G (triplicate), TSS (six samples), and pH. O/G and TSS samples were preserved according to Standard Methods and analyzed by WVU-CEE. pH was measured immediately.

Following initial sampling, 10 gallons of the coolant (V_{system}) were pumped from the storage tank to the feed tank. The system was then started and the temperature was allowed to increase to 110°F using heat/energy from the pump and the rotational device. After reaching 110°F, the temperature was maintained at 110 ± 2°F using a heat exchanger. General operational conditions were as follows: membrane rotational speed = 1750 rpm, vessel pressure = 42 psi, return flow rate = 1.1-1.2 gal/min, V_{system} = 10 gal (for semi-batch operation, feed tank volume decreased with time during batchdown). During the course of the experiment, temperature and rotational excursions were conducted. The system was run in the recycle mode at CF = 4X at a feed tank temperature of 140°F. Following the temperature excursion, the system's rotational speed was varied between 1750 and 1000 rpm in the recycle mode at CF=4X and a feed tank temperature of 110°F. Permeate flow rate and turbidity, feed tank and permeate temperature, vessel pressure, and return flow rate were measured frequently. A calibrated 15 gallon carboy was used to collect the permeate. The hydraulically-based concentration factor was calculated by measuring the volume of permeate produced and the Equations 1 and 2.

In this run, the system was operated in semi-batch mode until a concentration factor of 4X was reached (30 gallons of permeate accumulated, 218 hours). The coolant was concentrated in 1X increments from 1X to 4X. During each CF increment (except 4X), the system was run in recycle mode for 48 hours. The 4X increment lasted for 70 hours. During the middle portion of the 4X recycle operation, the feed tank temperature and rotational speed were varied. Between 169 and 181 hours (12 hours total) the feed tank temperature was maintained at 140°F (rotational speed = 1750 rpm). Between 204 and 217 hours, the rotational speed was varied from 1750 to 1000 rpm, from 1000 to 1500 rpm, from 1500 to 1250 rpm, and then back to 1750 rpm for the remainder of the run. For each rotational speed, the feed tank temperature was 110°F and the system was operated in recycle mode for at least 4 hours or until the flux stabilized. During batchdown, the CF was increased from 4X to 6X (6.7 gallons of concentrate left in the system, 64 hours total). During the 5X and 6X increments, the system was run in the recycle mode for 24 hours and 37 hours, respectively. At predetermined times, composite samples of the permeate were taken for O/G and TSS. Concentrate samples were taken for TSS at predetermined times. All samples were analyzed by WVU-CEE.

At the conclusion of batchdown operation, all concentrate was drained from the system and its volume measured. Concentrate samples of TSS (duplicate) and O/G were taken. The remaining concentrate was saved and used in residual treatment experiments (to be described in a subsequent report). The system was flushed with hot tap water. A new washing solution was developed based on discussions with SpinTek. Thirty mL of *Dawn* dishwashing liquid was added to 5 gallons of distilled water and the pH was adjusted to 12 using with 38 mL of 10.5 M NaOH. The washing solution was heated to 136°F and the system was washed. Following washing, the system was flushed and CWF determined as described previously.

Experimental Data and Discussion

In Table 8, a summary of the permeate results is presented. In Figure 30, the permeate flux and concentration factor versus time are presented. In Figure 31, the feed tank temperature versus operational time is presented. The average flux during each CF-recycle period is presented in Table 8. The flux during the semibatch operation and temperature and rotational excursions (during CF = 4X) were not included in the average flux calculation. As in the Run 2- ceramic membrane run, the flux decreased during CF transition but did not recover in the subsequent recycle operation. The flux for semibatch operation (not including data after 166 hours) ranged from 204 to 263 gal/ft^2-d, and averaged 235 ± 13 gal/ft^2-d. During batchdown, the flux ranged from 149 to 179 gal/ft^2-d and averaged 165 ± 8 gal/ft^2-d. As will be discussed later, the flux did not recover after the rotational excursions thus, the fluxes during batchdown were lower than what would be expected if the system was continually operated at 1750 rpm. The average flux for the entire run was not included in Table 8 for this reason.

In Figure 32, the permeate flux versus operational time during temperature and rotational excursions are presented. When the temperature was increased from 110 to 140°F the average flux increased by about 45 percent (220 to 319 gal/ft^2-d). The viscosity of water decreases by about 24 percent over this temperature range thus; only a portion of the increase in flux can be attributed to the decrease in viscosity with increasing temperature. After the temperature was decreased to 110°F the flux returned to its pre-temperature excursion value.

Average fluxes during rotational excursions are presented in Table 9. When the rotational speed was decreased from 1750 to 1000 rpm the flux decreased from 230 to 140 gal/ft^2-d. When the rotational speed was increased, the flux did not fully recover, even after the rotational speed was increased to its maximum value of 1750 rpm. It is hypothesized that when the rotational speed was initially decreased, the membrane became fouled and subsequent increases in the rotational speed were not able to clean the membrane effectively. Thus, in a full-scale system, the maximum rotational speed should be used.

In Figure 33, the permeate flux versus the concentration factor, best-fit linear regression and the 95% confidence limits are presented. The data taken prior to the temperature and rotational excursions (circles represented by o) were used in the linear regression analysis ($r^2 = 0.99$). The triangles represent data taken at 5X and 6X (after the excursions). The actual permeate fluxes at 5X and 6x were lower than the predicted because the membrane flux did not recover following the rotational speed excursions. As mentioned previously, during full-scale operation, the rotational speed should not be reduced from its maximum value.

In Figure 34, permeate turbidity versus time is presented. The permeate turbidity remained relatively constant, ranging from 0.13 NTU to 0.67 NTU and averaging 0.23 ± 0.09 NTU. Turbidity was not affected by temperature or rotational speed.

In Figure 35, permeate O/G and TSS and concentrate TSS are presented versus operational time. Permeate O/G increased over the course of the run from <1 to 35 mg/L (average 20 ± 14.5 mg/L). The permeate TSS remained stable during the entire run, ranging from <1 to 4 mg/L (average: 0.8 ± 1.3 mg/L). Permeate O/G and TSS were not affected by temperature or rotational speed. The initial O/G and TSS concentrations were 19,840 mg/L and 2,210 mg/L, respectively. The concentrate TSS ranged from 505 mg/L to 1450 mg/L during the course of the run and did not agree with the hydraulically-based CF for the reasons discussed earlier. O/G and TSS removal efficiencies were greater than 99 percent.

Following the treatment run, the system was cleaned as described earlier (flushed, washed with the *Dawn*, pH=12 solution, flushed). The CWF was 481 gal/ft^2-d. After cleaning, the membrane was visually inspected and no whitish "cake" was observed. In subsequent runs, the *Dawn*, pH=12 washing solution will be used.

Table 1
Characteristics of PV-100K Polymeric Membrane

Characteristic	Value
Manufacturer	Advanced Membrane Technology
Material	Polyvinyliden-fluoride
Area	0.53 ft^2
MWCO[1]	100,000
Max. Operating Pressure,	150 psi
Max. Operating Temperature	120°F

[1] Molecular weight cutoff.

Table 2
Summary of Permeate Results for PV-100K Membrane - Run 1

Parameter	Range		Average ± std[1]			% Removal
	High	Low	Entire Run	Semibatch	Batchdown	
Flux, gal/ft^2-d[1]	90	26	64 ± 14	73 ± 4.8	50 ± 11.7	---
Turbidity, NTU	1213	8.6	81 ± 182	20 ± 9.3	175 ± 267	---
O/G, mg/L	1191	88	221 ± 300	98 ± 9.7	289 ± 363	98.8
TSS, mg/L	250	11	56 ± 63	53 ± 28	57 ± 74	99.3
pH	7.33	7.02	7.13 ± 0.09	7.13 ± 0.13	7.13 ± 0.06	---

[1] At 110±2°F

Table 3
Summary of Permeate Results for PV-100K Membrane - Run 2

Parameter	Range		Average ± std[1]			% Removal
	High	Low	Entire Run	Semibatch	Batchdown	
Flux, gal/ft^2-d[1]	178	21	104 ± 44	125 ± 32[2] 138 ± 26[3] 128 ± 31[4]	61 ± 31	---
Turbidity, NTU	1509	1.4	210 ± 313	95 ± 85[2] 70 ± 65[3] 90 ± 80[4]	597 ± 459	---
O/G, mg/L	4390	81	832 ± 1172	426 ± 494[4]	1481 ± 1684	94.8
TSS, mg/L	329	< 1	120 ± 111	82 ± 107[4]	161 ± 107	99.0
pH	7.63	7.27	7.40 ± 0.09	7.39 ± 0.11[4]	7.41 ± 0.06	---

[1] At 110±2°F. For 3 to 21.5 hours. [2] For 3 to 21.5 hours. [3] For 21.5 to 26.5 hours. [4] For the entire semibatch operation.

Table 4
Summary of Permeate Results for PV-100K Membrane - Run 3

Parameter	Range		Average ± std[1]			% Removal
	High	Low	Entire Run	Semibatch	Batchdown	
Flux, gal/ft^2-d[1]	102	17	69 ± 17.5	78 ± 8.5	45 ± 14.4	---
Turbidity, NTU	1380	0.6	147 ± 355	4.3 ± 6.1	595 ± 518	---
O/G, mg/L	9,660	76	945 ± 2,632	89 ± 11	2,310 ± 4,123	98.2
TSS, mg/L	401	< 1	59 ± 114	3.4 ± 3.4	147 ± 151	99.5
pH	7.84	7.39	7.63 ± 0.08	7.65 ± 0.07	7.57 ± 0.08	---

[1] At 110±2°F

Table 5
Characteristics of Ceramic Membrane

Characteristic	**Value**
Manufacture	TiO_2 - Al_2O_3
Material	TRUMEN
Area	0.53 ft^2
Mean Pore Size	0.11 μm
Max. Pore Size	0.7 μm
Maximum Temperature	>160°F

Table 6
Summary of Permeate Results for Ceramic Membrane - Run 1

Parameter	Range		Average ± std[1]			% Removal
	High	Low	Entire Run	Semibatch	Batchdown	
Flux, gal/ft^2-d[1]	20	128	87.3 ± 29.7	98.9 ± 17.8	60.8 ± 17.0	--
Turbidity, NTU	2.1	760	80.6 ± 181	8.0 ± 6.1	355 ± 255	--
O/G, mg/L	106	464	187 ± 107	121 ± 13	252 ± 121	99.4
TSS, mg/L	< 1	49	12 ± 15	3 ± 2.5	23 ± 17	99.5
pH	7.00	7.67	7.56 ± 0.17	7.54 ± 0.19	7.61 ± 0.03	--

[1] At 110±2°F

Table 7
Summary of Permeate Results for Ceramic Membrane - Run 2

Parameter	Range		Average ± std			% Removal
	High	Low	Entire Run	Semibatch[1]	Batchdown[1]	
Flux, gal/ft^2-d[2]	174	92	135 ± 23	142 ± 19	103 ± 9.0	---
Turbidity, NTU	9.0	0.65	1.21 ± 0.9	1.25 ± 0.96	0.99 ± 0.38	---
O/G, mg/L	42	32	37 ± 5.0	40 ± 3.4	32 ± 0	99.9
TSS, mg/L	< 1	32	8.8 ± 8.3	7.7 ± 6.9	15 ± 11	99.7

[1] Average taken from start of recycle mode to end of recycle mode for each CF. [2] At 110±2°F.

Table 8
Summary of Permeate Results for Ceramic Membrane - Run 3

Parameter	Range		Average ± std[1]			% Removal
	High	Low	Entire Run	Semibatch	Batchdown	
Flux, gal/ft^2-d[2]	263	149	NA[3]	235 ± 13	165 ± 8	---
Turbidity, NTU	0.13	0.67	0.23 ± 0.09	0.19 ± 0.04	0.27 ± 0.07	---
O/G, mg/L	0	35	20 ± 15	11 ± 10	33 ± 2.6	99.9
TSS, mg/L	< 1	4	< 1	1.2 ± 1.4	< 1	99.7

[1] Average taken from start of recycle mode to end of recycle mode for each CF. Fluxes after temperature and rotational excursions were not included. [2] At 110±2°F. [3] Not Applicable.

Table 9
Average Permeate Flux During Rotational Excursions

Time Period, hr	Rotational Speed, rpm	Average Flux, gal/ft^2-d
184 → 204	1750	230 ± 5.5
204 → 209	1000	140 ± 5.1
209 → 213	1500	157 ± 6.4
213 → 217	1250	166 ± 3.3
217 →218	1750	155 ± 5.0

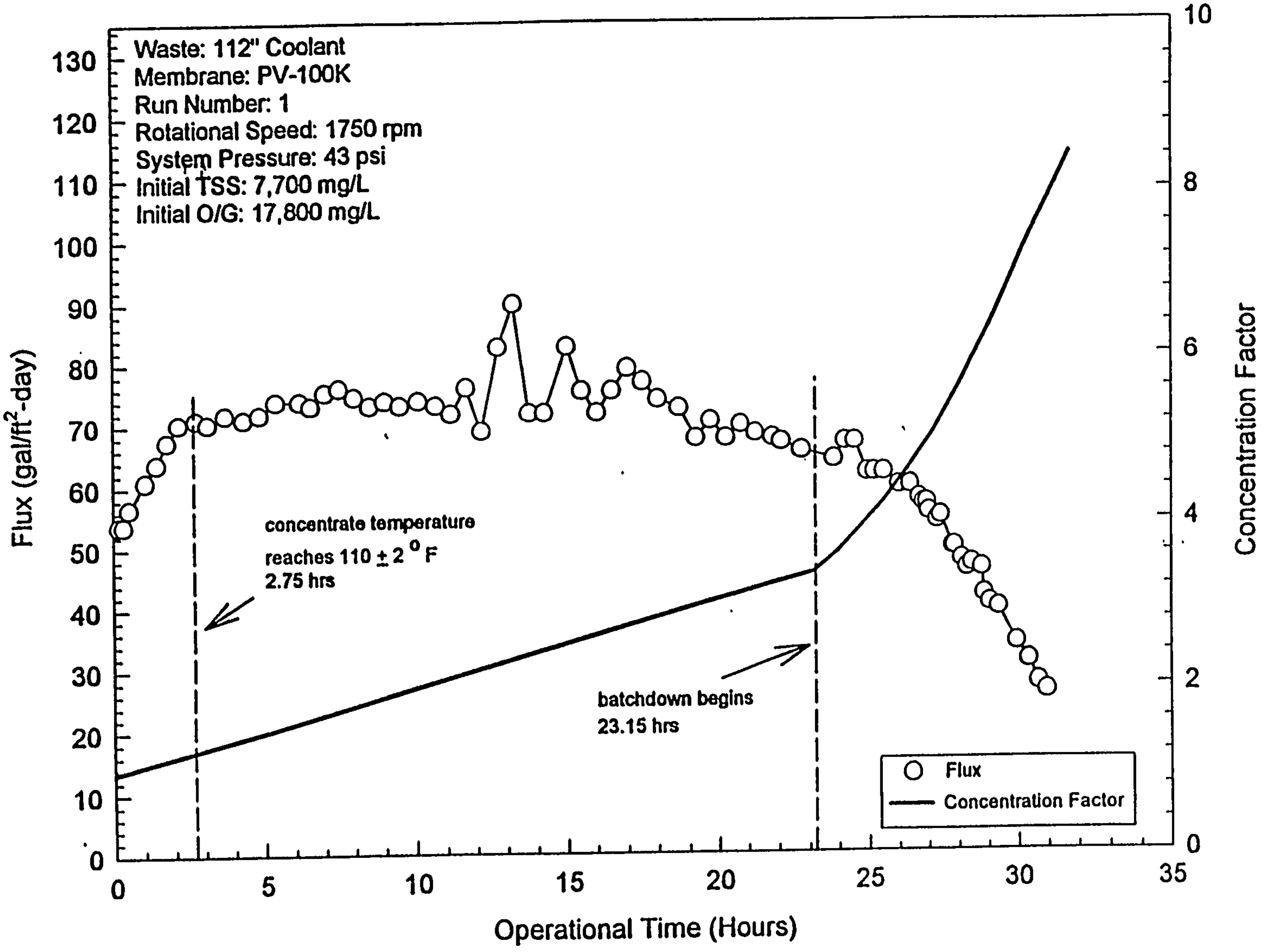

Figure 3. Permeate Flux and Concentration Factor Versus Time for Run 1-Polymeric Membrane.

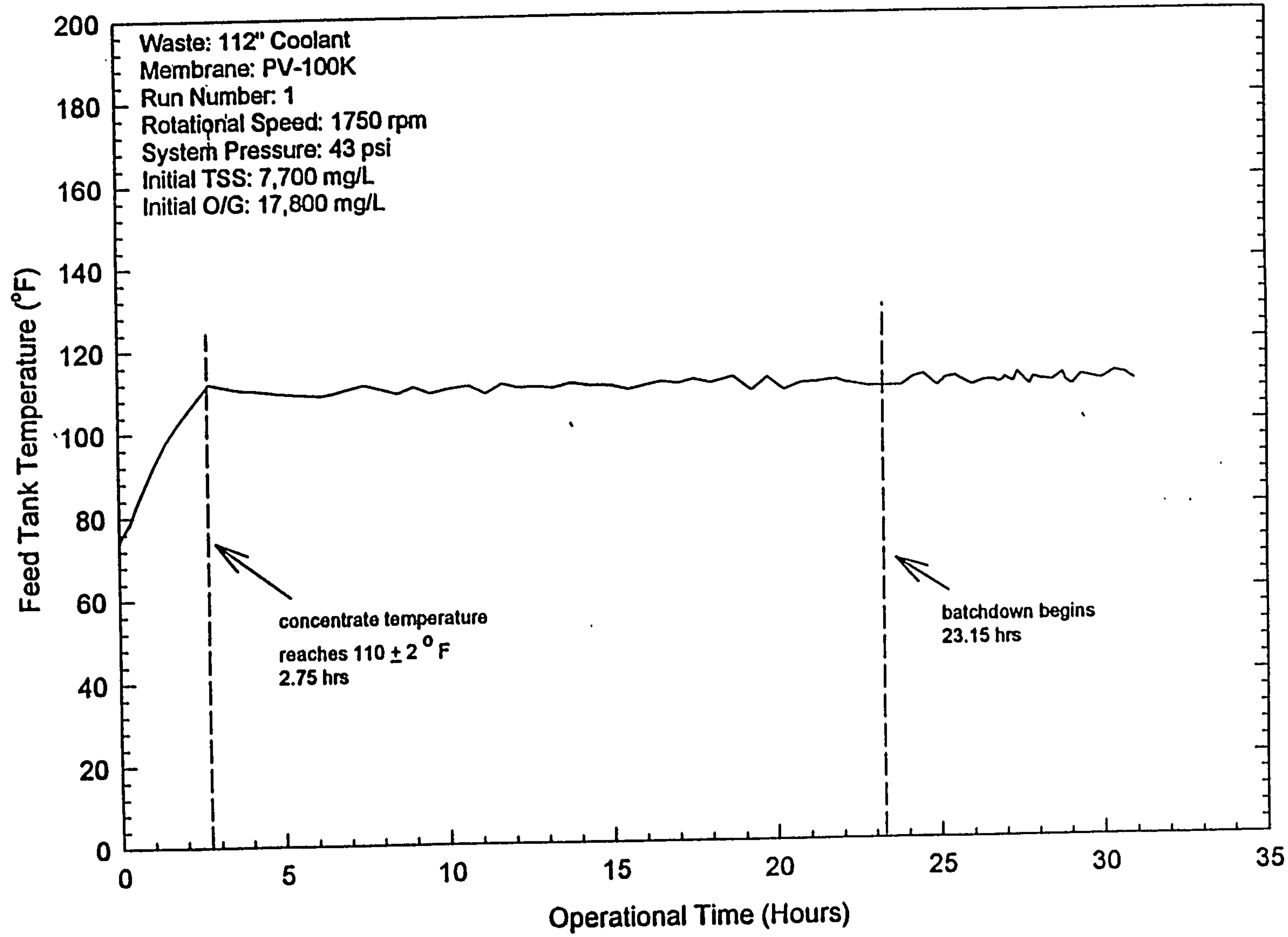

Figure 4. Feed Tank Temperature Versus Time for Run 1-Polymeric Membrane

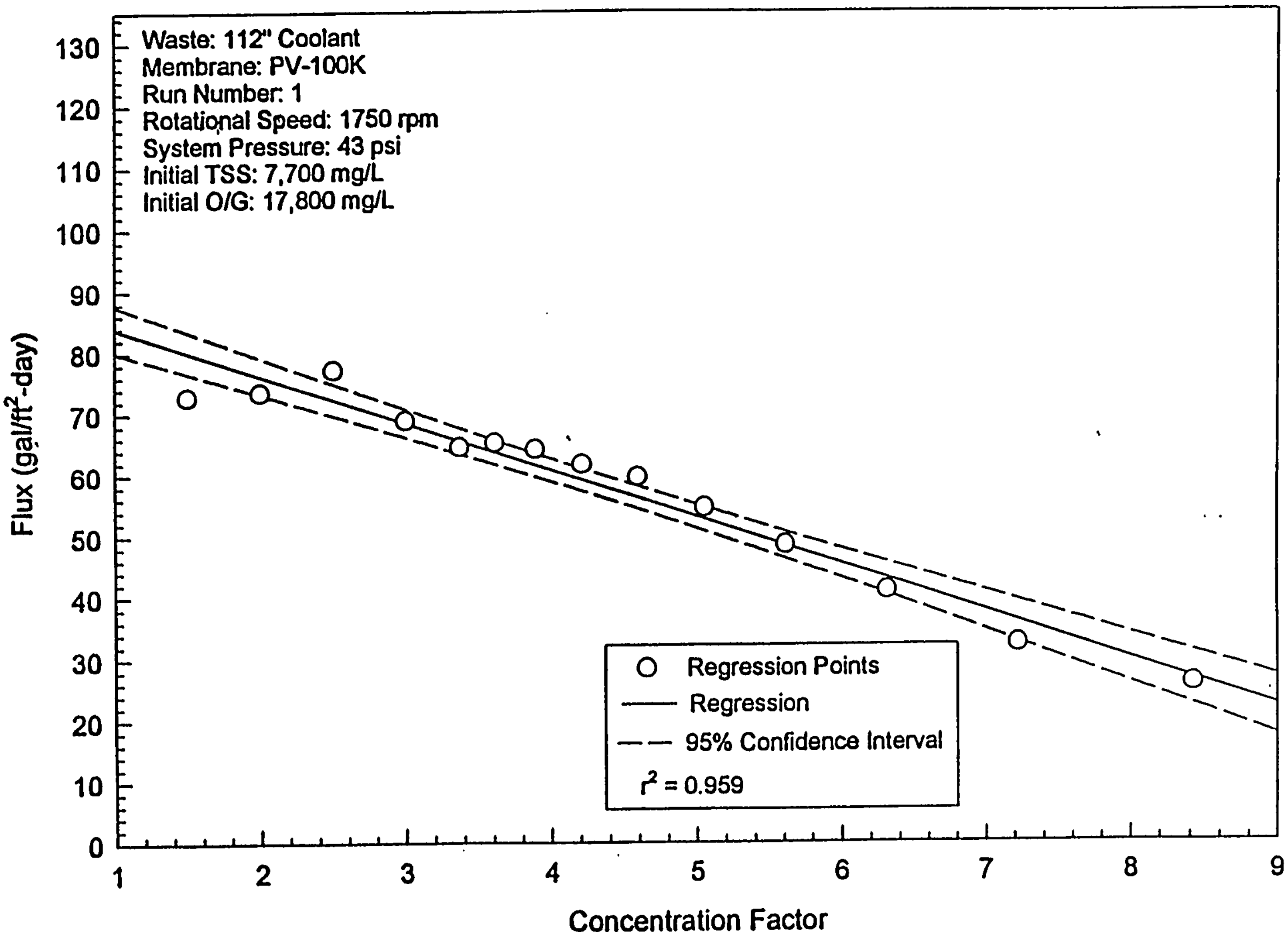

Figure 5. Permeate Flux Versus Concentration Factor for Run 1-Polymeric Membrane.

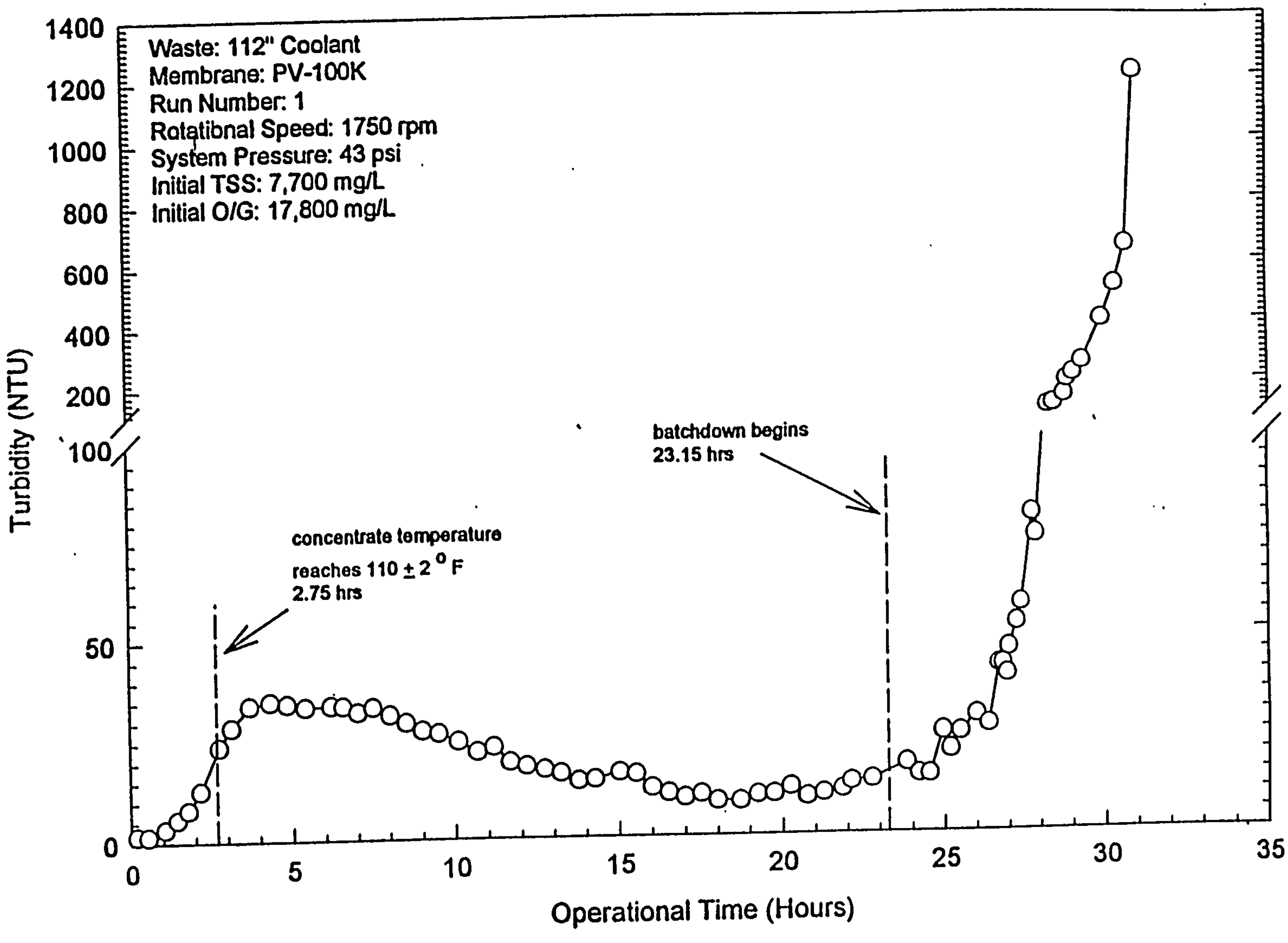

Figure 6. Permeate Turbidity Versus Time for Run 1-Polymeric Membrane.

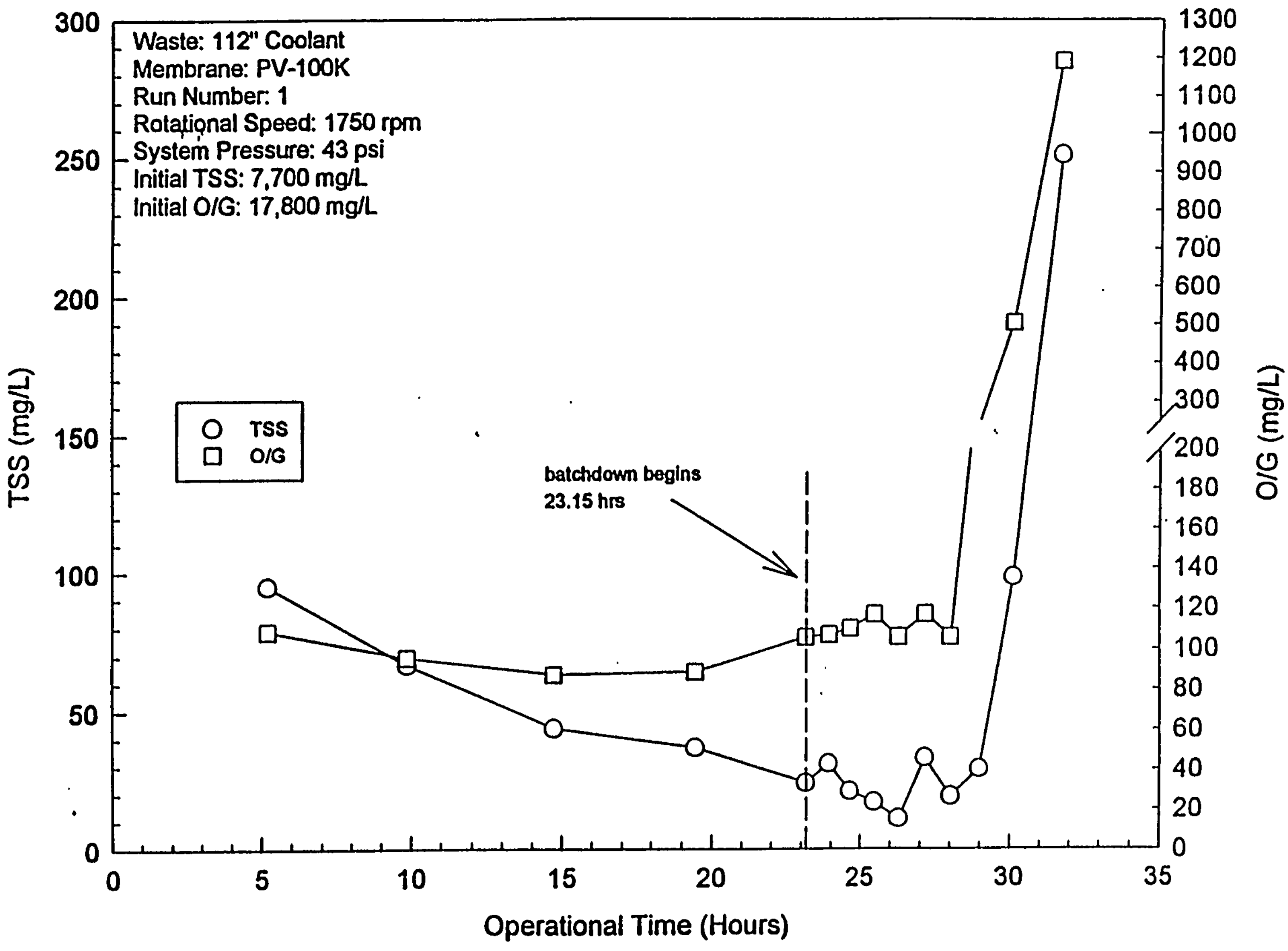

Figure 7. Permeate O/G and TSS Versus Time for Run 1-Polymeric Membrane.

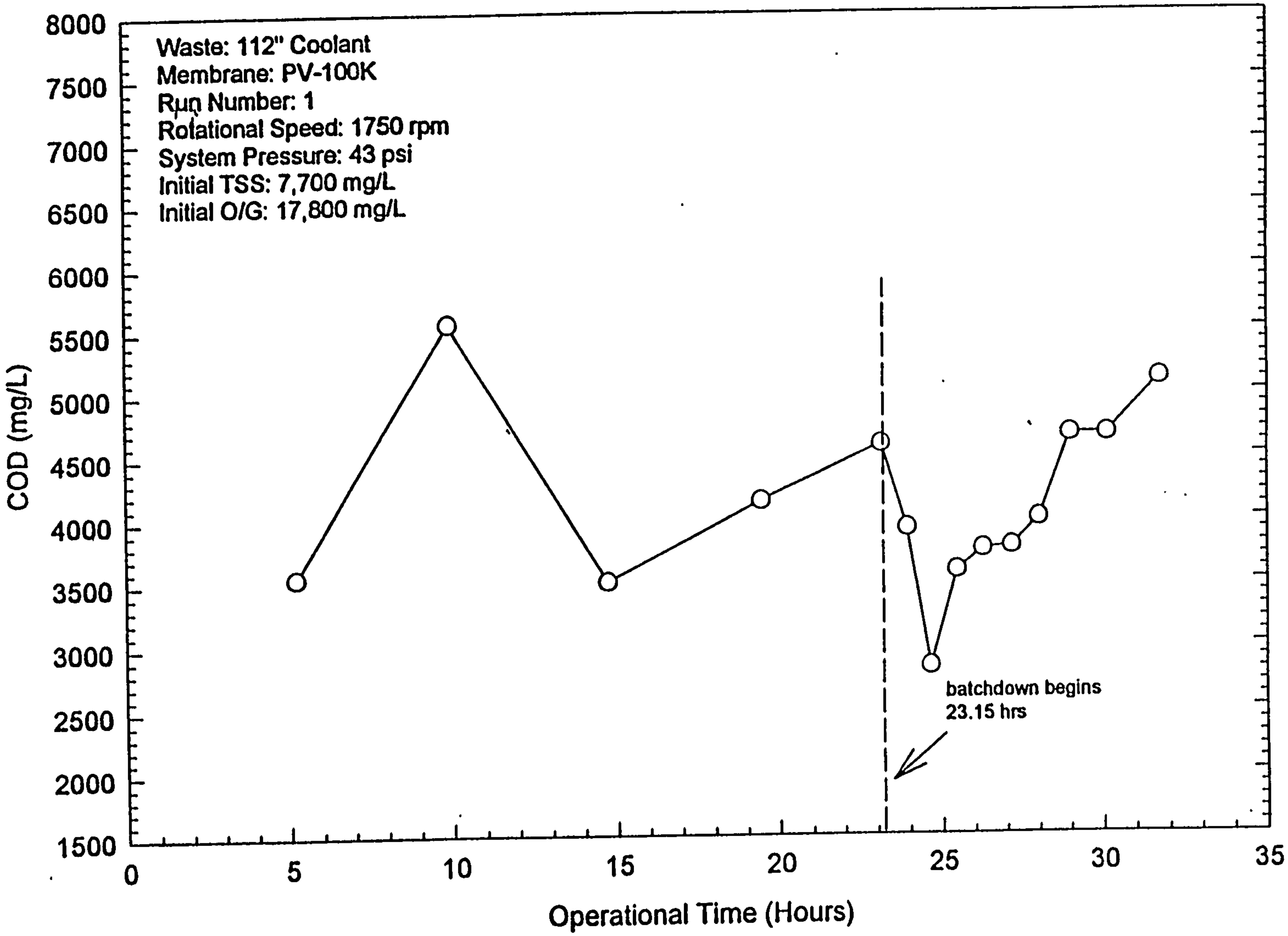

Figure 8. Permeate COD Versus Time for Run 1-Polymeric Membrane.

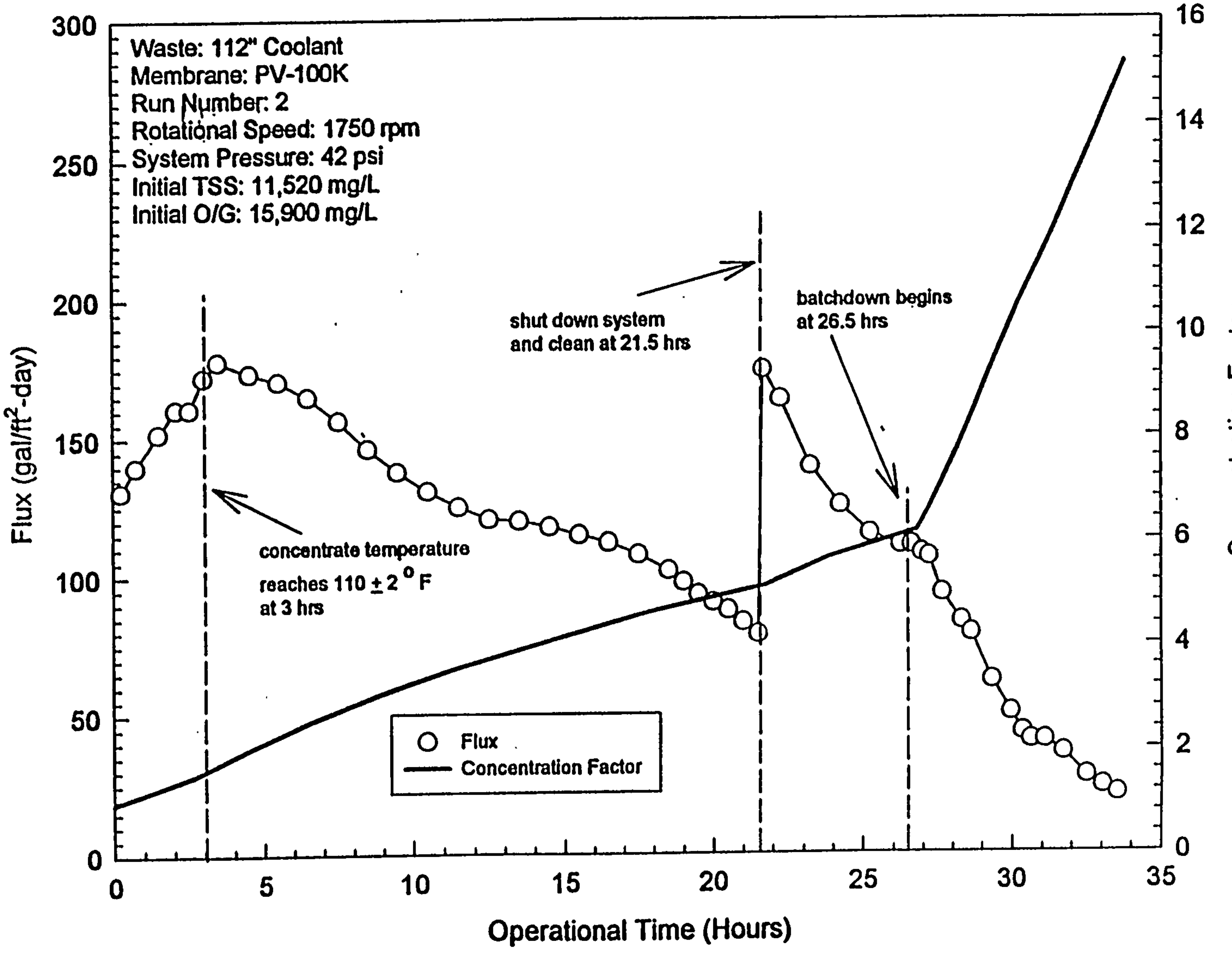

Figure 9. Permeate Flux and Concentration Factor Versus Time for Run 2-Polymeric Membrane.

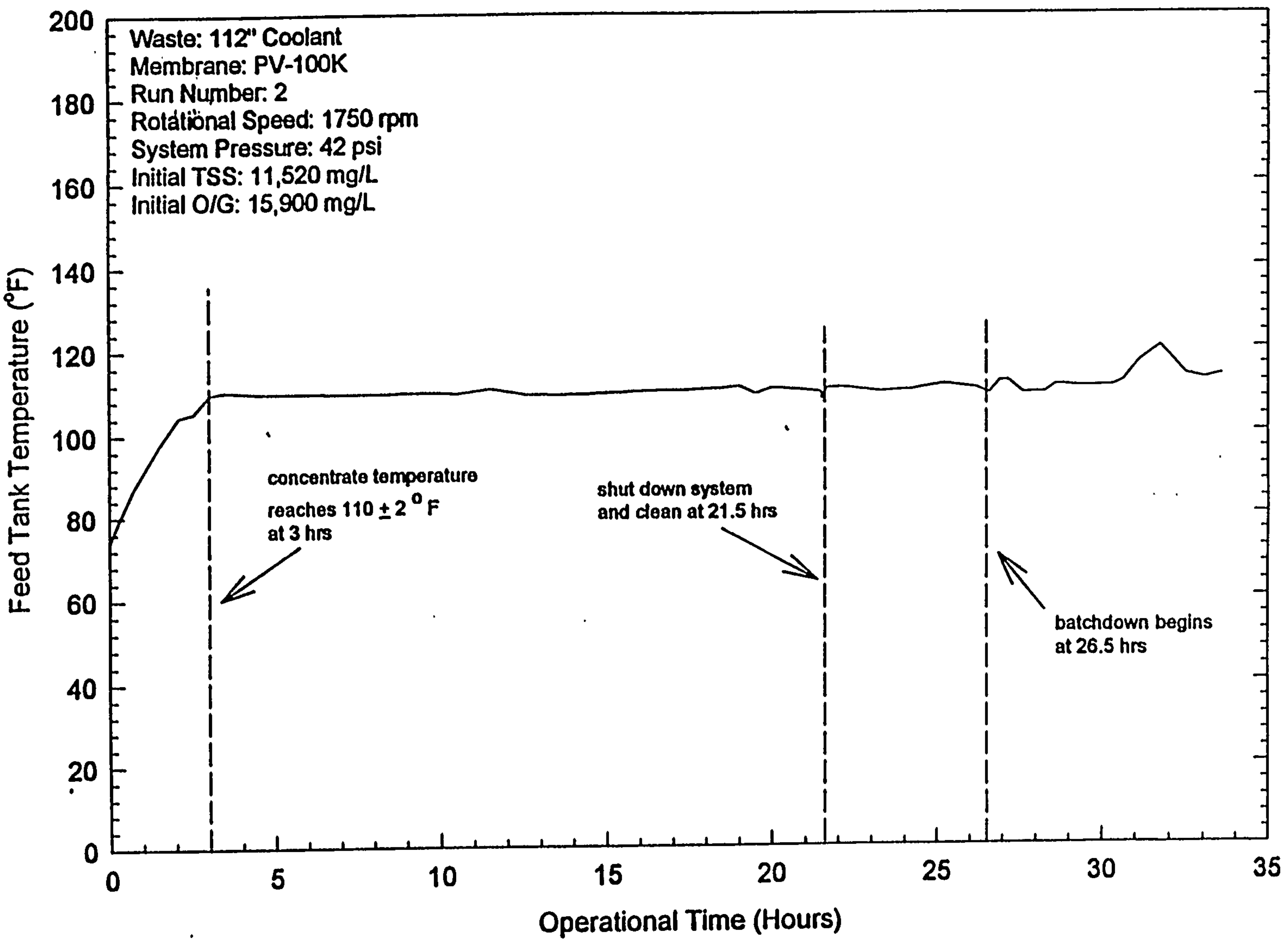

Figure 10. Feed Tank Temperature Versus Time for Run 2-Polymeric Membrane.

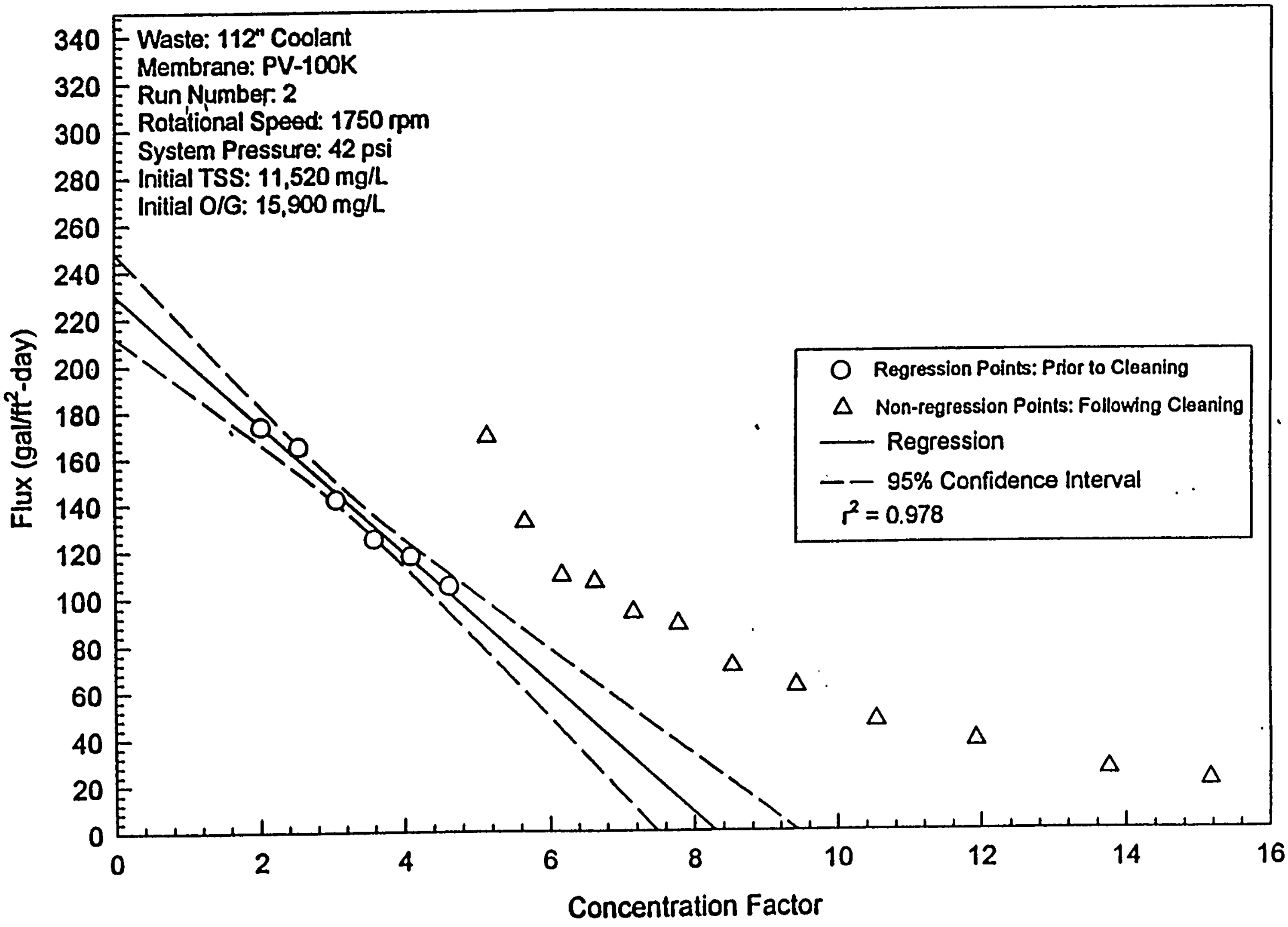

Figure 11. Permeate Flux Versus Concentration Factor for Run 2-Polymeric Membrane.

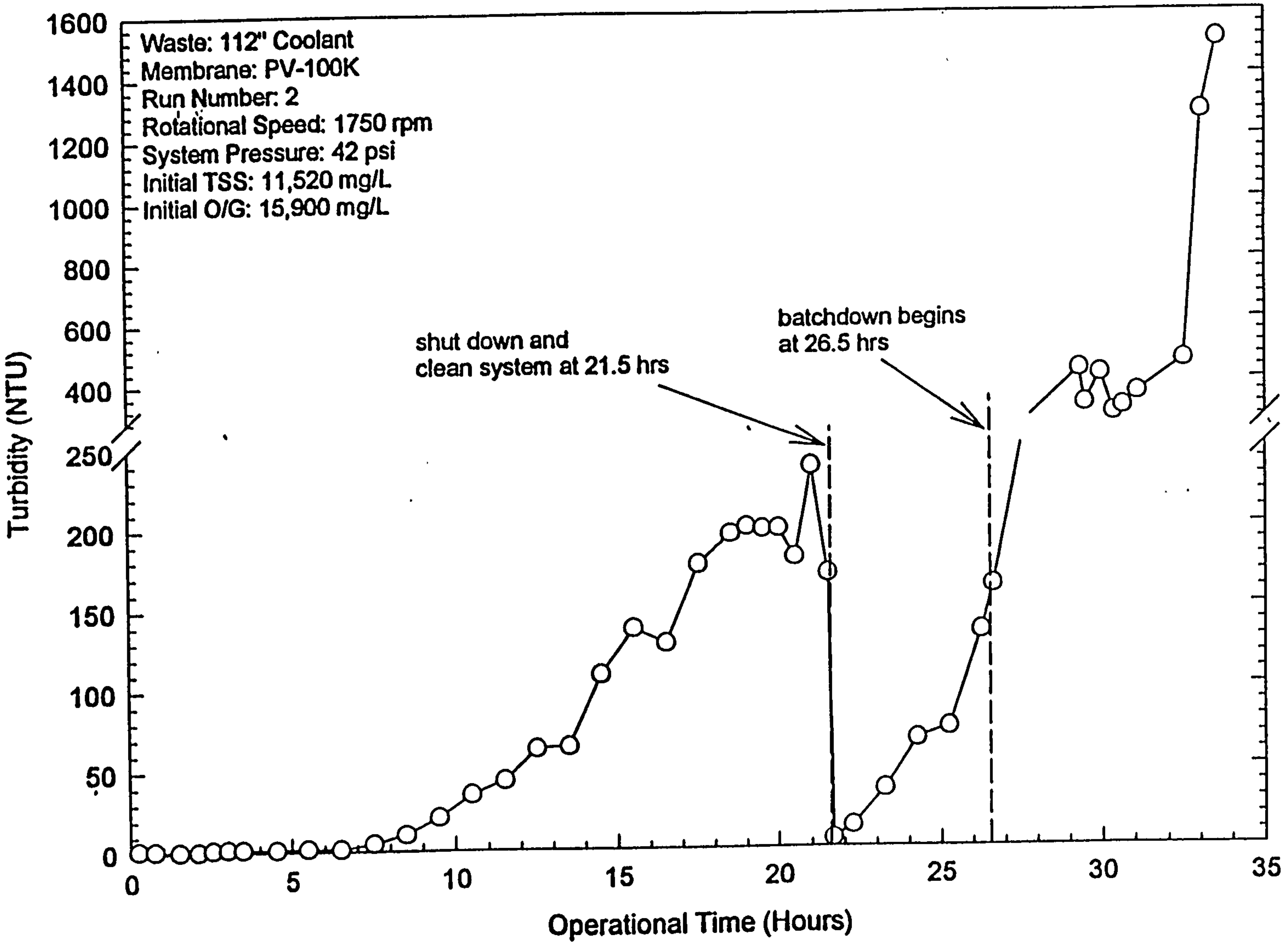

Figure 12. Permeate Turbidity Versus Time for Run 2-Polymeric Membrane.

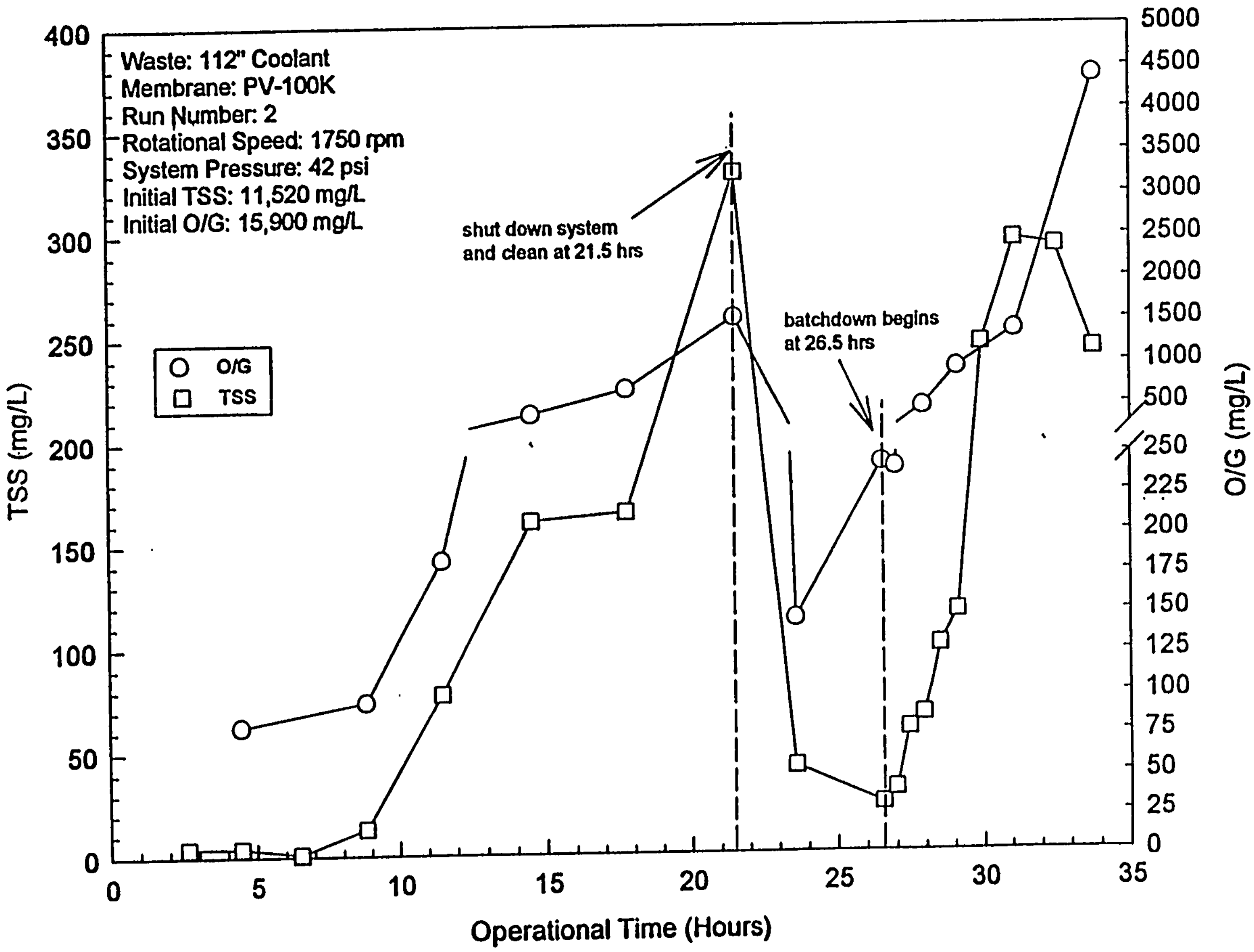

Figure 13. Permeate O/G and TSS Versus Time for Run 2-Polymeric Membrane.

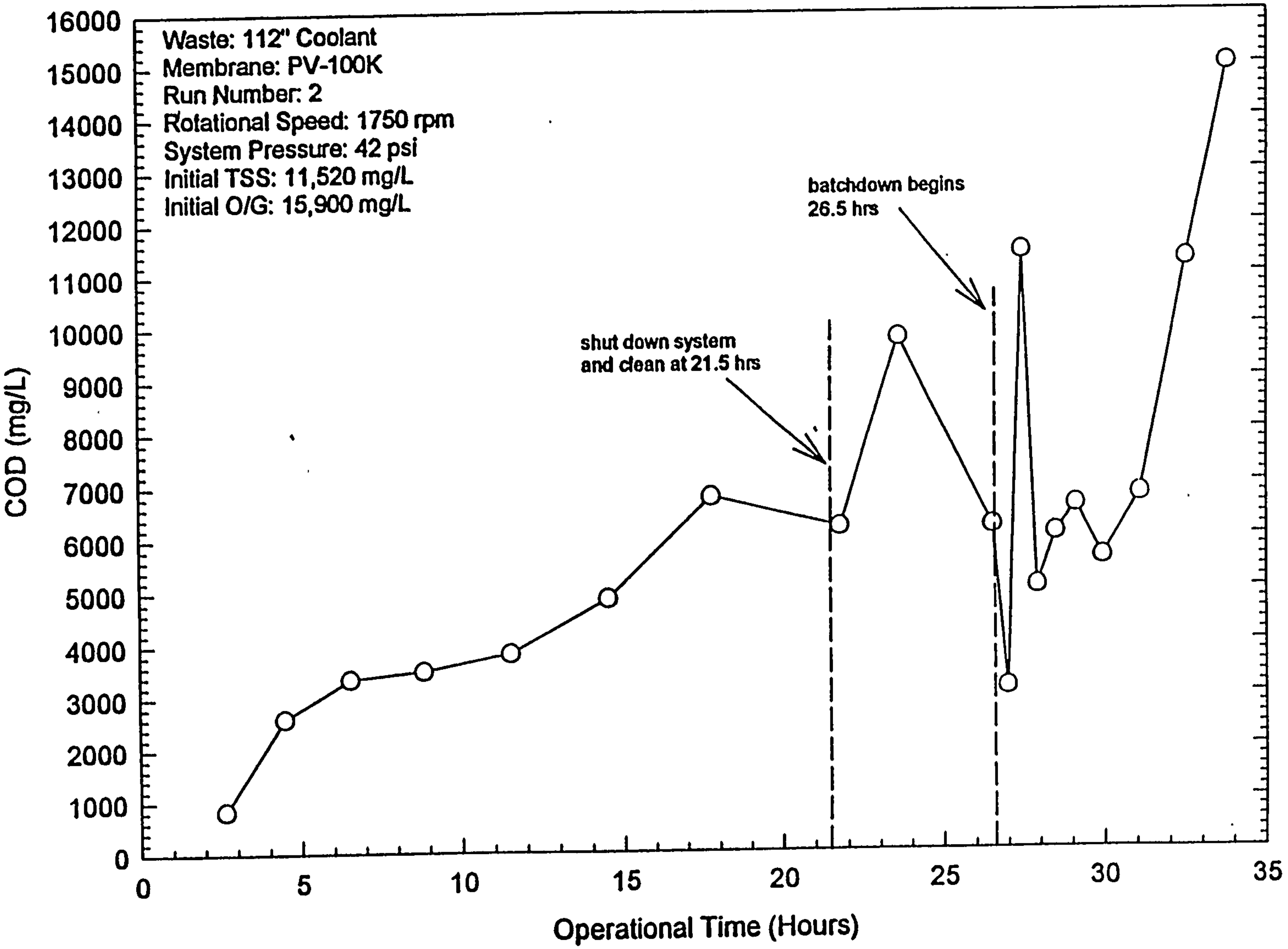

Figure 14. Permeate COD Versus Time for Run 2-Polymeric Membrane.

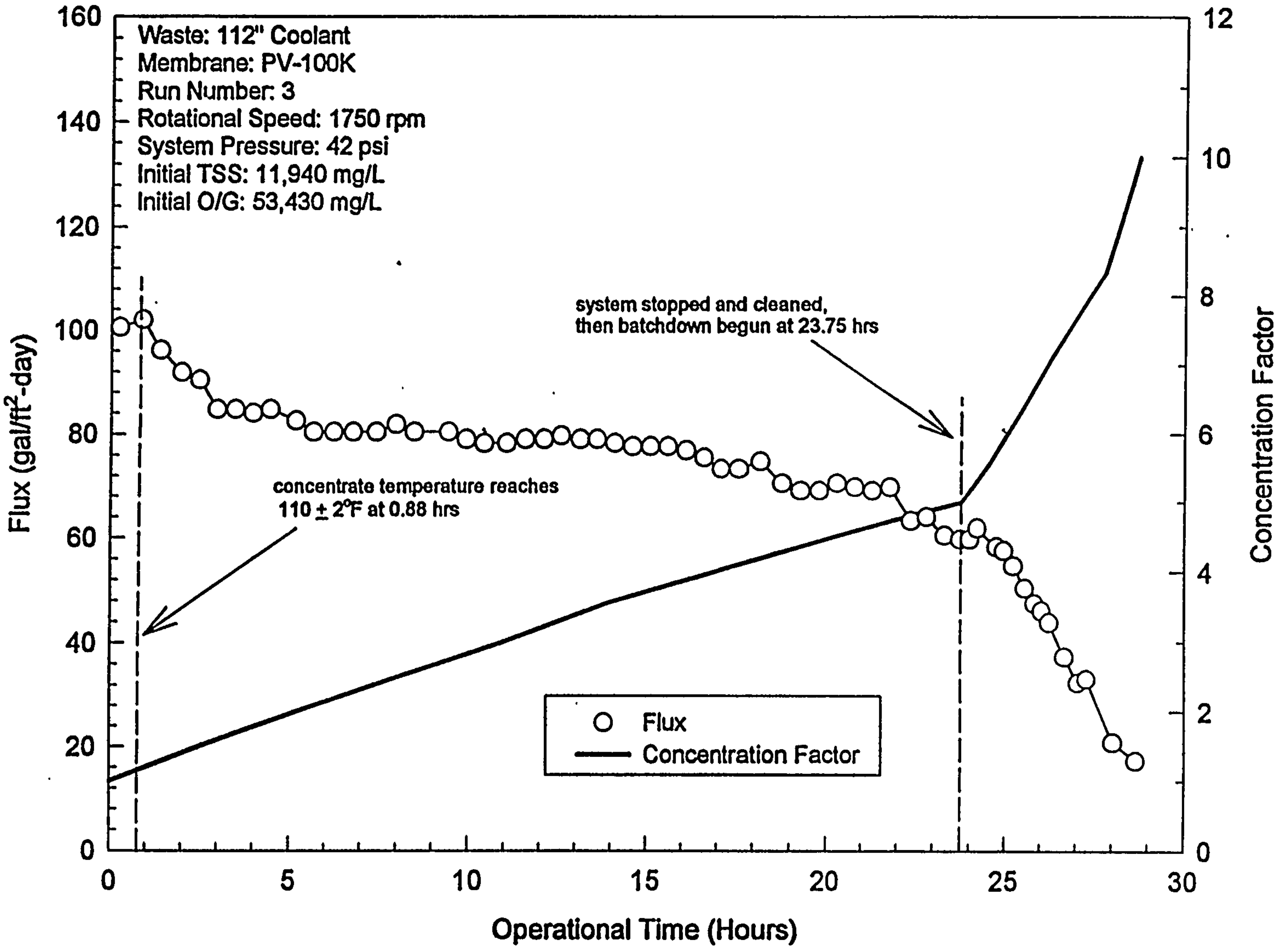

Figure 15. Permeate Flux and Concentration Factor Versus Time for Run 3-Polymeric Membrane.

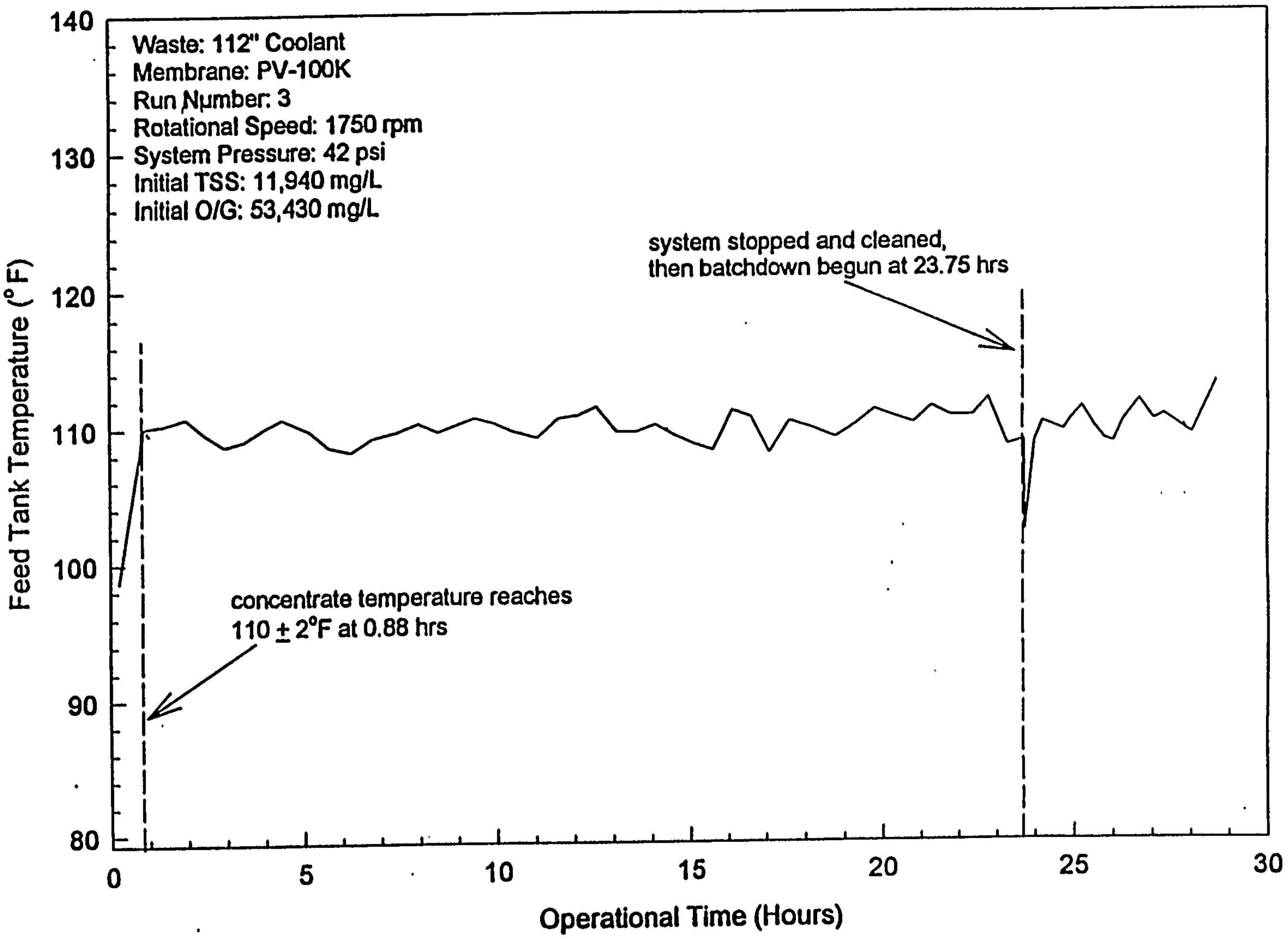

Figure 16. Feed Tank Temperature Versus Time for Run 3-Polymeric Membrane

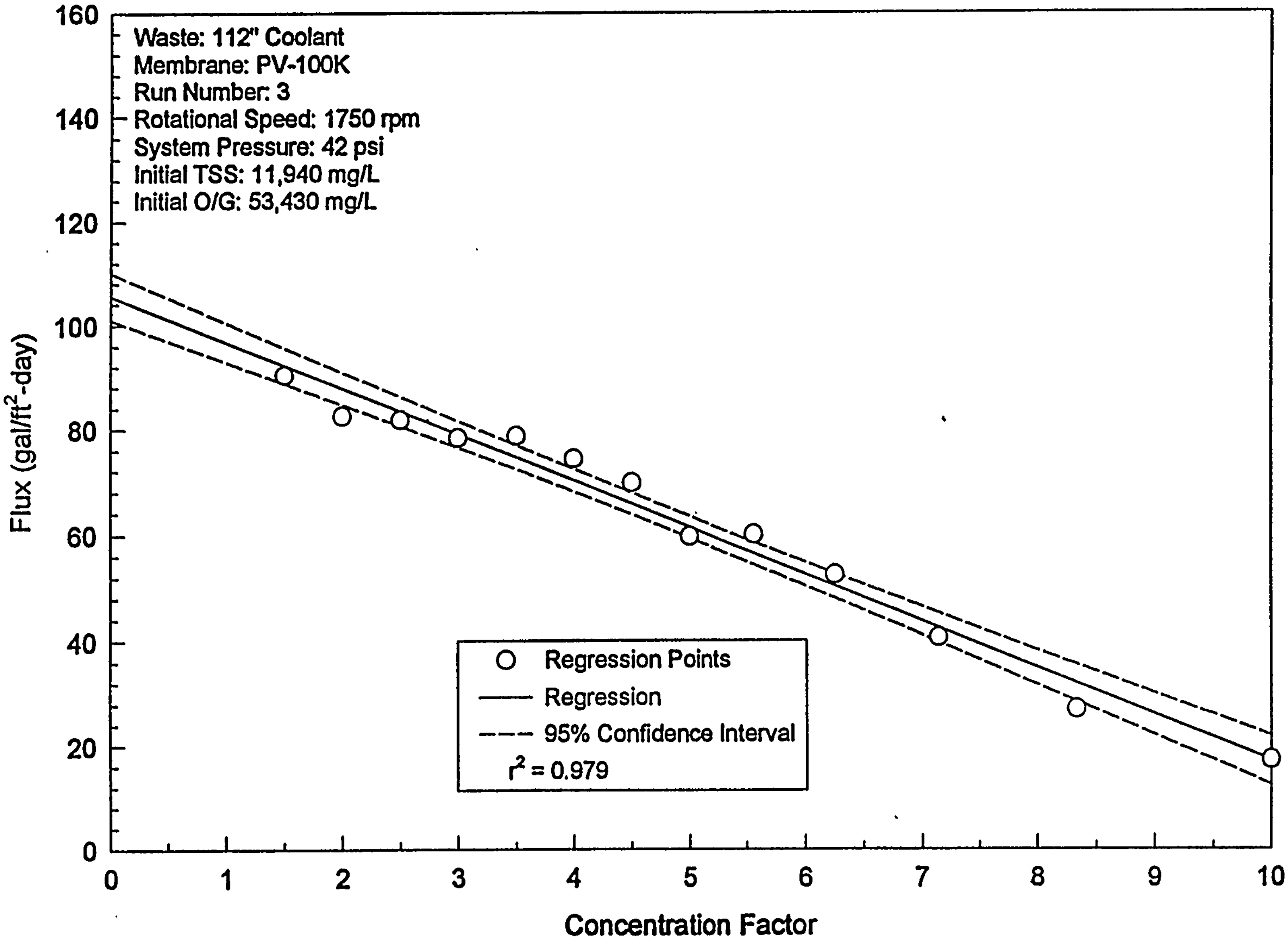

Figure 17. Permeate Flux Versus Concentration Factor for Run 3-Polymeric Membrane.

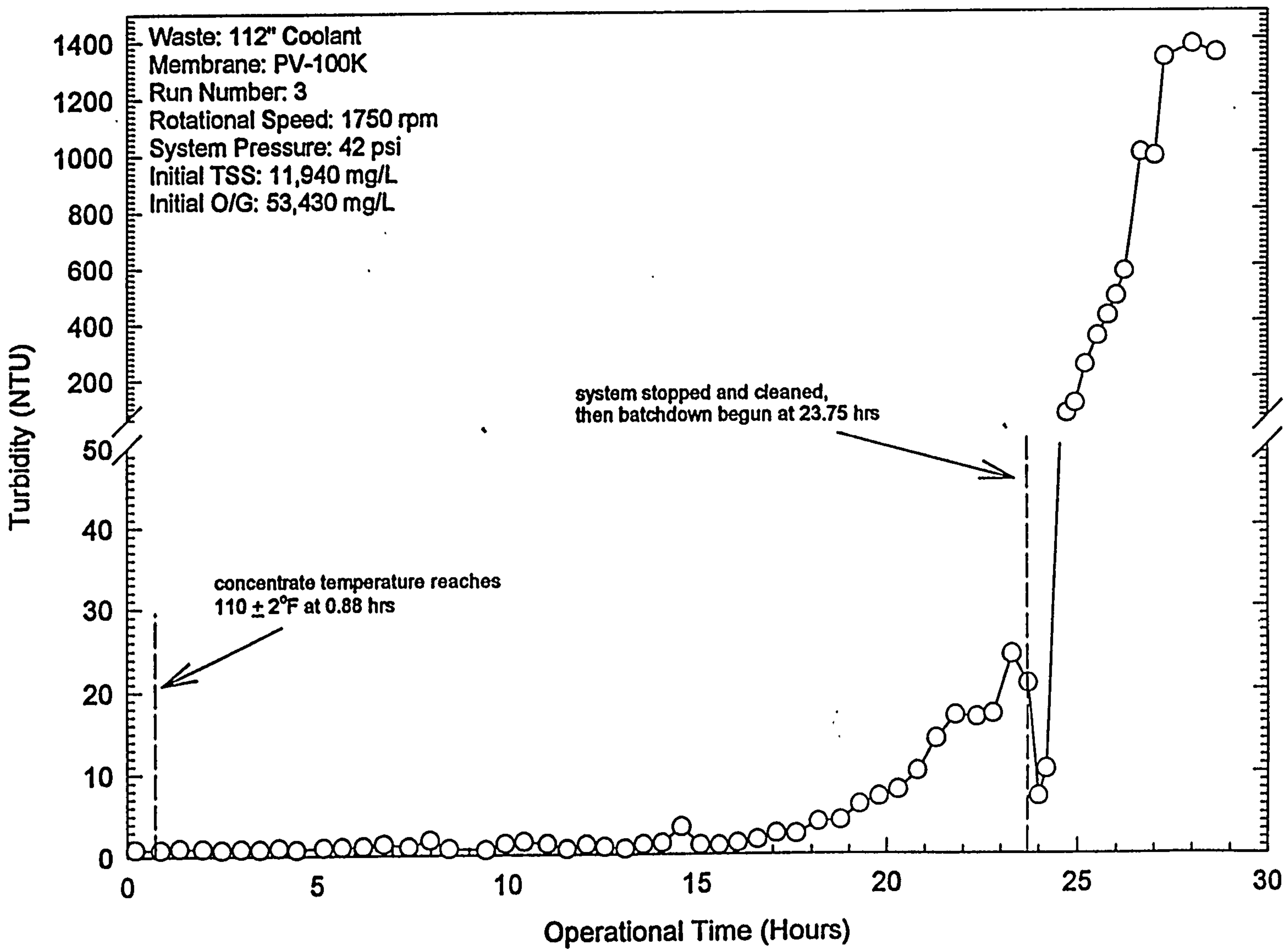

Figure 18. Permeate Turbidity Versus Time for Run 3-Polymeric Membrane.

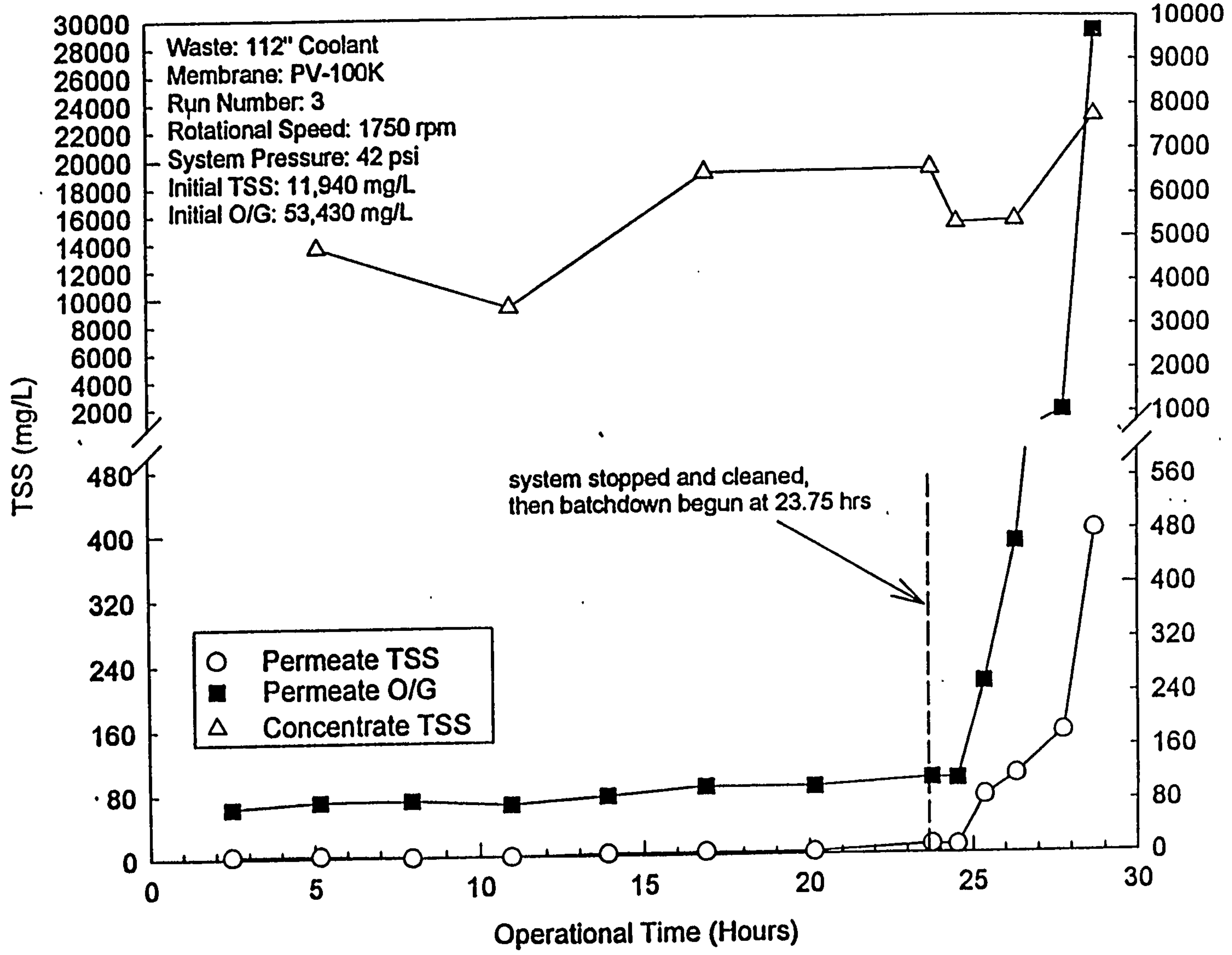

Figure 19. Permeate O/G and TSS Versus Time for Run 3-Polymeric Membrane.

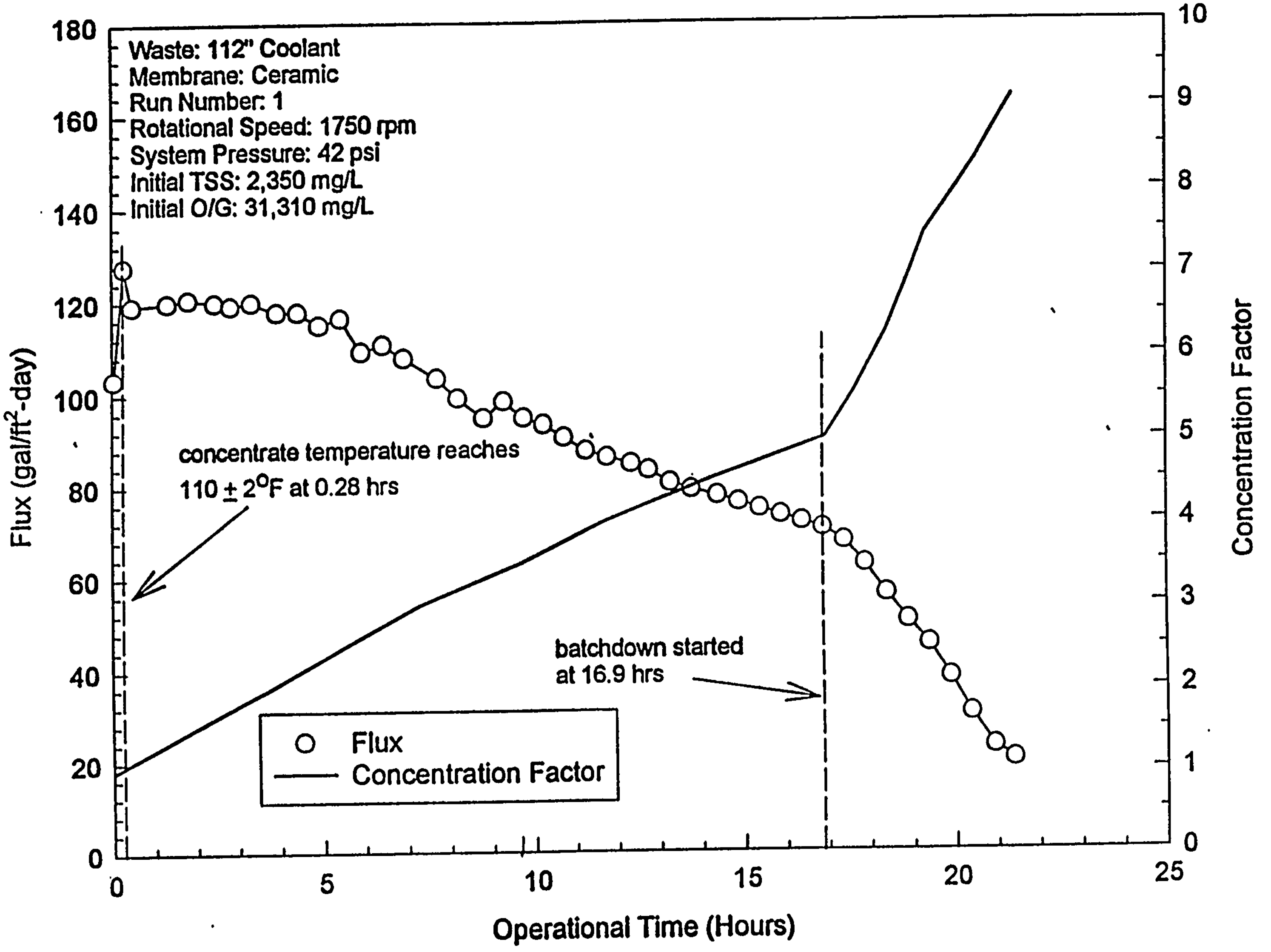

Figure 20. Permeate Flux and Concentration Factor Versus Time for Run 1-Ceramic Membrane.

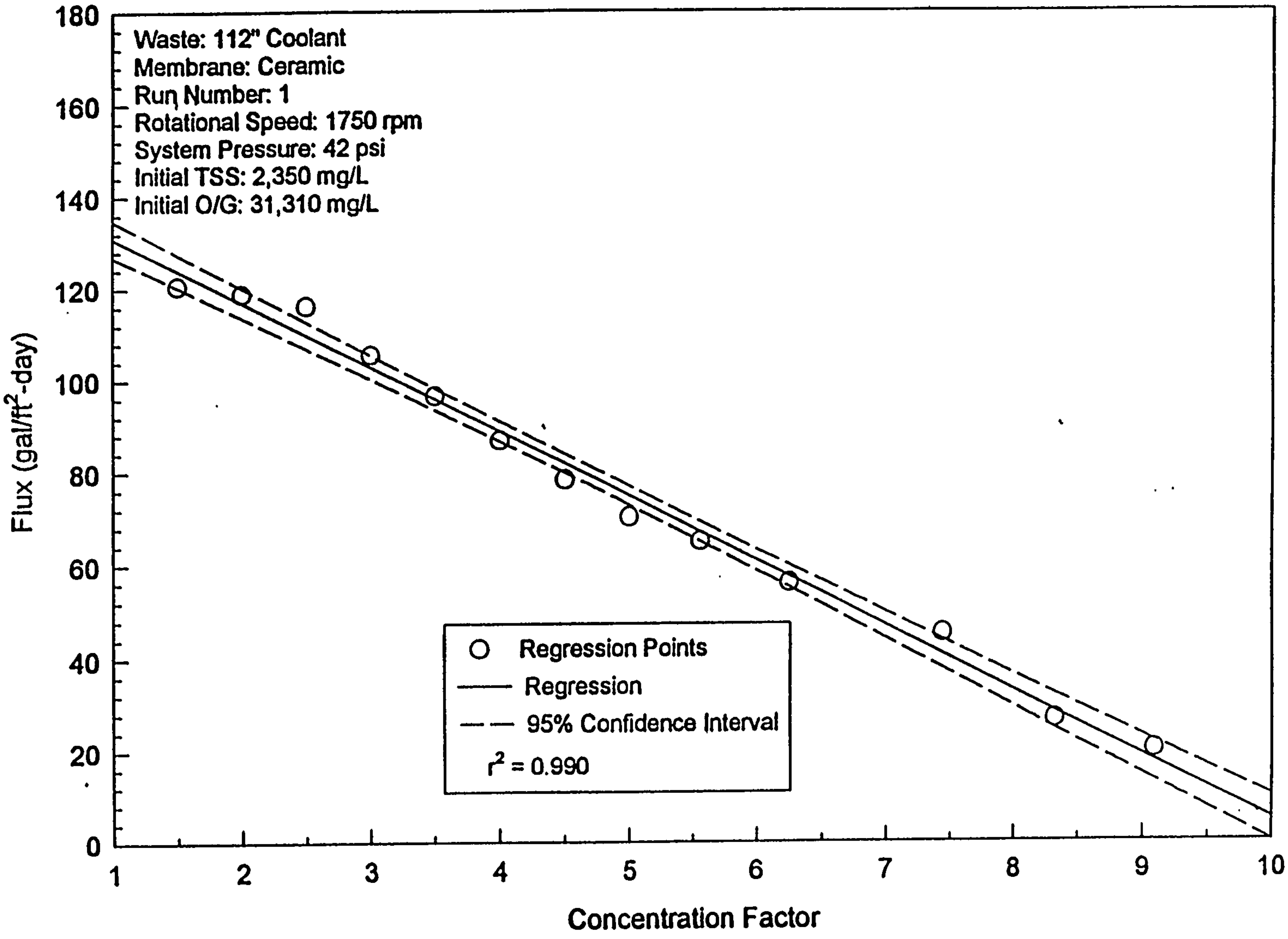

Figure 21. Permeate Flux Versus Concentration Factor for Run 1-Ceramic Membrane.

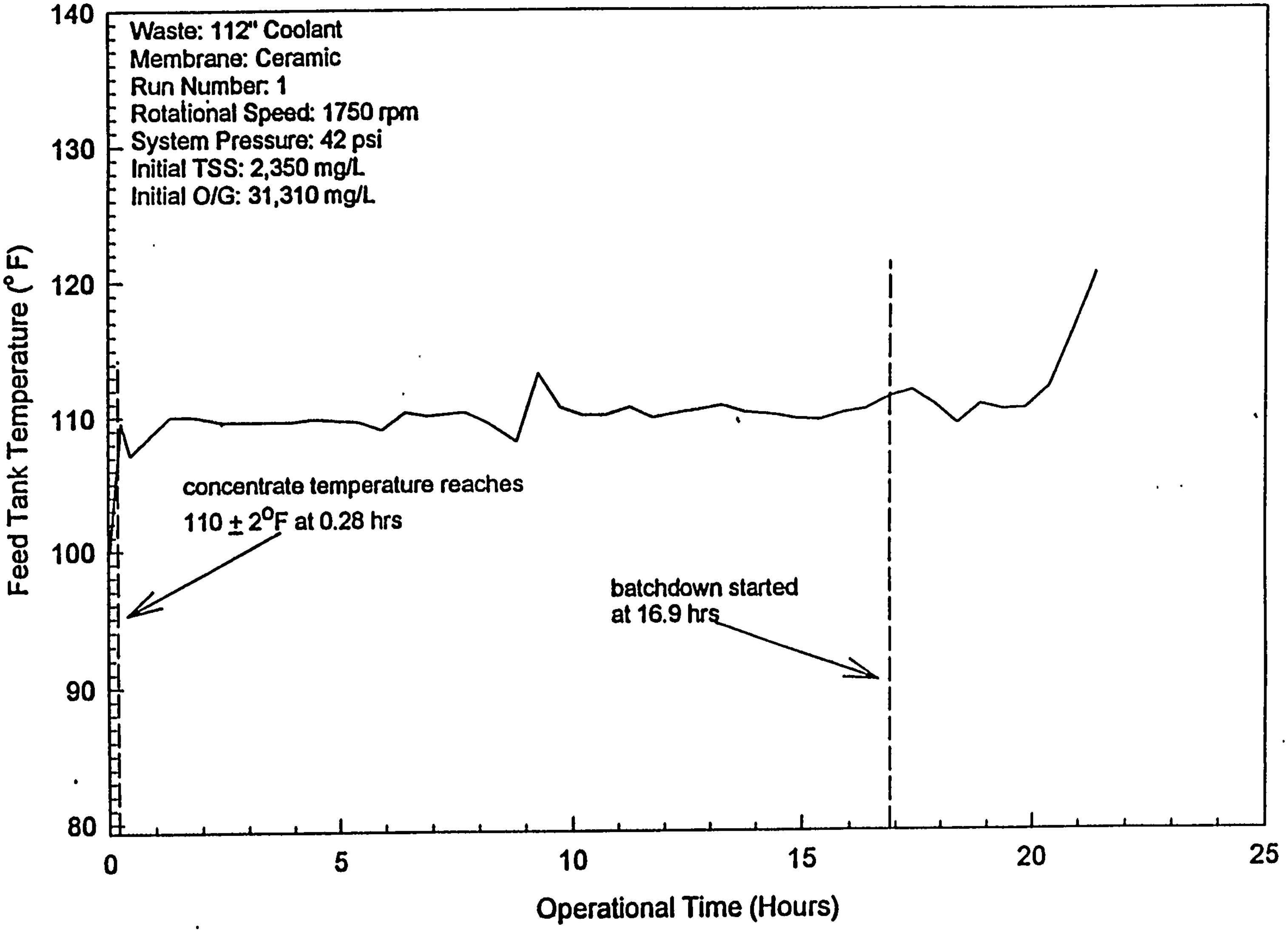

Figure 22. Feed Tank Temperature Versus Time for Run 1-Ceramic Membrane.

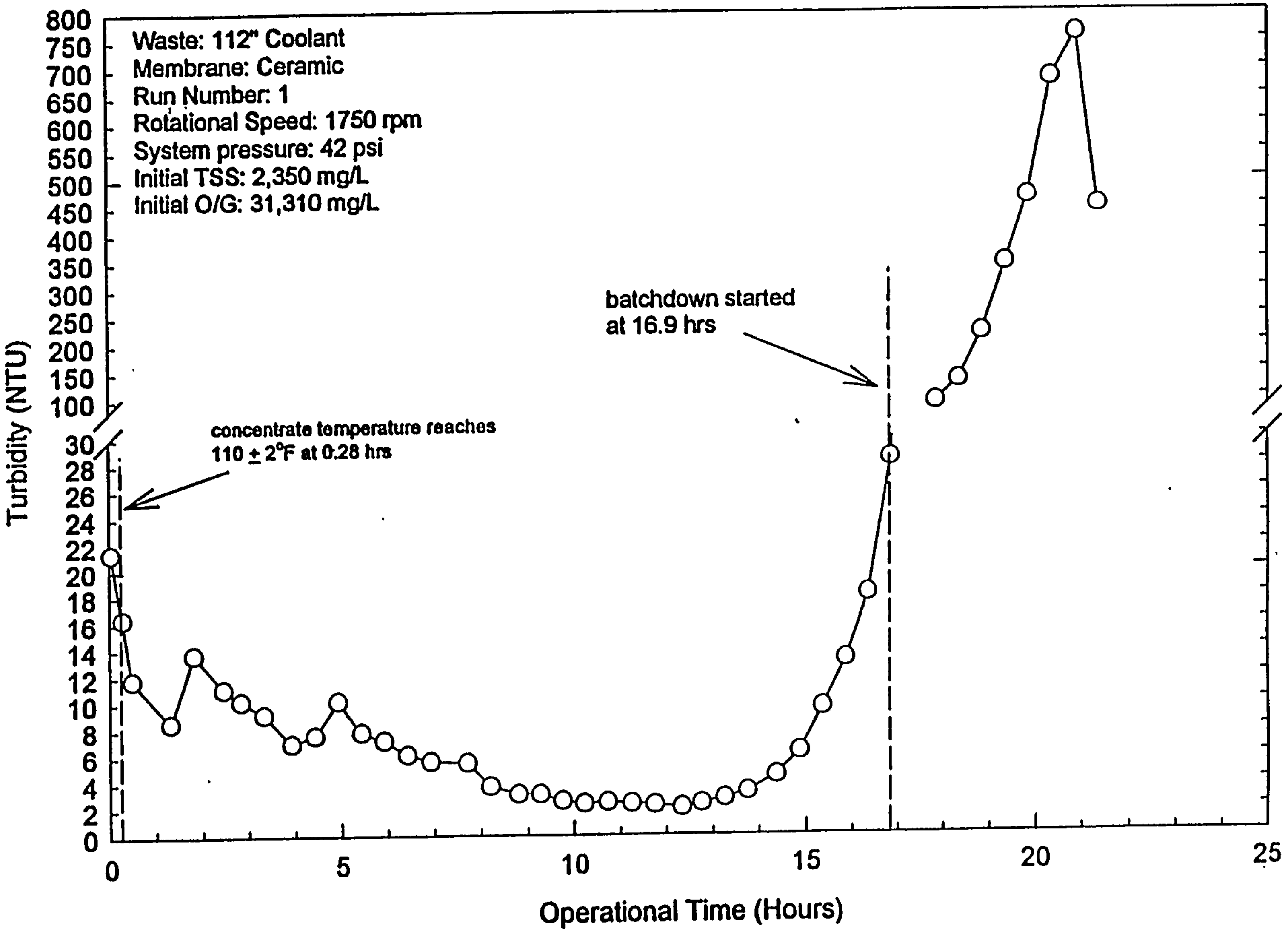

Figure 23. Permeate Turbidity Versus Time for Run 1-Ceramic Membrane.

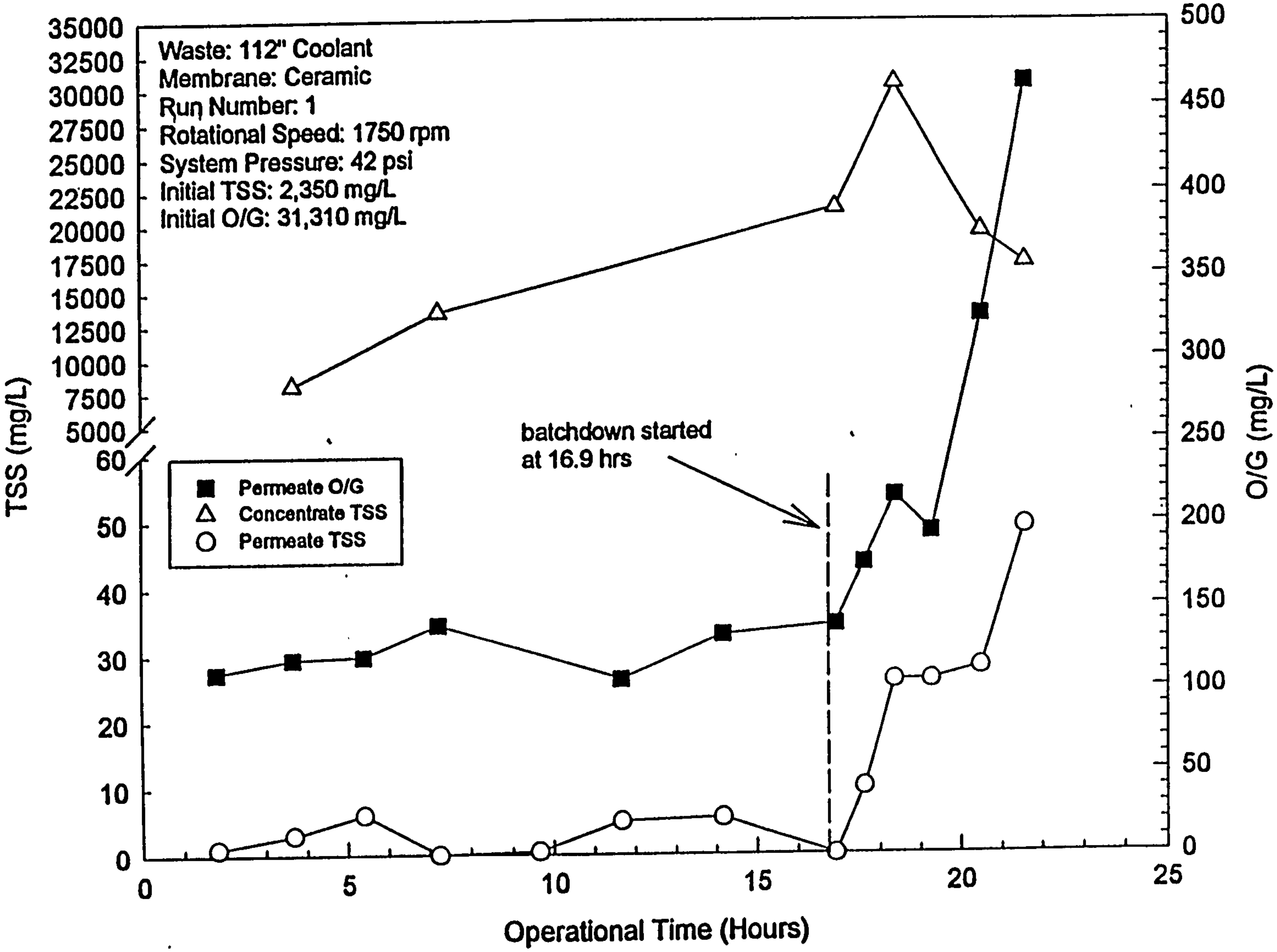

Figure 24. Permeate O/G and TSS Versus Time for Run 1-Ceramic Membrane.

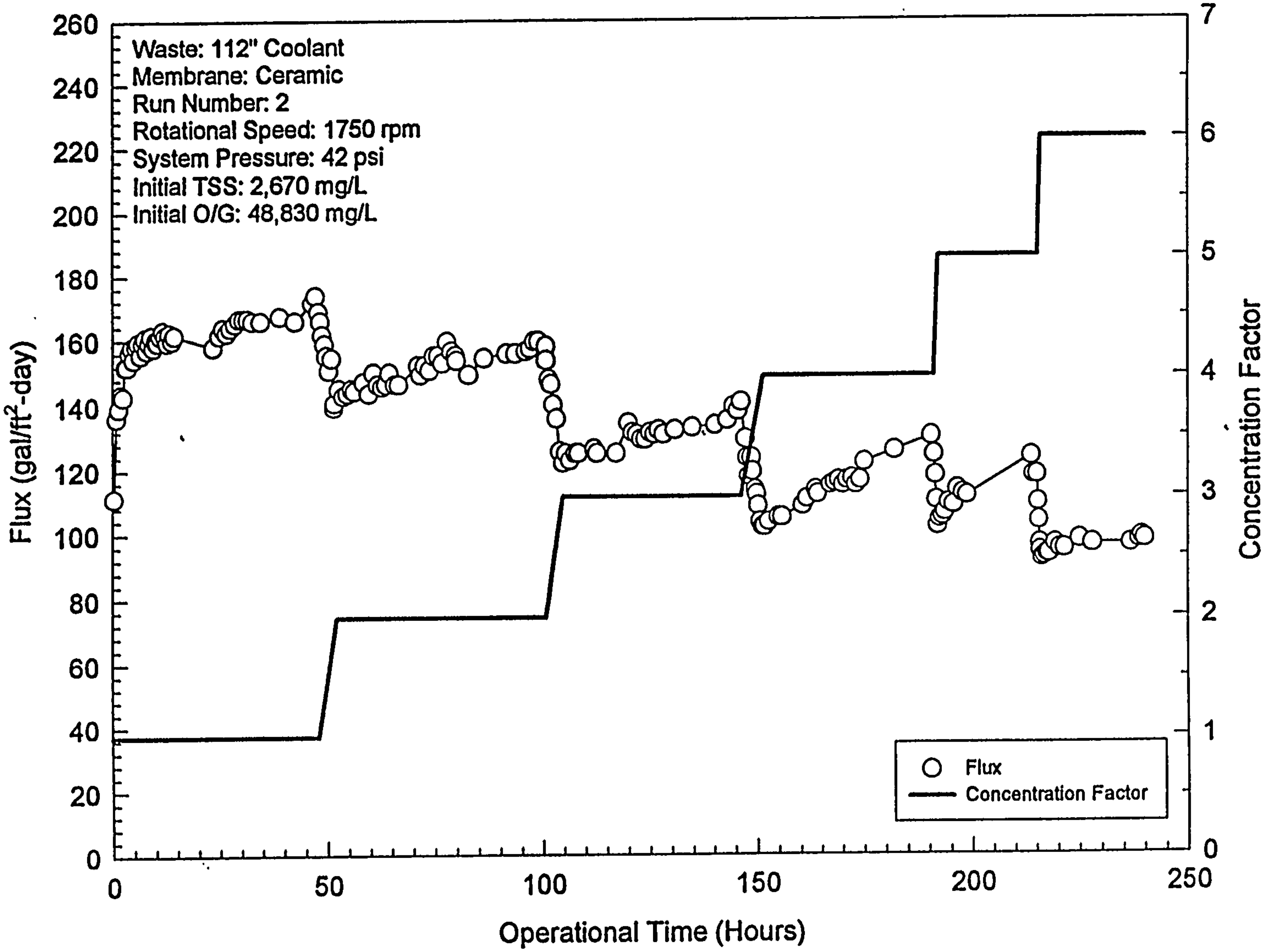

Figure 25. Permeate Flux and Concentration Factor Versus Time for Run 2-Ceramic Membrane.

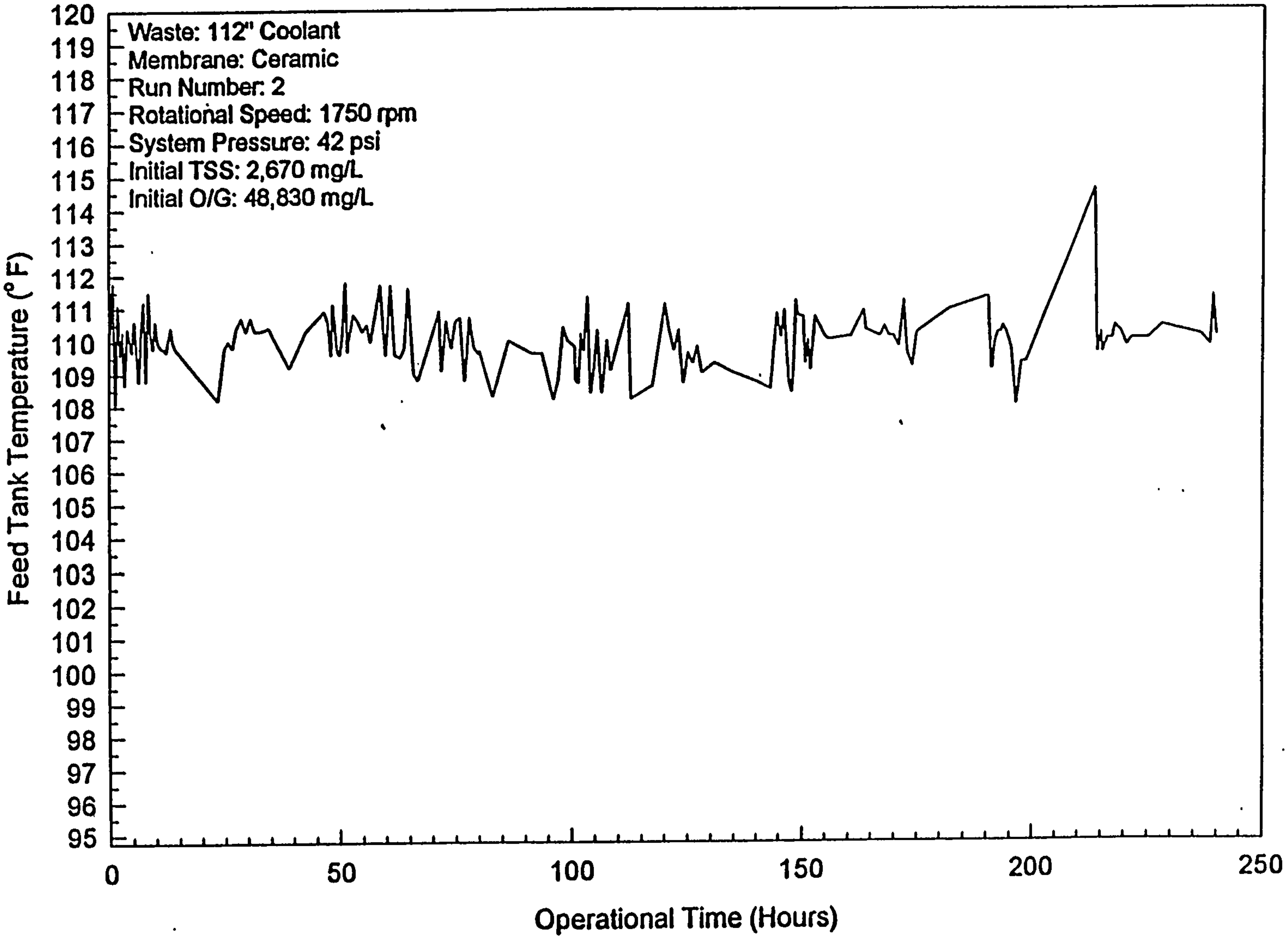

Figure 26. Feed Tank Temperature Versus Time for Run 2-Ceramic Membrane.

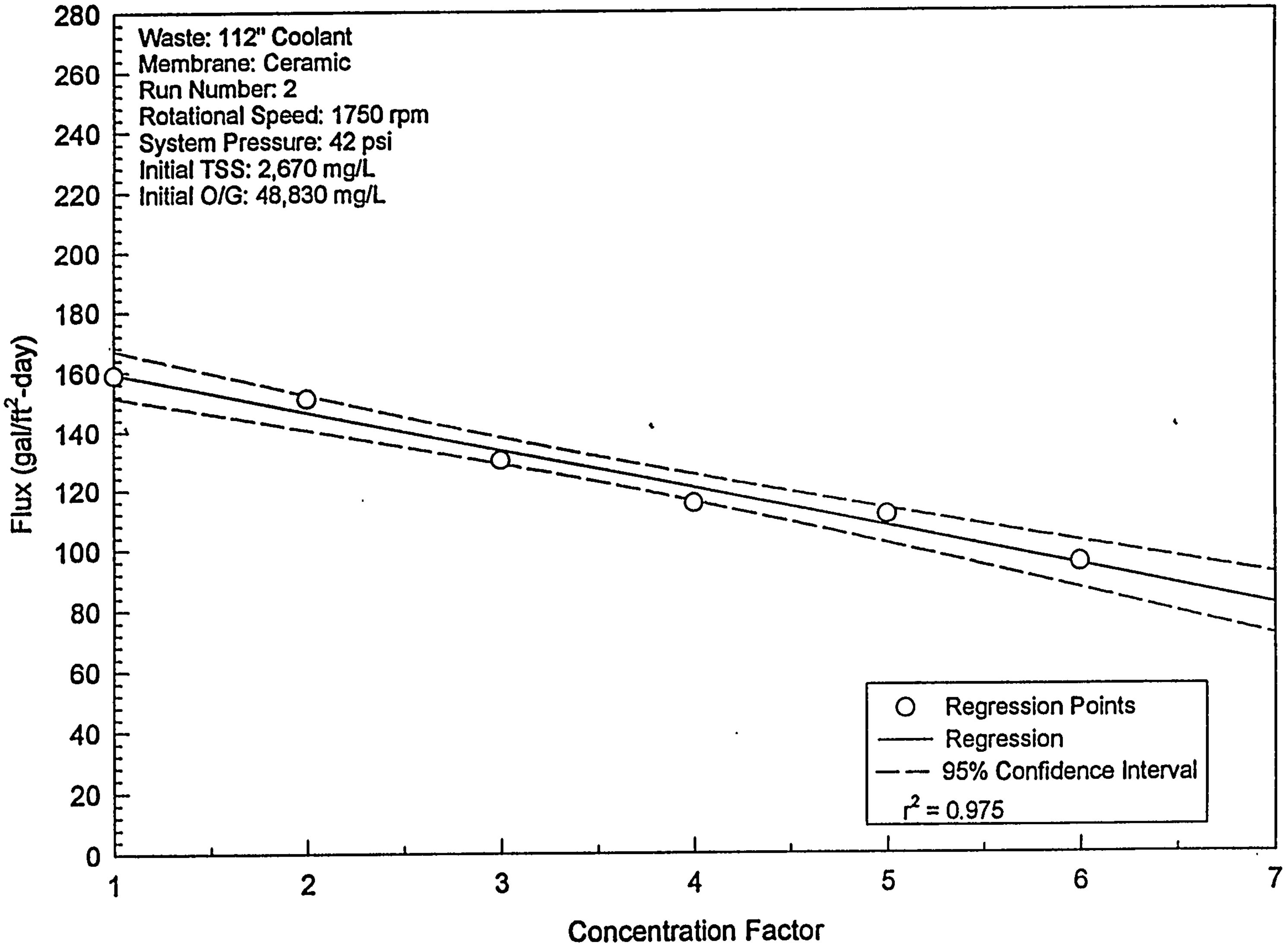

Figure 27. Permeate Flux Versus Concentration Factor for Run 2-Ceramic Membrane.

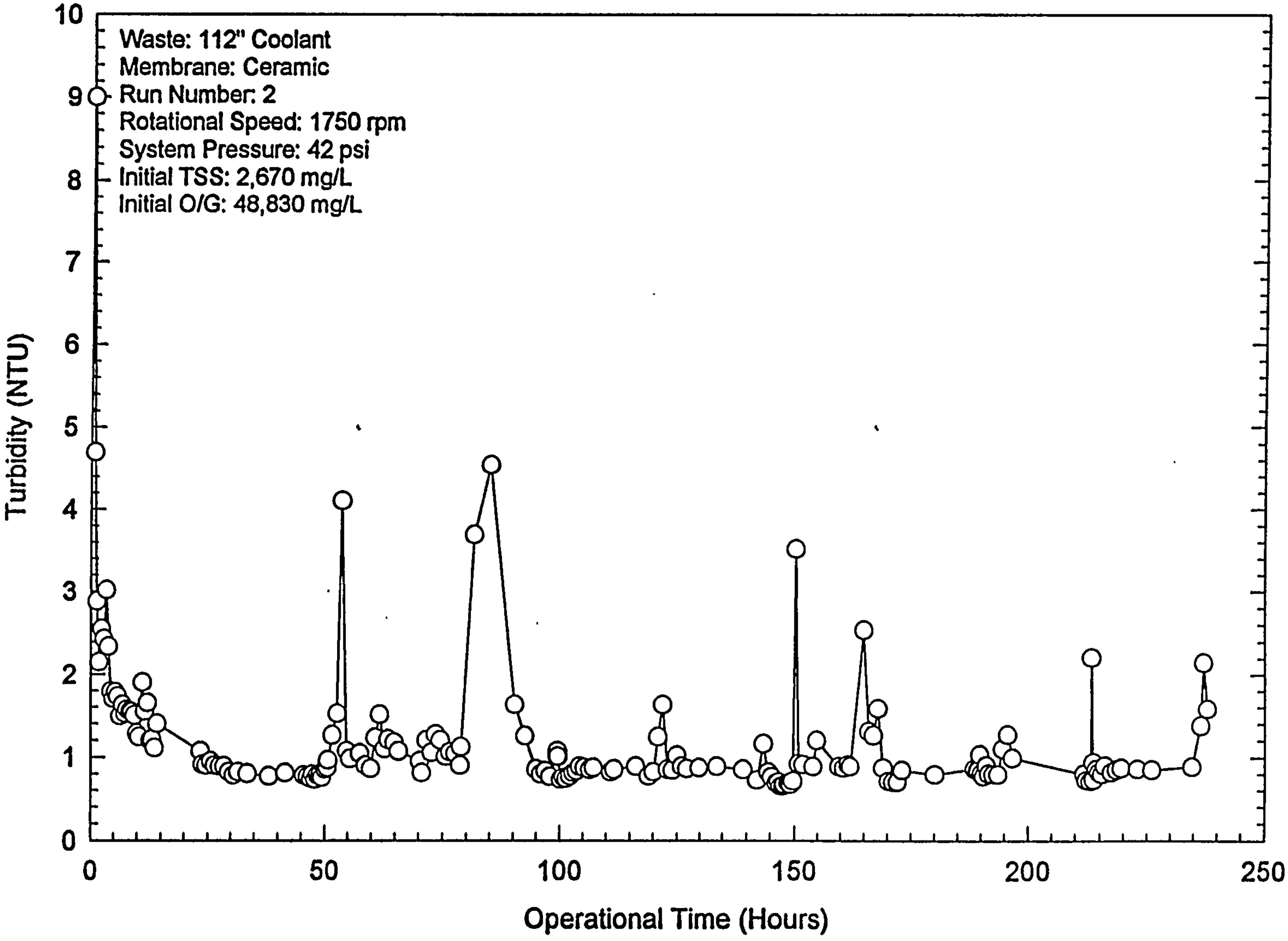

Figure 28. Permeate Turbidity Versus Time for Run 2-Ceramic Membrane.

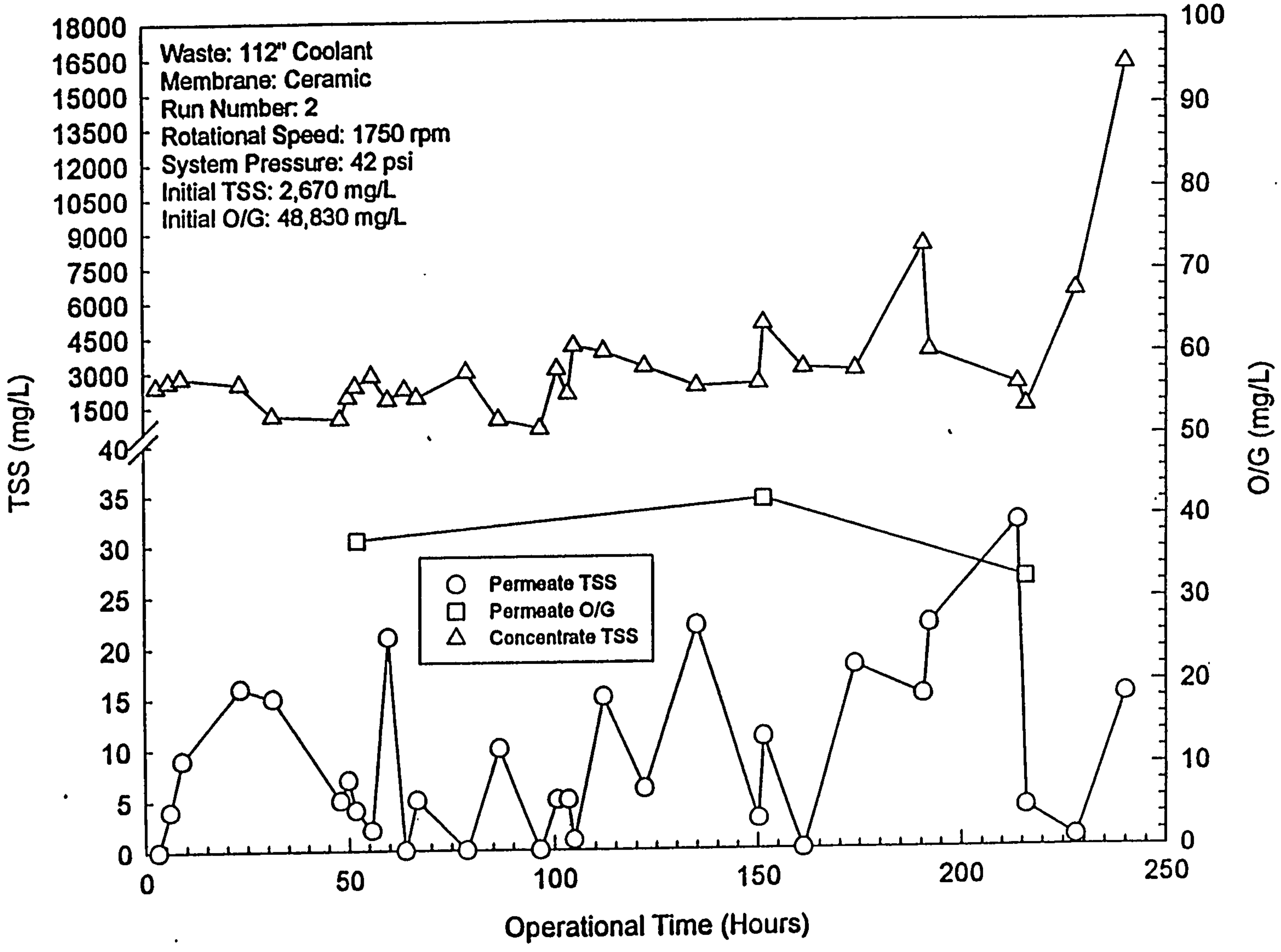

Figure 29. Permeate O/G and TSS Versus Time for Run 2-Ceramic Membrane.

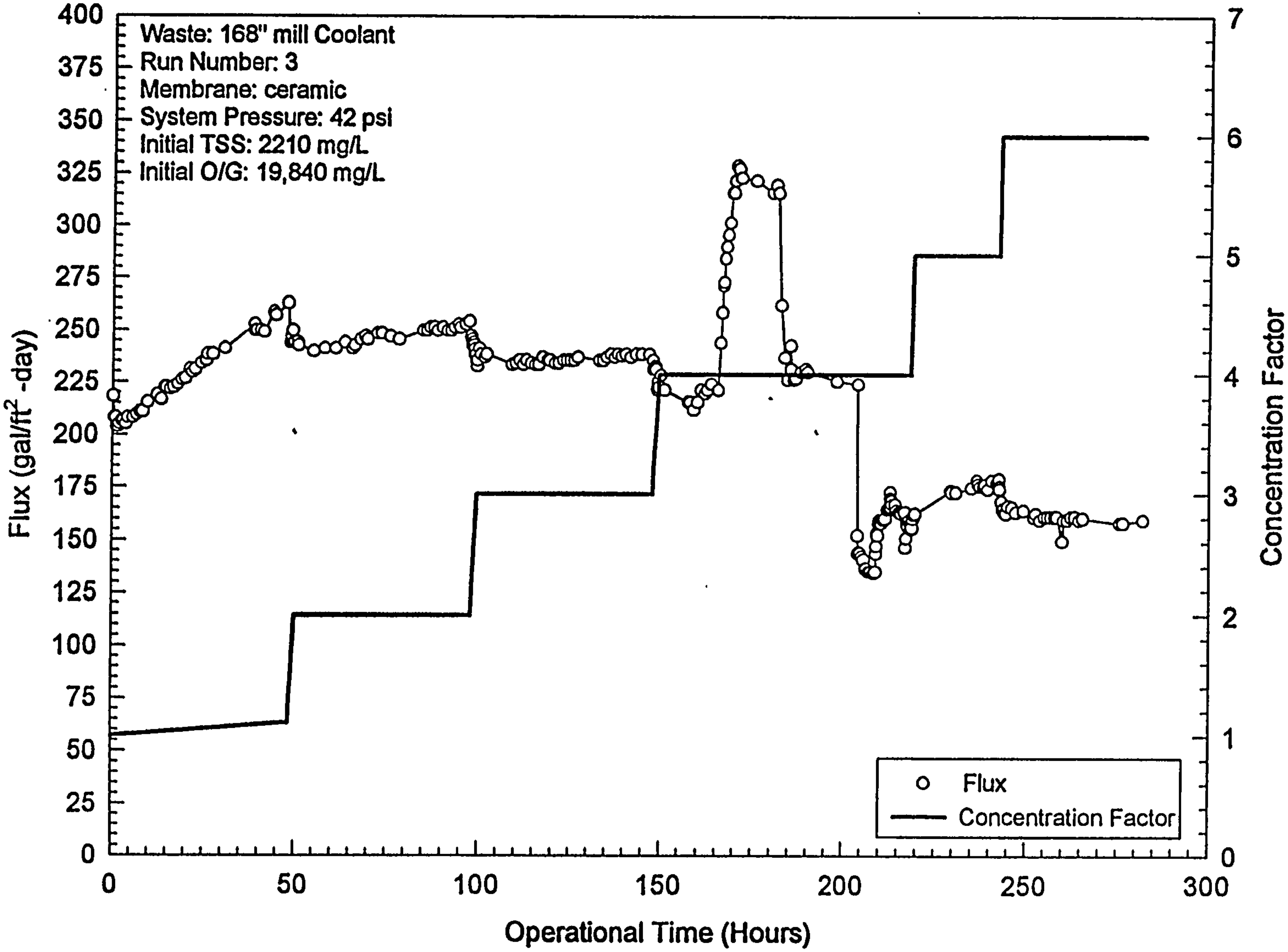

Figure 30. Permeate Flux and Concentration Factor Versus Time for Run 3-Ceramic Membrane.

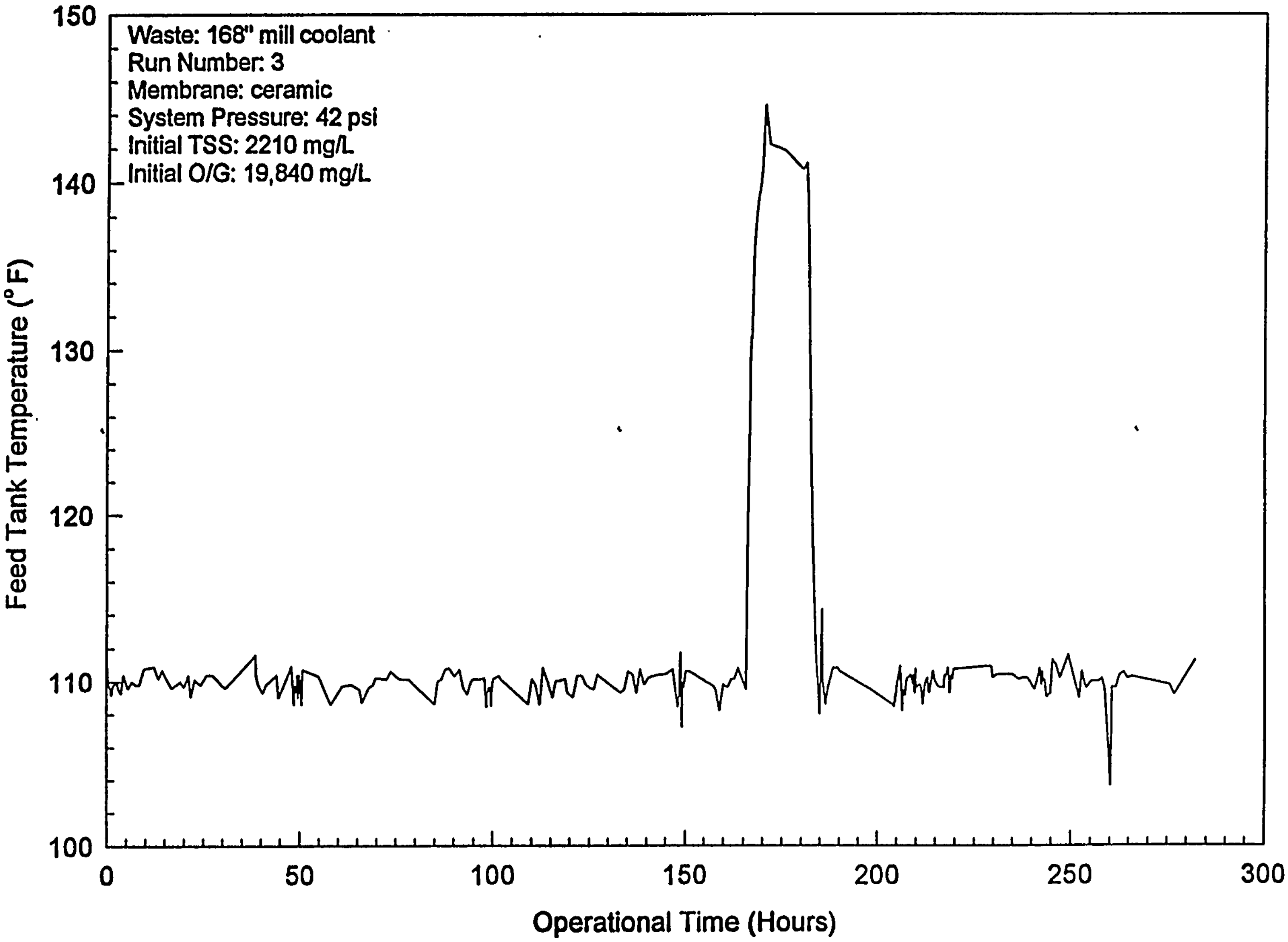

Figure 31. Feed Tank Temperature Versus Time for Run 3-Ceramic Membrane.

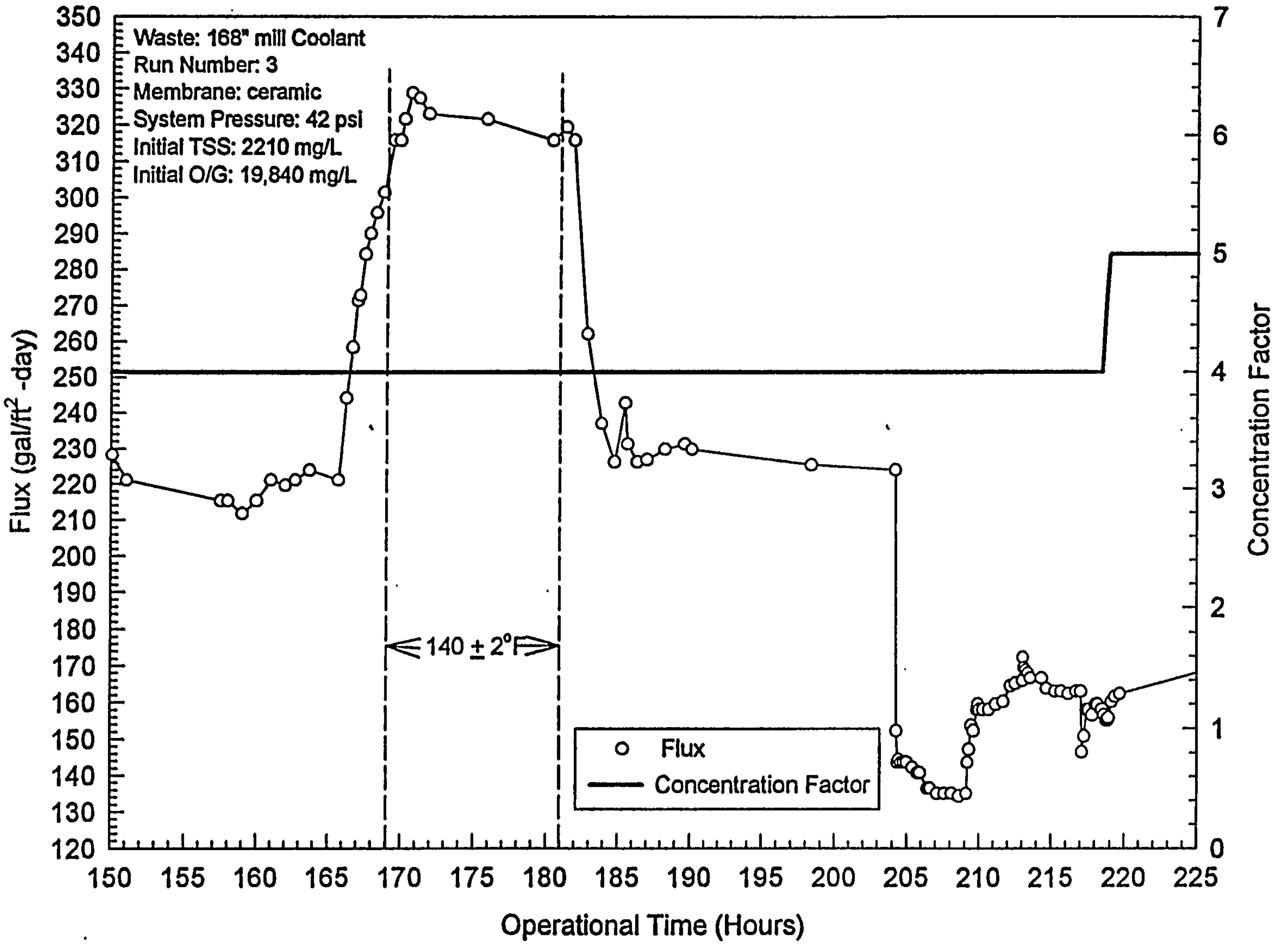

Figure 32. Permeate Flux Versus Time During Temperature and Rotational Excursions

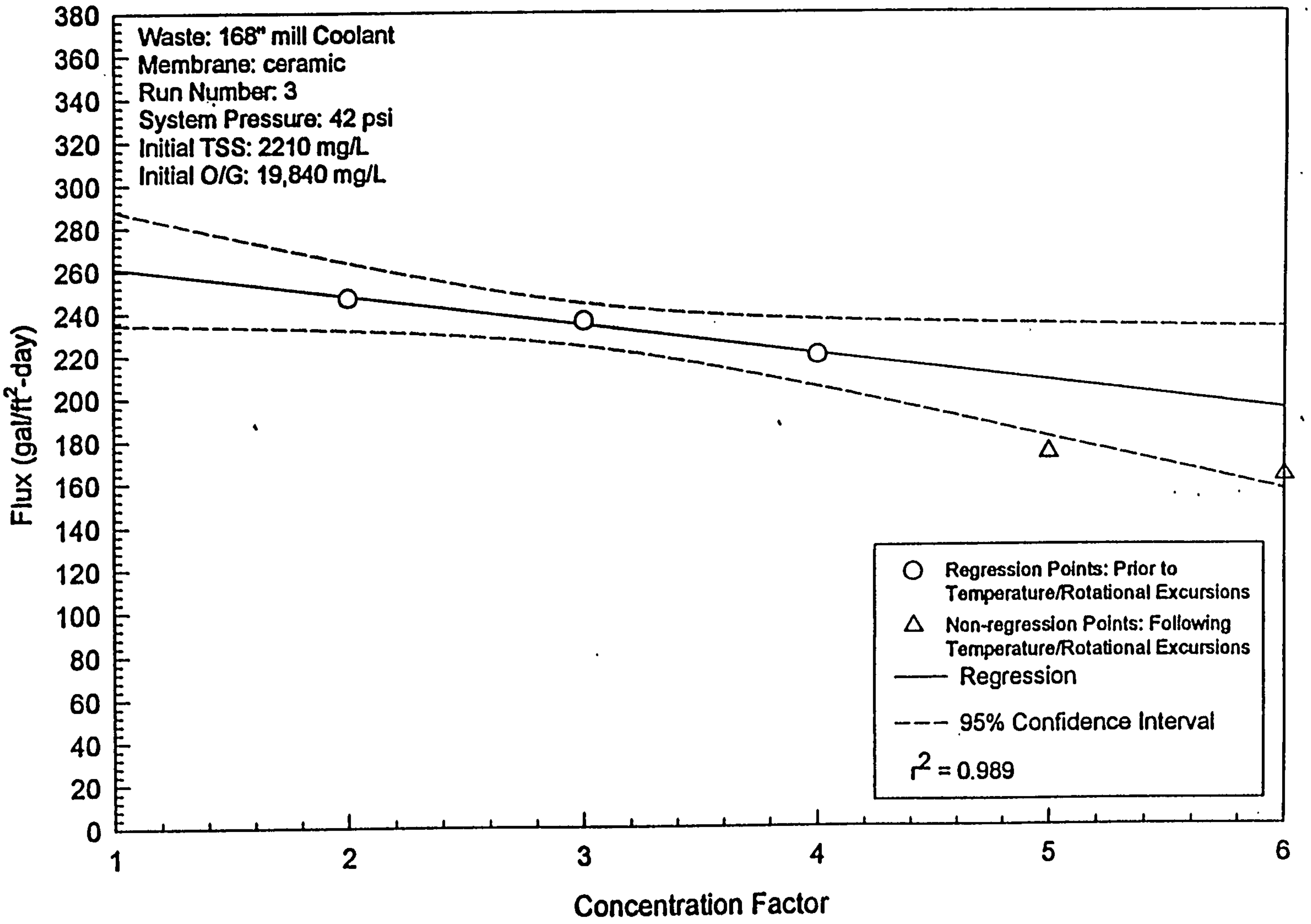

Figure 33. Permeate Flux Versus Concentration Factor for Run 3-Ceramic Membrane.

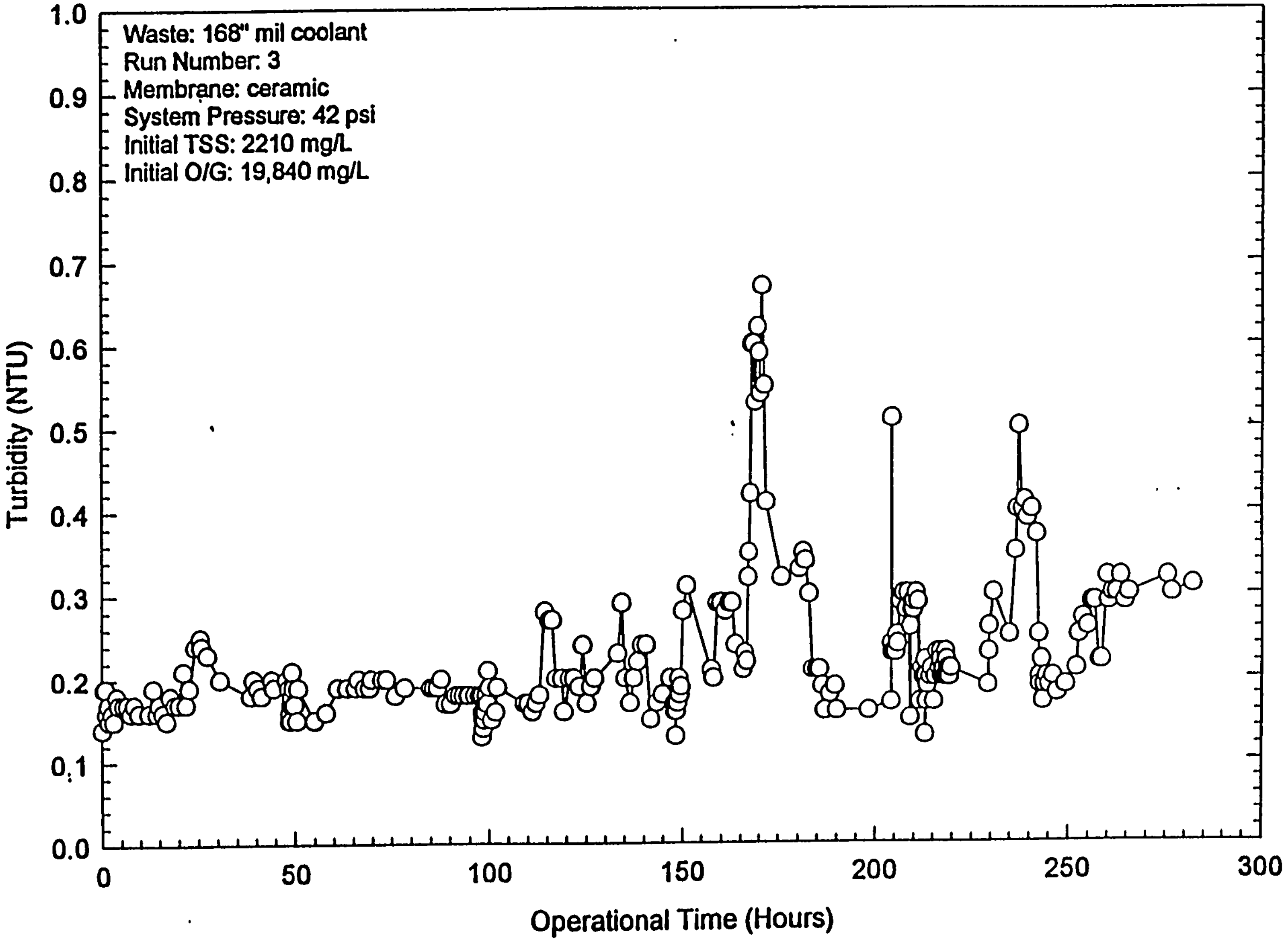

Figure 34. Permeate Turbidity Versus Time for Run 3-Ceramic Membrane.

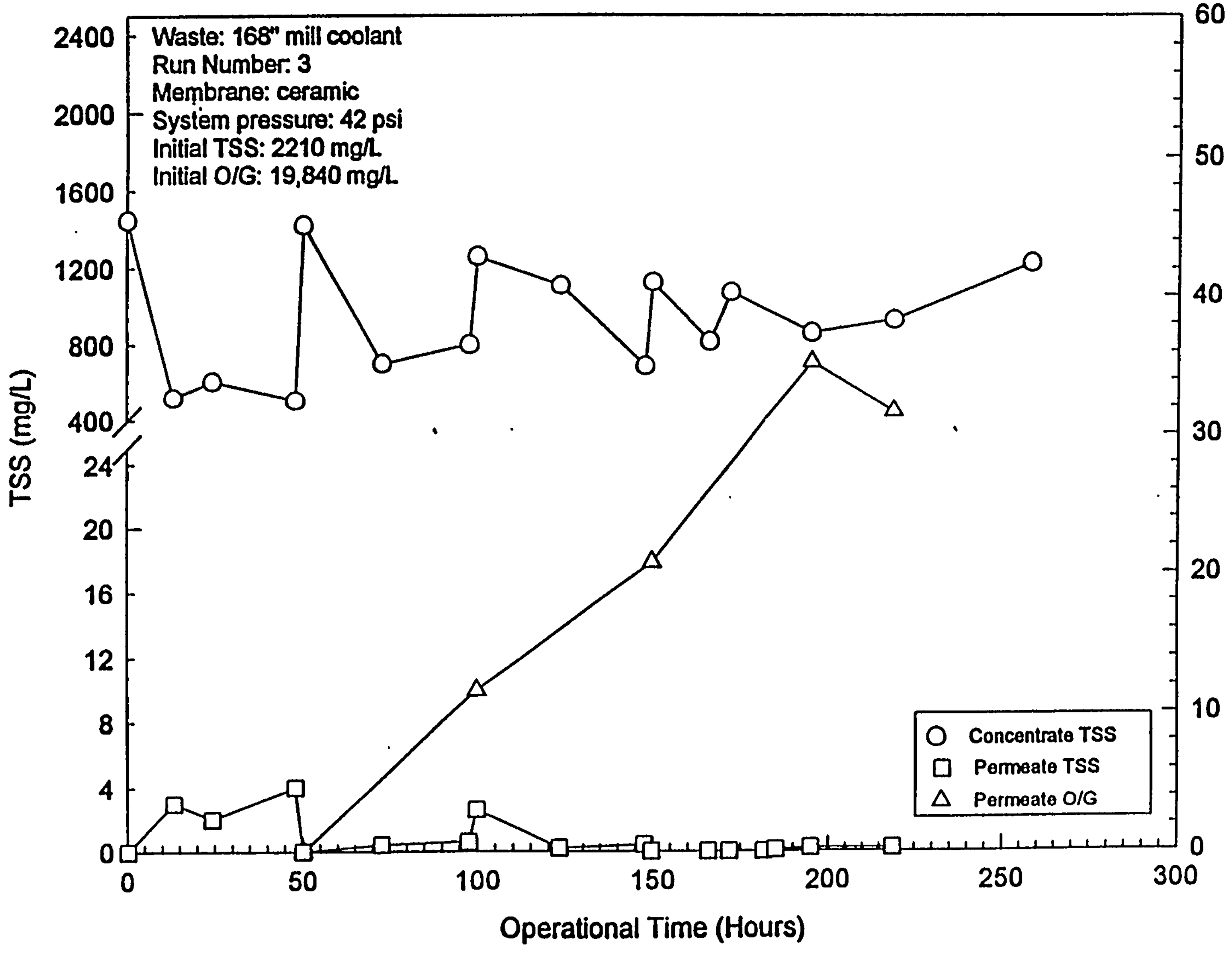

Figure 35. Permeate O/G and TSS Versus Time for Run 3-Ceramic Membrane.

7.0 CONCLUSIONS

Summaries of the permeate flux and water quality results from the six coolant runs are presented in Tables 10 and 11, respectively. Based on data obtained during this portion of the research, it appears that the SpinTek centrifugal system equipped with a ceramic membrane is able to satisfactorly treat a cocentrated oil/grease waste stream. Such a waste stream exists at the RMI facility in Astabula, Ohio. Specific conclusions are presented below.

1. The ceramic membrane is superior to the PV-100K polymeric membrane and should be used in the full-scale centrifugal system. Reasons are as follows: 1) the PV-100K membrane flux was significantly lower than that observed for the ceramic membrane, 2) the maximum operation temperature for the PV-100K membrane is 120°F, compared with >160°F for the ceramic membrane (it is advantageous to run the system at the highest temperature possible as this results in a larger flux), and 3) permanent fouling occurred in all three PV-100K runs indicating membranes would have to be installed frequently in full-scale operation. No permanent fouling was observed for the ceramic membrane.

2. A whitish "cake" was observed on the ceramic membrane when *Biz* detergent was used in the washing solution. The presence of this "cake" decreased the clean water flux substantially. When a pH adjustment step was included in place of *Biz*, the system was effectively cleaned without a loss in the clean water flux. In the full-scale centrifugal system, the following washing solution should be used: 30 mL *Dawn* dishwashing soap/5 gal tap water, pH = 12 (using NaOH or KOH), temperature ≈ 135°F. For the tubular membrane portion of the system, a polymeric membrane will most likely be used thus, the original washing solution recipe (*Biz, Dawn,* 135°F) can be employed. Because waste characteristics can change with time, the washing solution recipe in full-scale operation should be periodically evaluated.

3. For the PV-100K membrane, permeate quality deteriorated significantly during batchdown operation. For the ceramic membrane Runs 2 and 3, there was no difference in permeate quality (turbidity, O/G and TSS) between semibatch operation and batchdown.

4. Increasing the temperature from 110 to 140°F increased the average flux by about 45 percent (220 to 319 gal/ft^2-d). Only a portion (about 24 %) of the increase in flux could be attributed to the decrease in viscosity with increasing temperature. For the ceramic membrane, the maximum operational temperature of the full-scale centrifugal system will most likely be determined by other portions of the system (*e.g.*, fittings). SpinTek personnel should be consulted regarding this issue.

5. Decreasing the rotational speed decreased the permeate flux and the flux did not recover when the rotational speed was increased back to its maximum value (1750 rpm). It is hypothesized that when the rotational speed was initially decreased, the membrane became fouled and subsequent increases in the rotational speed were not able to clean the membrane effectively. Thus, in a full-scale system, the maximum rotational speed should be used.

6. Permeate water quality (turbidity O/G and TSS) were not affected by temperature or rotational speed excursions. Turbidity appears to be a good real-time indicator of permeate TSS and O/G

concentrations. Turbidity and permeate flux can be used to judge when the system should be cleaned.

Table 10. Summary of Permeate Flux Results

Run ID	**Average Flux[1] ± std gal/ft^2-d**			**CWF[2] gal/ft^2-d**	
	Entire Run	Semibatch	Batchdown	Before	After
PV-100K					
Run 1	64 ± 14	73 ± 4.8	50 ± 12	118	118
Run 2	104 ± 44	128 ± 31	61 ± 31	589	172
Run 3	69 ± 18	78 ± 8.5	45 ± 14	718	115
Ceramic					
Run 1	87 ± 30	99 ± 18	61 ± 17	320	159
Run 2	135 ± 23	142 ± 19	103 ± 9	513	431
Run 3	NA	235 ± 13	165 ± 8	431	481

[1] At 110±2°F. [2] Clean Water Flux. Not Applicable.

Table 11. Summary of Permeate Water Quality Results

Parameter	Average ± std			Removal %
	Entire Run	Semibatch	Batchdown	
Turbidity, NTU				
PV-100K				
Run 1	81 ± 182	20 ± 9.3	175 ± 267	NA
Run 2	210 ± 313	90 ± 80	597 ± 459	
Run 3	147 ± 355	4.3 ± 6.1	595 ± 518	
Ceramic				
Run 1	81 ± 181	8.0 ± 6.1	355 ± 255	NA
Run 2	1.21 ± 0.9	1.25 ± 0.96	0.99 v 0.38	
Run 3	0.23 ± 0.09	0.19 ± 0.04	0.27 ± 0.07	
O/G, mg/L				
PV-100K				
Run 1	221 ± 300	98 ± 10	289 ± 363	98.8
Run 2	832 ± 1,172	426 ± 494	1,481 ± 1,684	94.8
Run 3	945 ± 2,632	89 ± 11	2,310 ± 4,132	99.6
Ceramic				
Run 1	187 ± 107	121 ± 13	252 ± 121	99.4
Run 2	37 ± 5	40 ± 3	32 ± 0	99.9
Run 3	20 ± 15	11 ± 10	33 ± 2.6	99.9
TSS, mg/L				
PV-100K				
Run 1	56 ± 63	53 ± 28	57 ± 74	99.3
Run 2	120 ± 111	82 ± 107	161 ± 107	99.0
Run 3	59 ± 114	3.4 ± 3.4	147 ± 151	99.5
Ceramic				
Run 1	12 ± 15	3 ± 2.5	23 ± 17	99.5
Run 2	8.8 ± 8.3	7.7 ± 6.9	15 ± 11	99.7
Run 3	< 1	1.2 ± 1.4	< 1	99.7

NA: Not Applicable

8.0 WORK PLANNED FOR NEXT QUARTER

The following work will be conducted during the fourth quarter: 1) aquistion of oil/grease samples from DOE's RMI facility in Astubula, Ohio and 2) treatment of the same O/G waste using a ceramic membrane.

9.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES

There were no significant technical difficulties.

ENVIRONMENTAL POLLUTION CONTROL DEVICES BASED ON NOVEL FORMS OF CARBON

Report for Period
ending September 30, 1996

Work Performed Under METC Task 2.4
as Part of the WVU-DOE Cooperative Agreement.
Contract No.: DE-FC21-92MC29467

For :
U.S. Department of Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By:
Albert Brennsteiner, John W. Zondlo, Alfred H. Stiller and Yue Xu
West Virginia University, Department of Chemical Engineering
Morgantown, West Virginia 26506

September 1996

DISCLAIMER

ABSTRACT

The use of electrochemistry is an effective means to remove heavy metal contaminants from aqueous effluent streams. This project deals with the development and testing of novel carbon materials for the electrochemical removal of radionuclides and heavy metal ions from aqueous wastes. Both electroplating and electrosorption are employed for this task. The purpose of this work was to develop and test carbonaceous materials for their ability to perform this task. This is accomplished by using a "packed-bed" of the carbon material in a "flow-through" electrochemical remediation cell.

The concentration of heavy metal ions and radionuclides in waste streams is usually very low: typically 1 to 1000 ppm. These low concentrations lead to relatively low current densities and low conversion of electroactive species at electrodes with a small surface area. Therefore, the electrolytic cell must be designed to increase the degree of conversion of the electroactive species (metal ion) as it passes through the remediation cell. This increase in conversion can be obtained by two means: (1) the use of a 3-dimensional electrode (increase surface area and residence time) and (2) turbulence (rotated electrode or flowing stream). The desire is to create a very high ratio of electrode surface area to electrolyte volume within the cell, coupled with turbulent mass transport within the electrode material. In this type of electrode, it is possible to control conversion efficiency using the length of electrode material through which the contaminated waste must pass.

This purpose of this work is to develop and test a cathodic material with an extremely large ratio of surface area to volume. This will be accomplished by using a packed-bed of conductive carbon, either as carbon fibers or carbon foams. Activation of these materials can lead to even greater surface areas. The increased surface area can help minimize the length of electrode through which the solution must pass. This decreased distance leads to reduced resistance to solution flow.

The removal of cadmium, lead, copper, nickel, strontium and uranium from aqueous samples using a small-scale model of the electrolytic cell has been demonstrated. Removal efficiencies above 90% have been observed for the reductive removal of Cd, Pb, Cu and Ni. Removal of U via electrolytic precipitation was 99% effective. The removal of Sr utilized electrosorption and a removal efficiency of 68.0% was obtained using a coating of MnO_2.

The continuous removal of lead was demonstrated over a 72 hour period. The removal of lead over this period was continually greater than 90%. This corresponds to an effluent concentration of less than 10 ppm for an inlet solution feed of 100 ppm.

A scaled-up version of the prototype was constructed and tested to handle even greater flow rates of contaminated wastes. After several modifications, a removal efficiency of 99% was achieved for lead. A "real" sample obtained from the EPA's cleanup process at Fort Benjamin Harrison was treated. 95% removal of lead was achieved for a single pass through the electrolytic cell.

The final objective is to transfer this technology for "on-site" use in industrial applications.

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EXECUTIVE SUMMARY

Novel types of carbon materials have been tested for their ability to remove radionuclides and heavy metal ions from aqueous samples via electroplating and electrosorption. Removal efficiencies above 90% have been obtained for the reductive removal of cadmium, lead and copper. Long-term removal of lead over using electroplating on carbon nanofibers, vapor grown carbon fibers and carbon foam decreased the concentration of a 100 ppm lead feed to less than 10 ppm. Even after 72 hours, the carbon nanofibers showed no sign of saturation. For the 72 hour period, a capacity of 1.025 $g_{Pb}/g_{carbon\ fibers}$ was realized. An even greater capacity is expected at saturation.

Removal via electrosorption using carbon nanofibers produced similar results, but with an early drop-off of efficiency and a much smaller capacity (7.1 X 10^{-4} $g_{Pb}/g_{carbon\ fibers}$ for a 57 hour period). This is due to the neutralization of the charge on the carbon surface caused by the electrosorption of the positively charged metal ions. It is envisioned that for aqueous streams, electrosorption will be applied to remove radionuclides and other metal ions that have reduction potentials more negative than that of water. The removal of radionuclides would decrease the volume of radioactive waste produced from the clean-up of decommissioned nuclear sites.

The recovery of metals was demonstrated using lead ions. The recovered effluent resulted in a 7-fold concentration of lead after reversal of the potential applied to the carbon material. This resulted in a decrease in the volume of lead waste by one-seventh.

The removal of nickel ions from solution directly onto the bare carbon materials was inefficient. By plating a small amount of copper onto the carbon surface prior to use, enhanced removal efficiencies were obtained. At pH 7.0, a removal efficiencies of 89% was achieved.

The electrosorption of strontium was also examined. A removal efficiency of 34.4% was achieved using "pelletized" carbon nanofibers. At pH 10.0 and using a coating of MnO_2, removal efficiencies were improved to 68.0%.

The electrolytic removal of uranium was exceptional. For a inlet feed concentration of 100 ppm uranium, an effluent stream was obtained with uranium concentrations near or below 1 ppm. Long-term removal of uranium was accomplished using the smaller remediation cell. For a 42 hour period, excellent removal of uranium was achieved (99%). Drop-off in removal efficiency was observed beyond 42 hours due to clogging of the carbon material. Recovery and concentration of the uranium waste decreased the volume from 2 liters down to 95 mL and concentrated the uranium to 1543 ppm. This is equivalent to a capacity of 0.15 $g_{uranium}/g_{carbon}$.

A scaled-up version of the prototype was constructed and tested to handle even greater flow rates of contaminated wastes. After several modifications, a removal efficiency of 99% was achieved for lead. A "real" sample obtained from the EPA's cleanup process at Fort Benjamin Harrison was treated. 95% removal of lead was achieved for a single pass through the electrolytic cell.

ELECTROCHEMICAL REMEDIATION OF RADIONUCLIDES AND HEAVY METAL IONS

INTRODUCTION

PURPOSE

The purpose of this research was to assess the feasibility of using novel carbonaceous devices to remove radionuclides and heavy metal contaminants from aqueous streams. This work addresses the fabrication, behavior and analytical utility of these novel carbonaceous materials. Electrochemical techniques were employed to meet this objective.

BACKGROUND

Currently, there exists a rather serious problem associated with several nuclear production and processing facilities in the US. Over the years, large quantities of low-level radioactive waste, some in aqueous solution, have been accumulating at these sites in storage. Moreover, at some sites, this material has already leaked out and contaminated the soil over wide surface areas. In order to attack this problem, the DOE has expressed its desire to investigate new remediation techniques by promoting extremely applied and focused research programs. The work centers on the development of new procedures to remove and concentrate heavy metals and radionuclides from these low-level wastes. Most of the waste is in the form of aqueous solution and the new technique must be capable of reducing the concentration of contaminants down to parts per million (ppm) and in some cases parts per billion (ppb). Since the quantity of these wastes is large, the remediation procedure must be efficient and capable of being scaled-up in a predictable manner.

Industrial contacts have voiced a desire to remove several heavy metals from various aqueous streams. Heavy metal contaminants discussed include nickel, cadmium, copper, lead and chromium. Based on the standard reduction potentials, all of these metals should be excellent candidates for the technique developed in this program.

A technique which can be used to remove metal ions from solution is the adsorption and/or electrochemical reduction of the metal ions. In this procedure, a conductive surface acts as a electrode. A negative potential is applied to the electrode surface. The metal ions in solution are transported to the electrode surface via convection. Upon reaching the electrode surface, the metal ions are adsorbed. If the potential is sufficiently negative the ions will be reduced and plated onto the electrode surface. In either case the metal ions are removed from the aqueous waste. While such a procedure is effective at lowering the concentration of the metallic species on the small scale, application at the large scale has been hindered due to the relatively small surface area of the metallic electrodes. This work assesses the performance of novel carbonaceous materials with large surface areas to remove radionuclides and heavy metal ions and their ability to be scaled up to applicable size.

Electrochemical techniques pertain to measurements involving the flow of electrons during a reduction-oxidation (redox) reaction. These measurements involve current, potential, charge and/or time as the primary variables. Electrochemical studies typically incorporate the use of a three-electrode system. This system includes a working, reference and auxiliary electrode. The redox reaction occurs at the surface of the working electrode as a result of the applied potential. Therefore it is critical to chose a suitable material for the working electrode. To be effective, the material must meet certain requirements. The electrode material should exhibit good electrical conductivity, be chemically inert over the potential region of interest, show favorable electrochemical behavior with the analyte(s), have a low background current and have an easily reproducible surface (1). Carbon materials are well suited for use as working electrodes. Graphite (2,3), carbon paste (4), glassy carbon (5) and reticulated vitreous carbon (RVC) (6) are a few examples of carbonaceous materials that have been used as working electrodes. A detailed discussion of carbon electrodes has been compiled by Dryhurst and McAllister (7).

Carbonaceous materials have been used to produce flow-through porous electrodes. Electrode materials have included graphite chips (8) or granules (9), glassy carbon grains (10), crushed graphite (11), screens of graphite (12) or carbon (13) and RVC (14). These porous flow-through electrodes have been used to remove metal ions from aqueous solutions. This is of major importance to industrial processes and environmental applications. The large surface area of porous flow-through electrodes makes them excellent candidates for the removal of metal ions from solution. Removal efficiencies approaching 100% can be achieved (14).

Discussions with METC personnel have ascertained the identity of several transuranics and radionuclides of concern. These metals include uranium, plutonium, cesium and strontium. Due to their large negative standard reduction potentials and the instability of the solid metals in water, electrosorption will be assessed for their removal. Due to the hazards of working with radioactive species, non-radioactive isotopes or surrogates will be employed. Strontium and uranium can readily be measured using polarography.

Once the metals have been removed from solution and accumulated onto the electrode surface, they can be recovered by reversing the applied potential. To recover selectively the metals, careful control of the applied potential is crucial. By applying a potential sufficiently negative to electroplate only one metal, it can be removed from the stream. Subsequent remediation cells with increasingly more negative potentials can be used in series to remove other metals. Each metal can thus be removed and recovered selectively.

The present work addressed the use of various carbon materials, including carbon fibers and porous carbon foams, to produce porous flow-through electrodes for the removal of metal ions from aqueous media. The resulting effluent was monitored downstream to assess the decrease in analyte concentration and hence the electrode efficiency.

EXPERIMENTAL METHODOLOGY

A schematic diagram of the small-scale apparatus used to carry out these experiments is shown in Figure 1. It was designed in the WVU lab and was produced by the WVU machinist. The carbonaceous material to be tested is placed between a platinum mesh screen and a filter. The platinum mesh is used to establish electrical contact to the carbon material. The purpose of the filter is two-fold. The filter prevents the carbon from flowing out of the cell and acts to distribute the solution evenly throughout the carbon bed. A predetermined potential is applied to the carbon material using a potentiostat. The sample solution is pumped through the carbon material and the unwanted metal contaminants are removed.

For the cathodic removal of metal ions, a potential more negative than the standard reduction potential of the metal ions of interest is applied to the working electrode (see Table I). This applied potential forces the plating of the metals onto the electrode surface thus removing the contaminant metal ions from solution.

Many metals can not be reduced at the electrode surface due to their large negative standard reduction potentials. The problem with these species is that their reduction potentials are more negative than that required to electrolyze water to hydrogen gas (H_2) and hydroxide ions (OH^-). Hence for aqueous solutions, water will be electrolyzed before these metals are electroplated onto the carbon surface. In these cases, electrosorption is utilized. Electrosorption incorporates a negative potential which is more positive than the potential to reduce the metal ion. The positive charge of the metal ion is attracted to the negative charge on the electrode surface. The metal ions are loosely bound to the electrode surface due to electrostatic forces and are removed from the aqueous stream. Due to the neutralization of charge on the electrode surface, lower removal efficiencies are expected. It is anticipated that most radionuclides will be removed by electrosorption since they generally have large negative standard reduction potentials (see Table I, for examples of Sr^{+2} and U^{+2}).

Experimental parameters require optimization to maximize the removal of these metal ions. These parameters include pH, electrolyte concentration, solution flow rate, applied potential and carbon bed thickness. Conversion efficiency, long-term stability and loading capacity must be assessed as well. Finally, the pressure drop and flow rate characteristics of the solution flowing through the carbon bed need to be evaluated.

CARBON SAMPLES

Several carbonaceous electrode materials have been obtained from a variety of sources for comparison. To date, the following samples have been obtained.

- Reticulated Vitreous Carbon (RVC) Foam (Duocel®, 3% density) from Energy Research & Generation, Inc. (ERG, Oakland, CA). 10, 20, 30, 45, 80 and 100 pores per inch (ppi).
- Vapor Grown Carbon Fibers (VGCF) (Pyrograf III, Lot ADNH, 104-107) from Applied Sciences, Inc. (ASI, Cedarville, OH).
- Vapor Grown Carbon Fibers (Pyrograf III, Lot AN-201, "as grown") from Applied Sciences, Inc.
- Vapor Grown Carbon Fibers (Pyrograf III, Lot AN-201, "oxidized") from Applied Sciences, Inc.
- Vapor Grown Carbon Fibers (Pyrograf III, Lot P-2, "pelletized", regular "AN" fiber) from Applied Sciences, Inc.
- "Coal-Derived" Carbon Foam (23 NMP, Sample # 001) from WVU (Morgantown, WV).
- "Coal-Derived" Carbon Foam (23 NMP, Sample # 001, crushed foam, 420-707 μm) from WVU.
- Carbon Nanofibers (#1094-A) from PSU (University Park, PA).
- Carbon Nanofibers (#1094-B) from PSU.
- Carbon Nanofibers (NF46, unsupported) from PSU.

- Graphite Fiber Mats from PSU.
- Carbon Nanofibers grown on Graphite Fiber Mats (#0295) from PSU.
- Graphite Felt (#0695-A) from PSU.
- Carbon Nanofibers grown on Graphite Felt (NF46, F100, CN37 and CF37 from PSU.

RETICULATED VITREOUS CARBON (15)

Reticulated Vitreous Carbon (RVC) is an open-pore material with a honeycomb (foam) structure (14). RVC is a form of carbon that combines some of the properties of glass with normal industrial carbon. RVC has a large void volume (97%) and a small surface area (0.09 m^2/gram). It has a self-supporting rigid structure, a low resistance to solution flow and is resistant to temperatures up to 600 °F in air (16). A more thorough discussion of vitreous carbon is available in the literature (17-21). The samples obtained had various pore sizes. Sizes included 10, 20, 30, 45, 80 and 100 ppi (pores per inch). The 100 ppi RVC foam was tested in our study.

VAPOR GROWN CARBON FIBER (22)

The VGCF manufactured by Applied Sciences, Inc. (ASI) is produced by a combination of catalytic and chemical vapor deposition processes which allow for control of the fiber dimensions, such as filament length and diameter. Lengths are available from 1 mm to tens of centimeters and diameters can vary from under 0.2 µm to over 100 mm. The resulting fiber has a lamellar morphology and is highly graphitic. Cross section and overall views of these samples were provided in the first year's quarterly reports.

"COAL-DERIVED" CARBON FOAM

These particular samples of carbon foam are made from coal-derived pitches produced at WVU by a novel solvent extraction process. By careful control of experimental parameters, an ultra-low ash foam precursor (pitch) is produced which upon careful heat treatment results in a low-density carbon foam structure. This "green" foam is then calcined at temperatures around 1000 °C and a highly conductive material results. The foam has an open-cell structure, enabling it to be used as an efficient carbon electrode.

CARBON NANOFIBERS

The carbon nanofibers were produced by our first year collaborators at The Pennsylvania State University (PSU). The nanofiber material is a microscopic version of a carbon fiber. It is produced by the metal catalyzed decomposition of certain hydrocarbons (23-24). Various degrees of graphitization are possible.

GRAPHITE FIBER MATS and GRAPHITE FELTS

Obtained commercially and supplied to WVU by Dr. Baker at The Pennsylvania State University.

All materials were treated with a dilute solution (10%) of nitric acid prior to use as the electrode material. If necessary to help activate and clean the surface, mild electrochemical of chemical methods were employed. A potential of +1.0 V was applied to the carbon surface for 10 minutes to remove contaminants and produce oxidized sites on the carbon surface. This was followed by applying a potential of -1.0 V for 10 minutes to condition the surface prior to use.

WORK PERFORMED THIS PERIOD

EXPERIMENTAL

Reagents:

Stock solutions of cadmium, lead, copper, uranium, strontium and nickel were prepared using atomic absorption standard solutions (Fisher Scientific, Fair Lawn, NJ) and deionized water. The deionized water used was further purified using a NANOpure™ ultrapure water purification system (Barnstead-Thermolyne, Dubuque, IA). These solutions were stored in polyethylene containers to avoid contamination. The 1.0×10^{-4} M mercury ion solution used for anodic stripping voltammetry was prepared from $Hg(NO_3)_2$ as needed. The mercury ion and sample solutions were prepared in 0.1 M KNO_3 (potassium nitrate) supporting electrolyte. The pH was adjusted using nitric acid. (All chemicals were certified ACS grade purchased from Fisher Scientific unless otherwise noted).

Surface Area Measurements:

The surface area of the samples has been determined and the data are shown in Table II. The surface areas were obtained at WVU using a Gemini III 2375 Surface Area Analyzer (Micrometrics Instrument Corporation, Norcross, GA). Standard operating procedures have been followed as outlined in the operator's manual.

A pre-weighed sample of the carbonaceous material was placed into a clean, dry sample tube. The sample was thoroughly de-gassed using nitrogen. The dewar was filled with liquid nitrogen and the system prepared for analysis.

The sample tube was connected to the analysis port and the balance tube was connected to the balance port. Care was taken to minimize exposure of the sample to air. The doors to the sample station were closed and the analysis initiated. Saturation pressure was set to 740.53 mm Hg and the evacuation rate to 599 mm Hg/min. The Gemini III 2375 performs automated analysis. It is capable of performing simultaneously BET multipoint and single point surface area, Langmuir surface area, t-method micropore and BJH adsorption/desorption distribution. The resulting data were recorded on an Epson plotter (Model P84FA).

Construction of the Small-Scale Remediation Cell:

The small-scale remediation cell (see Figure 1) was produced using a Lexan™ rod (5.0 cm diameter and 6.0 cm long). The flow channel (1.6 cm i.d.) was drilled through the Lexan™ body. A threaded Lexan™ plug was used to press the carbonaceous material between a platinum mesh and a piece of filter paper. The pressure on the carbon material can be easily adjusted by turning the threaded plug. The Ag/AgCl reference electrode (3M NaCl, Model RE-4, BioAnalytical Systems, Inc. (BAS) West Lafayette, IN) was introduced through a Lexan™ sleeve positioned 90 degrees to the flow channel. Leakage was prevented using an o-ring and compression fitting. A piece of coiled platinum wire inserted at the outlet of the cell was used as the auxiliary electrode. Contact to the working electrode was established via a platinum mesh.

Evaluation of Carbonaceous Materials:

The carbonaceous material to be studied was pretreated to clean, oxidize and assist in the wetting of the carbon surface. This was accomplished by placing the carbon material in a dilute solution (10%) of nitric acid overnight in a covered beaker. The material was then thoroughly washed using deionized water. The carbonaceous material was weighed and then positioned into the metal remediation cell using a clean glass rod.

The threaded plug is used to press the carbonaceous material between a platinum mesh and a piece of filter paper. The pressure on the carbon bed can be easily adjusted by turning the threaded plug. Rigid samples, such as RVC, were positioned using a snug fit. Electrical contact to the working electrode was established using the platinum mesh. The reference electrode was inserted into the sleeve and all three electrodes were connected to the potentiostat. The cell was connected to the pump using Tygon® tubing and a 0.1 M KNO_3 electrolyte solution was pumped through the flow cell. In order to help remove air bubbles and wet the electrode surface, a mild electrochemical pretreatment was used, The remediation cell was held at +1.00 V for 10 minutes followed by -1.00 V for 10 minutes.

The performance of the carbonaceous material under study was tested by placing a solution containing a known concentration of metal ions into the sample reservoir. The solution was then allowed to pass through the remediation cell at a known flow rate and applied potential. The potential of the remediation cell was controlled using a Power Module (Model PWR-3, BAS). The performance was assessed by measuring the metal ion concentration in the effluent and comparing it to the initial ion concentration. The percentage efficiency of the remediation cell is equal to the percentage of metal ions removed from the solution by the remediation cell. (For these studies, the effluent was collected in Nalgene® bottles for subsequent analysis via voltammetry and/or atomic absorption).

Construction of the Scaled-Up Remediation Cell:

Construction of the 3-inch prototype remediation cell has been completed. A schematic of the apparatus is shown in Figure 2. The assembled system includes a flowmeter to measure flow rates and a manometer to measure the pressure drop across the remediation cell. The remediation cell was produced by machining a 4-inch Lexan™ rod. The 3-inch diameter flow channel was drilled through the Lexan™ body. Via the threaded plug, the depth of the carbon electrode bed can be varied between 0.5 and 4.0 inches. Electrical contact was established in the same manner used in the smaller cell. The prototype has been assembled and parametric studies have begun. Several problems were encountered with this model (see Results and Discussion).

A redesigned version of the remediation cell (Figure 3) was designed and constructed to compensate for the problems associated with the above cell. This new galvanic cell utilizes a two electrode system. The anode reaction was separate from the cathode reaction using a porous ceramic cylinder(0.2 μm pores). The inner chamber contained the carbonaceous material (97.5 grams, WVU foam for this study) where the removal of metal ions takes place. The outer chamber contained the anolyte (anode-electrolyte) solution that was constantly replaced. This anolyte solution contained the species that is oxidized at the anode. The electrode system was enclosed in a Plexiglas® tube. A solution flow rate of 10.0 mL/min was used for the sample and a flow rate of 60 mL/min was used for the anolyte solution. The pH of the lab prepared solutions was adjusted to 3.5 using nitric aced prior to testing. The sample obtained from the Fort Benjamin Harrison Site was pumped through the cell without any prior treatment. The Ft. Ben Harrison sample had a pH of 4.7 and a constant plating current of 60 mA was employed.

ANALYTICAL METHODS

Anodic Stripping Voltammetry:

The thin-layer flow cell was purchased from BAS (Model LC-44-01000). The working electrode was glassy carbon disk (0.3 cm diameter) over which the solution flows. Two 50 μm spacers were used to produce the flow channel and direct the sample solution across the carbon disk. Prior to use, the glassy carbon disk was polished using successive slurries of 1.0, 0.3 and 0.05 μm alumina until a mirror-like surface was obtained. The glassy carbon disk was then rinsed thoroughly with deionized water and assembled. All potentials were measured relative to the Ag/AgCl reference electrode (BAS, Model RE-4). Stripping voltammograms at the thin-layer flow cell were obtained using the BAS Model CV-27 Voltammograph in conjunction with a Hewlett Packard Model 7044B X-Y Recorder.

Polarography:

Polarographic data was obtained using a Static Mercury Drop Electrode (SMDE) (Model 303A, EG&G Instruments, Princeton Applied Research, Princeton, NJ) in conjunction with a potentiostat (Model 263A, EG&G) and interface (Model 507, EG&G). This system has been received and has been assembled. Upon assembly and calibration, this instrument was utilized to measure the uranium ion concentrations in the effluent stream. (The 100 ppm solution being tested is below the detectable limits of Atomic Absorption. Polarography can be used to measure uranium concentrations down to 20 ppb). The SMDE can be used to measure many other metals in the ppb to ppm range.

Atomic Absorption:

Spectroscopic data were obtained using an Atomic Absorption Spectrophotometer (Model 2380, Perkin-Elmer, Norwalk, CT). Procedure and experimental conditions were outlined in the Perkin-Elmer "Analytical Methods of Atomic Absorption Spectrophotometry" Cookbook. All standards and samples were matrix-matched prior to analysis.

RESULTS and DISCUSSIONS

Several carbonaceous materials were examined for their performance as the cathode material. Each sample was tested to determine the effect of adsorption (no applied potential) and electroplating. Their performance was evaluated on their ability to remove the metal ions from solution.

To confirm minimal contribution from the platinum mesh to the metal removal, the system was first tested in the absence of any carbonaceous material. The response of the original feed solution was compared to the response of the effluent collected at -1.00 V. It was shown that the platinum mesh does not contribute significantly (<2%) to the removal of the metal ions. Therefore, the removal of the metal ions is due to their interaction with the carbonaceous material and is not significantly influenced by the platinum mesh.

In order to assess electrosorption, the carbon electrodes were tested for their ability to remove cadmium and lead at potentials more positive than that required to reduce the metal ions. The removal of cadmium was examined at various potentials above and below its theoretical reduction potential. Based on the initial cadmium feed concentration (100 ppb), the potential at which cadmium should be reduced is -0.58 V. Electrosorption removed 46% of the cadmium from the solution at -0.20 V and 78% at 0.40 V. At potentials negative to reduce the metal ions, electroplating removes 89% at 0.60 V, 91% at 0.80 V and nearly all (99%) at -1.00 V.

The removal of lead was conducted under the same conditions. The theoretical reduction potential for lead is -0.54 V. Removal of lead via electrosorption is shown also to occur at potentials more positive than the reduction potential. The removal efficiencies were 88% at -0.20 V and 92% at -0.04 V.

Continuous removal of lead from an aqueous stream using electroplating and electrosorption was used to demonstrate the long-term effectiveness of carbon nanofibers. The lead solution was passed through the remediation cell and the ions were removed by applying the desired potential to the carbon nanofibers. A potential of -1.00 V was applied to the remediation cell to remove lead ions via electroplating. Over a 72 hour period, removal efficiencies of 89% or greater were achieved for an inlet concentration of 100 ppm lead. Calculation of the lead removed over this period demonstrates a capacity of 1.025 $g_{Pb}/g_{carbon\ fibers}$, but it should be stressed that even at 72 hours, the carbon showed no signs of saturation (see Figure 4).

Removal of lead ions via electrosorption was carried out using a potential of -0.40 V. For an inlet lead ion concentration of 100 ppb, good removal efficiencies (90%) were obtained for the first 12 hours (see Figure 5). After that time a noticeable decrease in efficiency is evident. The removal efficiency drops to less than 50% after 57 hours. This decrease in removal efficiency is expected due to the neutralization of charge on the electrode surface. Regeneration of the carbon nanofibers would be required to maintain high removal efficiencies. For the first 12 hours of removal a capacity of 1.9 X 10^{-4} $g_{Pb}/g_{carbon\ fibers}$ is obtained. At 57 hours this capacity increases to 7.1 X 10^{-4} $g_{Pb}/g_{carbon\ fibers}$. Electrosorption of lead using a 100 ppm lead feed was attempted. Due to the smaller capacity of the carbon nanofibers when using electrosorption, the efficiency should drop off very quickly. As expected, within the first two hours the removal efficiency dropped to less than 20%.

The final desire is to recover the metals once they have been removed from the aqueous stream. It is possible to recover the metal ions plated on the electrode simply by reversing the potential. The metal ion concentration in the resulting effluent is greater than that of the feed. Electrodeposition and the subsequent recovery from carbon nanofibers was demonstrated with a 100 ppm lead ion feed. The lead ions were removed from the aqueous stream using an applied potential of -1.00 V. Subsequent recovery of the lead at an applied potential of +1.00 V, produced an effluent stream more concentrated in lead. The lead concentration in the recovered effluent was 730 ppm, demonstrating a 7:1 concentration of the waste stream. Individual metal ions can be removed and recovered selectively by these means.

A summary of the electrodeposition removal efficiencies for the carbon samples tested to date are shown in Table III. These results demonstrate that carbonaceous materials are extremely effective for the removal of heavy metal ions from solution.

Industrial Application:

Contact has been established with Paul Brezovec at Concurrent Technologies Center (CTC), Johnstown, PA. It has been proposed that the electrochemical method developed in this research be applied to their nickel-plating line for the removal and recovery of nickel. The final rinse stage produces an aqueous waste contaminated with nickel. The objective is to purify the rinse water for reuse and to recover the nickel for recycling back into the plating process.

Contact has also been made with Dr. Dave Szlag of the Environmental Protection Agency (EPA) in Cincinnati. The desire is to remove lead from aqueous solution obtained by washing lead from the contaminated soil on military firing ranges.

A local plating industry, Swanson Plating Company (Morgantown, WV) has been identified as a source of "real" samples for testing the remediation cell. Identification of the metals and acquisition of samples is ongoing.

Removal of nickel at the experimental conditions used for lead and cadmium resulted in low removal efficiencies. Due to the unique chemistry of nickel, careful attention must be given to increase the removal. Table IV demonstrates the need for tailoring the removal process for nickel. By applying a more negative potential (i.e. -1.60 V) enhanced removal efficiencies are obtained (97.9%). But at this potential the water is reduced and current is wasted due to the production of hydrogen gas. By simply pre-plating a small layer of copper or mercury, enhanced removal efficiencies are obtained. The removal efficiency is also dependent upon the pH of the contaminant solution. For a 100 ppm nickel solution at pH 7.0, a small layer of pre-plated copper and a remediation cell potential of -1.20 V, 89.1% of the nickel is removed.

Radionuclides:

Two metals have been identified that need to be removed from aqueous wastes generated at several nuclear production and processing facilities: strontium and uranium. Both of these metals are available in non-radioactive isotopes. Strontium can be easily measured by atomic absorption at all levels under study. Atomic absorption can not be used to determine uranium concentrations below 100 ppm. Therefore, polarography was used to facilitate the measurement of uranium. Polarography can determine uranium concentrations between 20 ppb and 500 ppm. The "static mercury drop electrode" used for polarography was received this quarter and has been assemble and tested.

No removal of strontium was observed using the untreated WVU foam. This was expected due to the relatively low surface area (0.5 m^2/g) of the "non-activated" carbon foam. Activation of the foam surface could increase the surface area to values necessary for the electrosorption of strontium and such an activation apparatus is in preparation.

The improvement in removal efficiencies using the higher surface area carbon fibers is shown in Table V. Removal of strontium using carbon nanofibers resulted in 5.90 % of the strontium to be removed. Usage of the ASI carbon fibers improved this removal to 34.4%. This improvement is most likely due to differences in the surface properties of the two carbon fibers. Alternative removal schemes, similar to those used for nickel have been examined to improve the removal of strontium from the aqueous waste. A coating of MnO_2 on the carbon surface improved the removal efficiency in a pH 11.0 solution to 68.0%.

The removal of uranium was examined using ASI carbon nanofibers. No removal was observed for adsorption alone (no applied potential). Upon applying a potential of -1.00 V, almost immediate removal of uranium is seen. Long-term removal of uranium was examined over a 48 hour period. Within the first hour the uranium concentration drops well below 5 ppm and it was not until the carbon material began to clog that any appreciable amount of uranium passed through. Even at 47 hours, when the solution flow has noticeably slowed, the effluent concentration only rose to 16.3 ppm. This large removal is believed to be caused by electrochemically-induced precipitation of the uranium oxide. Results for the long-term removal of uranium are shown in Figure 6.

Prototype Remediation Cell:

Several problems became apparent with the 3-inch diameter prototype electrolytic cell. Due to the increased distance between the electrodes and the large amount of carbon material used, a very large potential drop was experienced. This large potential drop was sufficient to prohibit the potential on the carbon material from becoming adequately negative to reduce the metal ions. Also the diameter of the carbon bed was increased but the depth of the bed was not. Even though more carbon cathode was used, the residence time in the cathode material was not increased. An improvement would be to increase the diameter less while increasing the depth of the bed. Also it was believed that the surface area of the auxiliary electrode was insufficient to handle the current necessary to reduce the metal ions. Therefore, a redesigned electrolytic cell was developed to address these problems. The new cell utilizes a two electrode system. The cathode is the carbon material used to remove the metals and the anode was a coil of platinum wire isolated from the cathode using the porous ceramic tube. Further development will be examined to use a form of carbon as the anode in order to minimize economic factors. A flow rate of 10 mL/min was used in the initial tests with this new cell design. Removal efficiencies for lead of lab generated samples was 99%. This is comparable to those obtained using the small remediation cell with a 10-fold increase in throughput.

A sample was provide by Dr. Szlag of the EPA from the lead solution generated by the treatment of contaminated soil at the Fort Benjamin Harrison Site. The concentration of lead in the sample as received was determined to be 279 ppm. Following a single pass through the scaled-up version of the electrolytic cell and with no sample pretreatment, 95% of the lead was removed. This corresponds to an effluent lead concentration of 14 ppm.

CONCLUSION

All proposed objectives have been met to date (see Table VI). An extensive search of the literature has been conducted in the areas of heavy metal removal, waste water treatment and water remediation. More than 150 papers have been amassed in these areas with special interest in the areas of electrochemistry and electrowinning using carbonaceous material. Further effort has been exerted in order to find applications of this technology to radionuclides.

The performance of the commercially available RVC as a cathode was satisfactory. Improved removal efficiencies were obtain with the novel carbon materials utilized in this study. Loose carbon nanofibers with a relatively large surface area (200 m^2/g) effectively removed over 90% of cadmium and lead from an aqueous sample. Vapor Grown Carbon Fibers supplied by ASI also worked well for the removal of cadmium and lead by electroplating. The "coal-derived" foam produced in the WVU laboratory was an excellent cathode material. The concentration of lead in the effluent dropped below detectable amounts after only 4 hours. This demonstrated removal efficiencies approaching 100%.

The current industrial application of this technology was for the removal of nickel from a rinse solution used in the plating industry. Upon selection of the proper experimental conditions, a removal efficiency of 89.1% was obtained. This was accomplished by adjusting the pH of the waste stream and by pre-plating a small amount of copper onto the carbon surface to help catalyze the reduction of nickel.

The removal of strontium was demonstrated using carbon nanofibers. Removal efficiencies up to 34.4% were obtained, but removal capacity was soon reached. Other removal schemes with similar metal reaction mechanisms to those used for nickel are being examined. The use of a coating of MnO_2, resulted in a removal efficiency of 68.0%.

The removal of uranium was examined using ASI "oxidized" nanofibers. Preliminary work demonstrated high removal efficiencies due to the production of a precipitate on the electrode surface. Such large removal efficiencies are due to electrically-induced precipitation. The 48-hour-long study of uranium removal demonstrated excellent results until the remediation cell clogged. Prior to clogging, maximum removal efficiencies were obtained (99%). Reduction of the volume of uranium waste was demonstrated by decreasing the waste volume from 2000 mL to 95 mL, and concentrating the uranium from 100 ppm to 1543 ppm. This demonstrates a large capacity of 0.15 $g_{uranium}/g_{carbon}$. The use of the 3-inch remediation cell and larger quantities of carbon will allow for the treatment of larger volumes of low level radioactive wastes.

The utility of this technique has been demonstrated on actual samples obtained from the cleanup of lead from soil at Fort Benjamin Harrison. The aqueous waste was generated by washing the lead from the contaminated soil with acetic acid. The decontaminated soil was returned to the site as clean fill.

The aqueous waste generated was contaminated with lead (279 ppm). This aqueous waste was purified using the electrolytic cell to remove 95% of the lead.

WORK PLANNED for NEXT PERIOD

In the next quarter, the following work will be conducted,

- Continue to improve the design of the scaled-up remediation cell.
- The removal of uranium will be further studied to determine the mechanism of removal.
- Test the remediation cell's ability to remove nickel from "REAL" industrial samples.
- Conduct parametric studies: pH, flow rate, etc.
- Develop recommendations for the scale-up of the remediation cell to handle even larger volumes of wastes generated by industry.

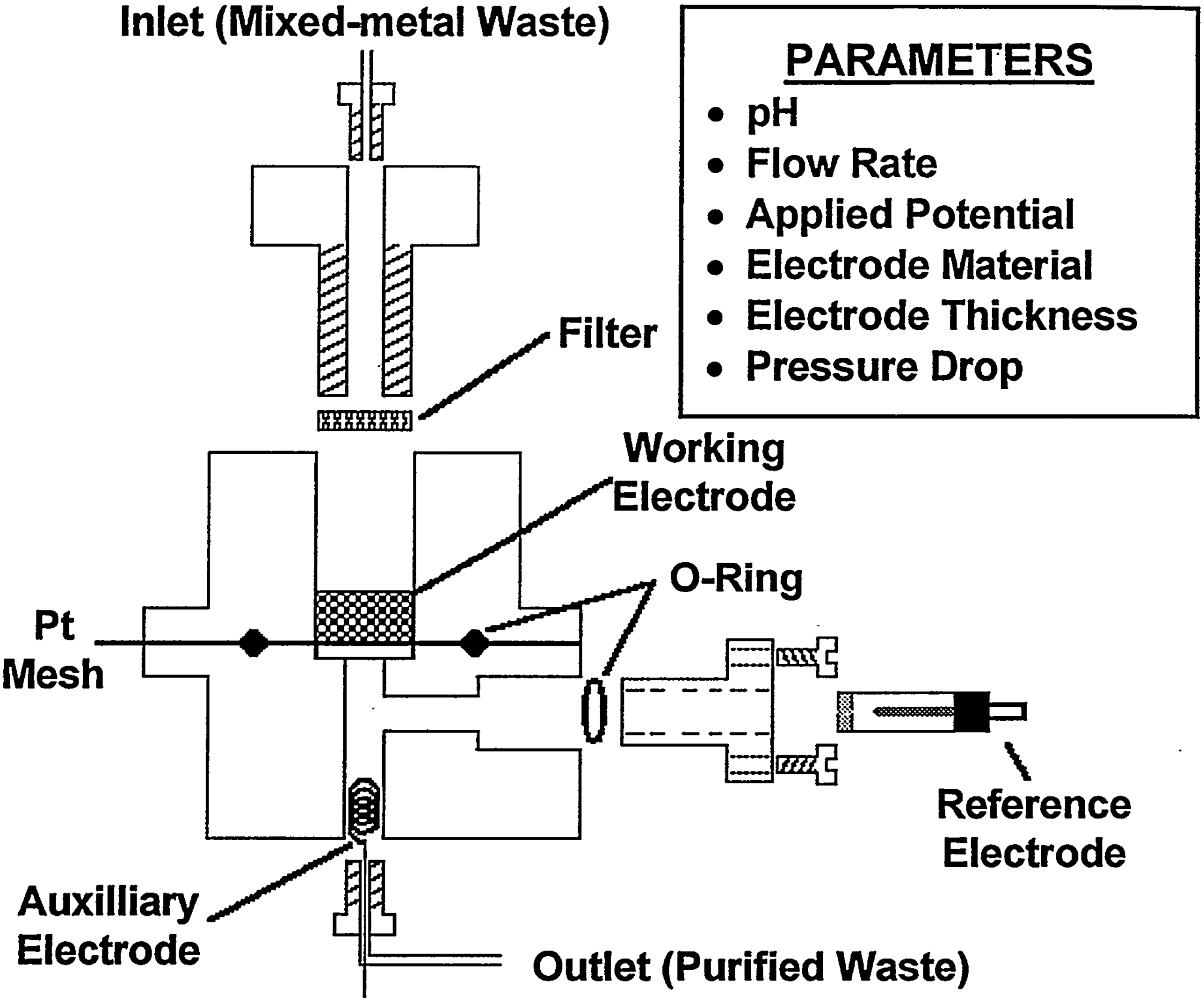


Figure 1. Schematic of Electrochemical Remediation Cell.

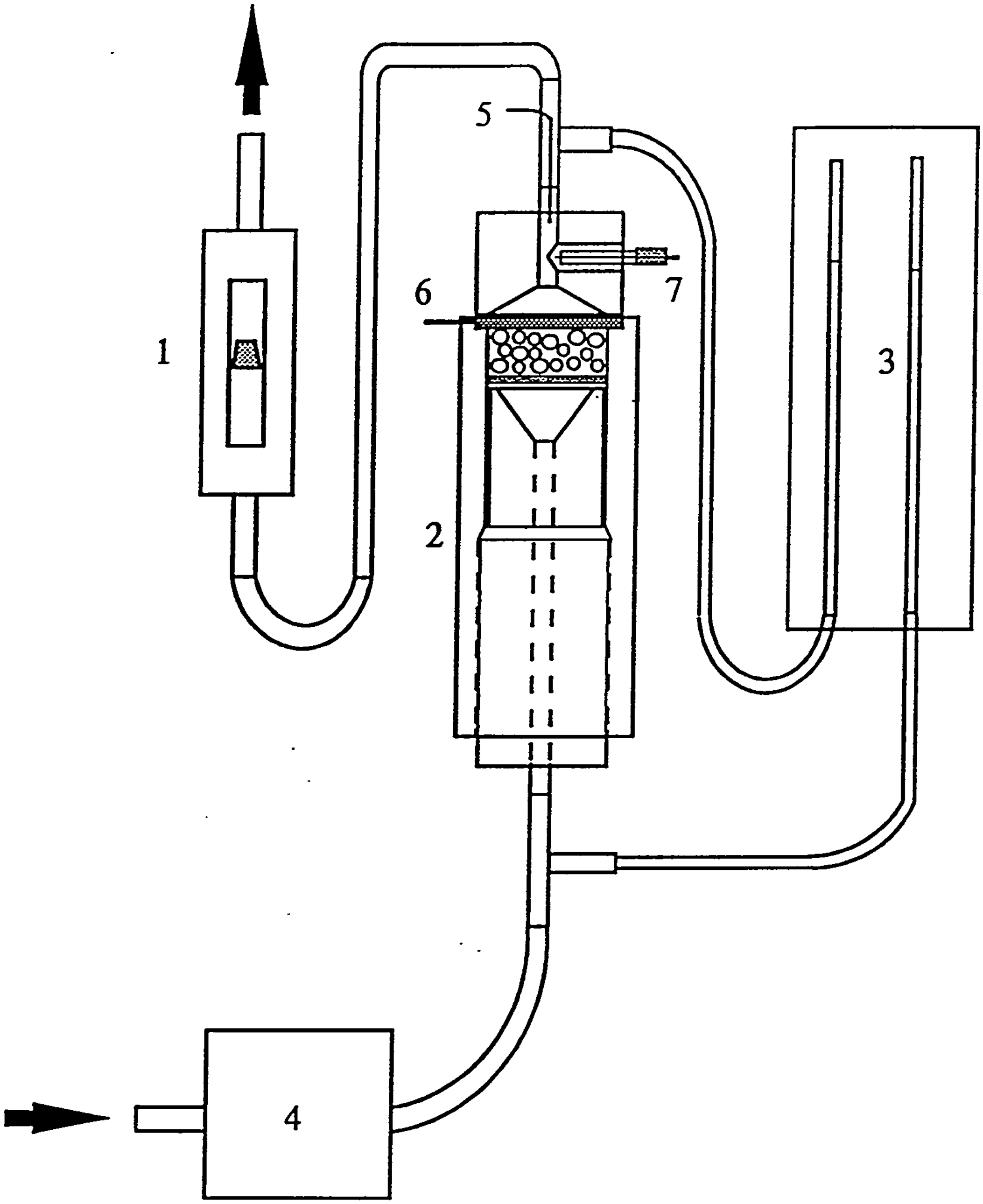


Figure 2. **Schematic of Scaled-Up 3-inch Remediation Cell.**
(1-Flowmeter; 2-Cell; 3-Manometer; 4-Pump; 5- Auxiliary Electrode; 6-Pt Contact to Working Electrode; 7-Reference Electrode.)

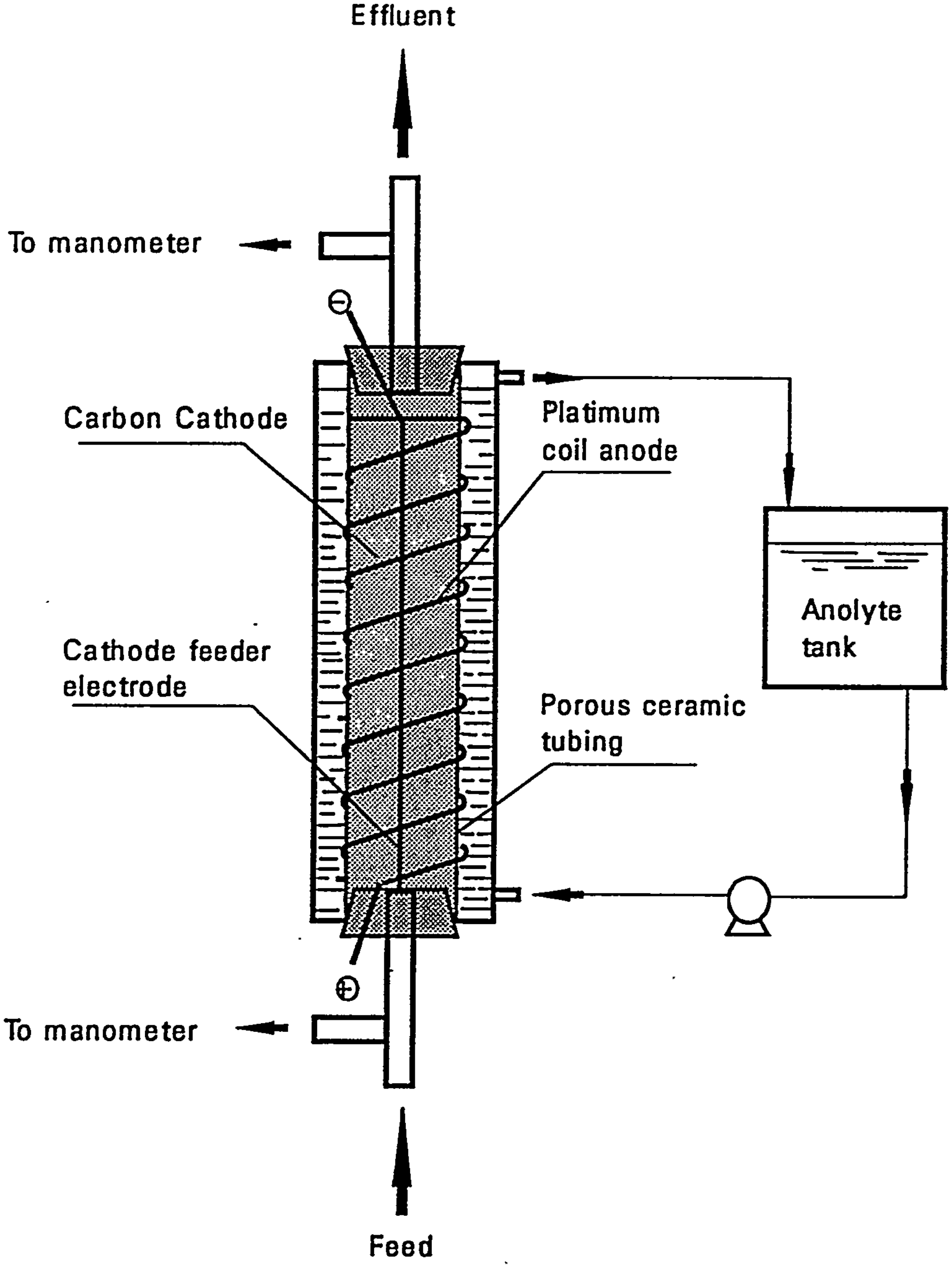
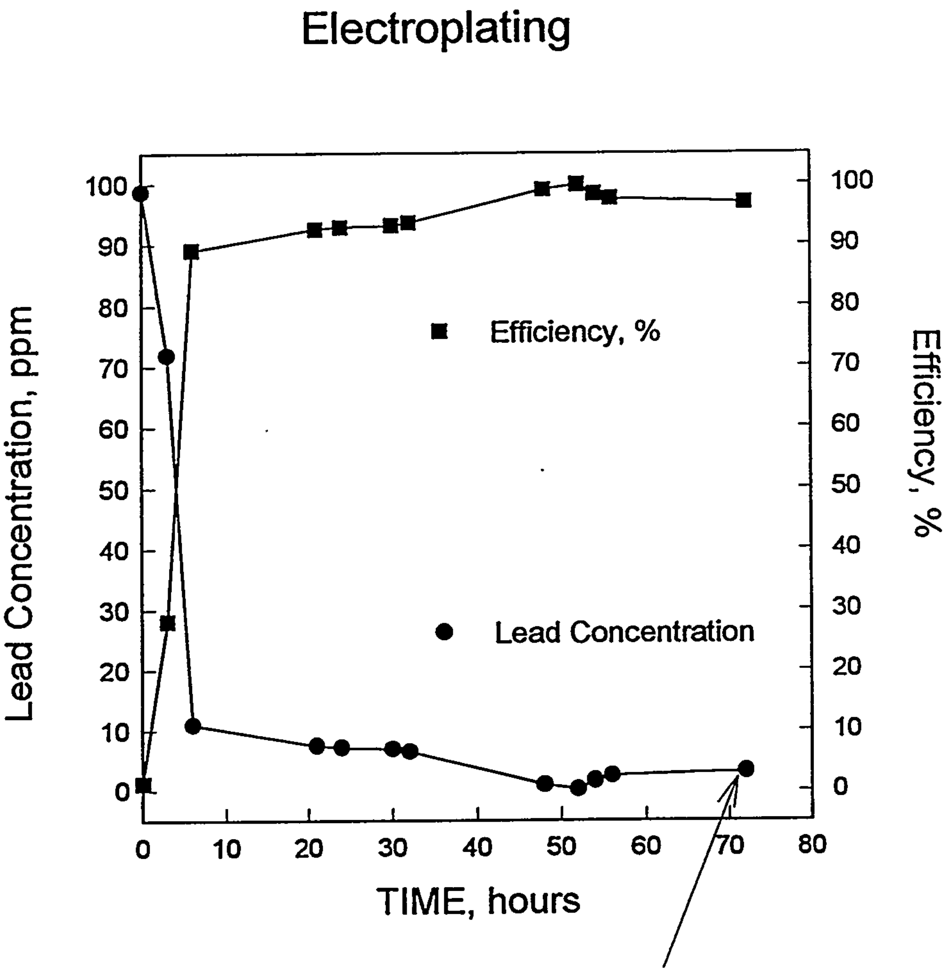


Figure 3. Schematic of 1.5-inch Galvanic Remediation Cell.



Capacity @ 72 hours > 1.025 grams Pb/ gram of carbon
(***NOT at Saturation***)

Flow Rate = 3.1 mL/min/(gram carbon)

Figure 4. Long-Term (72 hr) Removal of Lead Using Carbon Nanofibers. (E_{CELL} = -1.0 V)

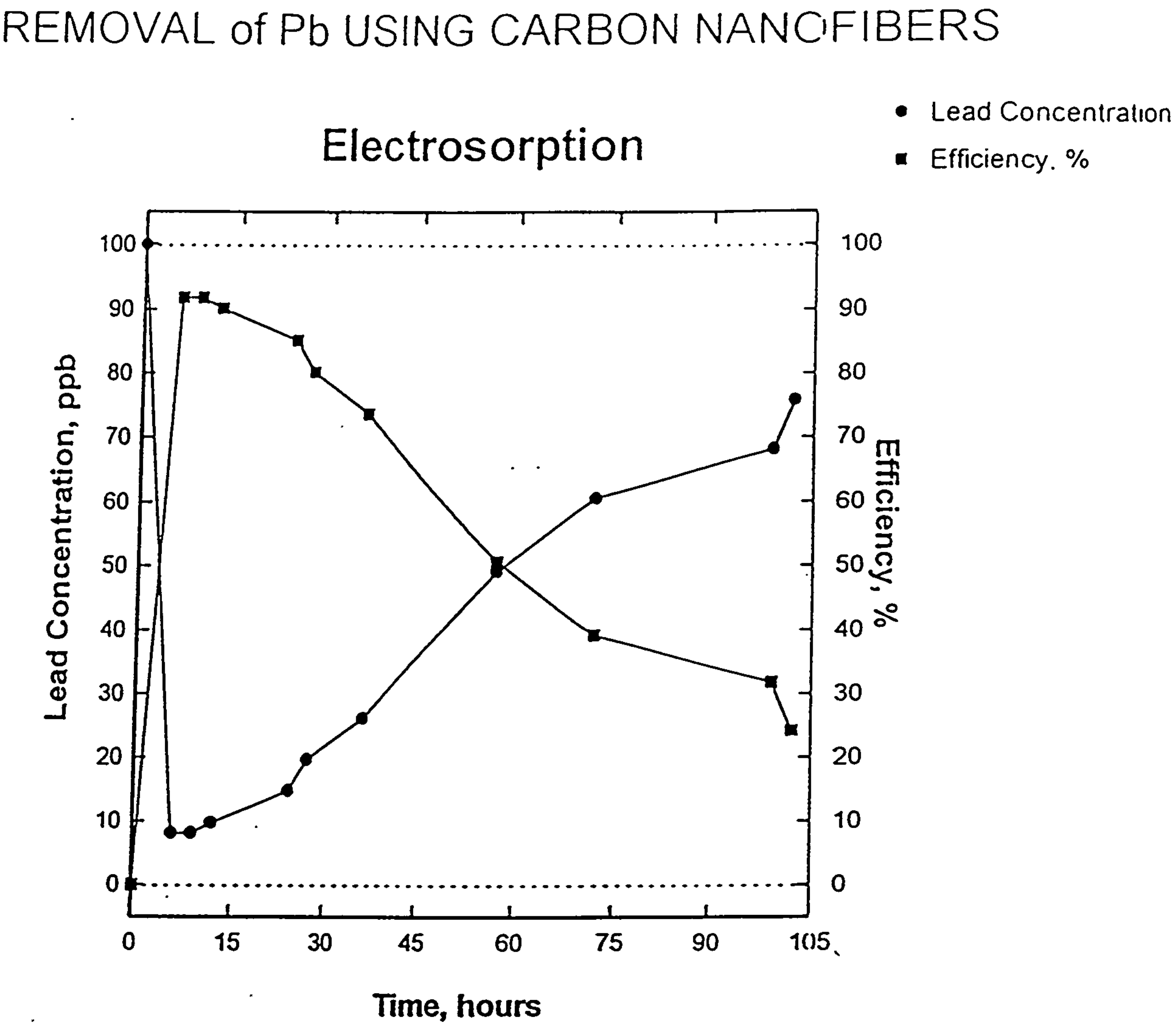


Figure 5. Long-Term (102 hr) Removal of Lead via Electrosorption.
(Conditions: E_{CELL} = -0.40 V)

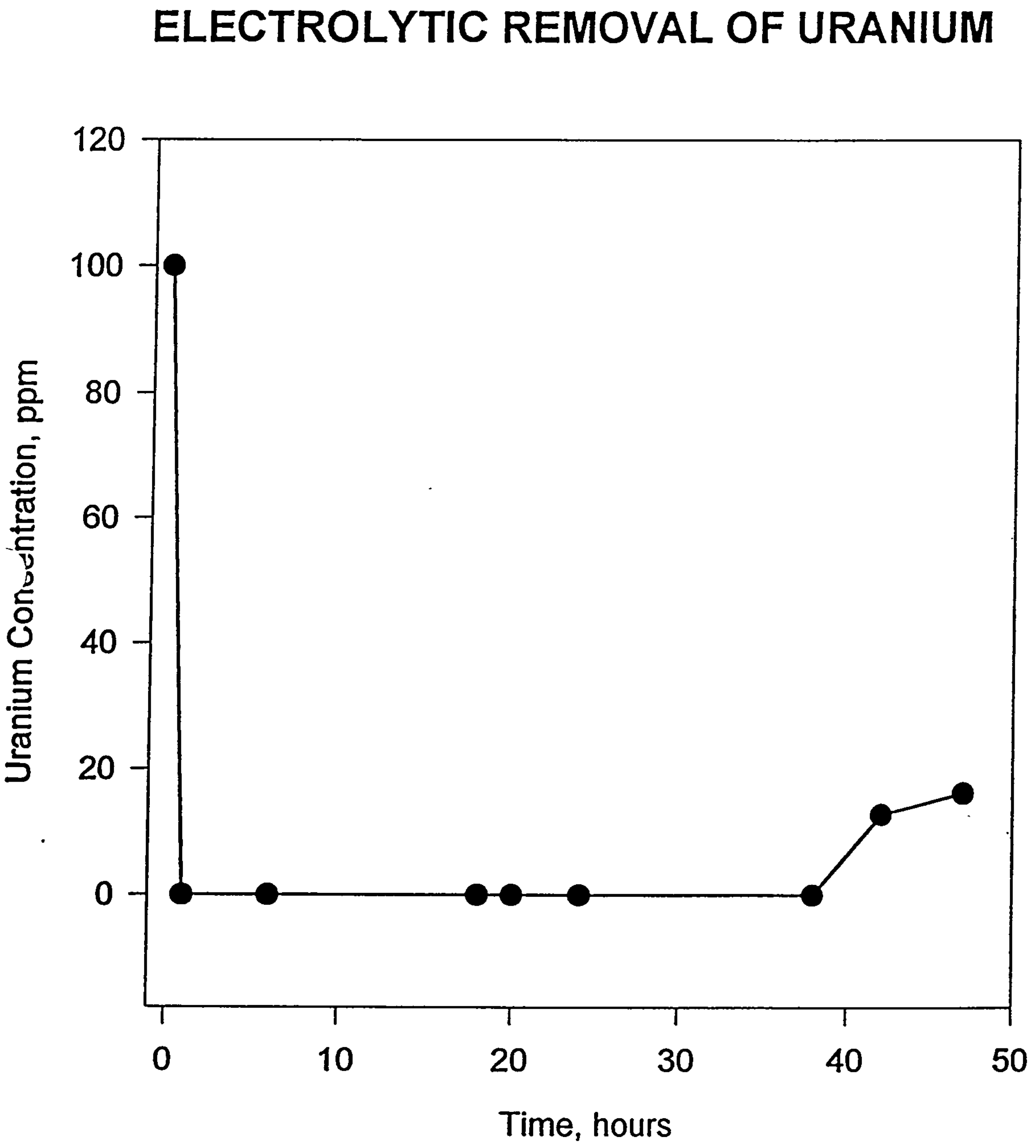


Figure 6. Long-Term 48 hr) Removal of Uranium.
(Conditions: same, E_{CELL} = -1.00 V)

TABLE I: STANDARD REDUCTION POTENTIALS OF THE METAL IONS OF INTEREST

Reduction Reaction	E, V (vs. Ag/AgCl)
ELECTROSORPTION	
Sr^{+2} (aq) + $2e^-$ → Sr (s)	-3.108
U^{+2} (aq) + $2e^-$ → U (s)	-2.022
$2H_2O$ (l) + $2e^-$ → H_2(g)+ $2OH^-$ (aq)	-1.050
ELECTROPLATING	
Cd^{+2} (aq) + $2e^-$ → Cd (s)	-0.624
Ni^{+2} (aq) + $2e^-$ → Ni (s)	-0.453
Pb^{+2} (aq) + $2e^-$ → Pb (s)	-0.348
Cu^{+2} (aq) + $2e^-$ → Cu(s)	+0.115

TABLE II: PROPERTIES of CARBON MATERIALS

Surface Areas

Material	Surface Area (m^2/gram) BET	Surface Area (m^2/gram) Langmuir
Vapor Grown Carbon Fibers (ASI)		
Regular "AN" Fiber	76.78 (73.6)*	122.66
Oxidized "AN" Fiber	25.78	-
Pelletized "AN" Fiber	20.25 (20.77)*	-
WVU "coal-derived" Foam (23 NMP)	0.52	-
Carbon Nanofibers		
1094-A	115.13 (227.8)*	186.78
1094-B	182.77 (192.3)*	286.03
NF-46	43.28	-
Graphite Fiber Mats	0.59	-
Graphite Mat with Fibers	99.92 (112.8)*	-
Graphite Felt	0.17	-
Graphite Felt with Fibers		
NF46	55.41	-
F100	54.54	-
CF37	13.96	-
CN37	35.31	-
VGCF	76.78 (73.6)*	122.66
RVC		
100 ppi	0.092	-
80 ppi	44.80	-
Activated Carbon	784.32	-

TABLE III: Removal Efficiencies for Cd and Pb

Cathode Material	Cathode Potential, V	Metal Removed	
		Cd	Pb
Platinum Mesh	0.00	0%	0%
	-1.00	0%	9%
RVC	0.00	16%	0%
	-1.00	50%	8%
Nanofibers (1094-A)	0.00	0%	0%
	-1.00	91%	95%
Nanofibers on Graphite Mats	0.00	0%	0%
	-1.00	90%	92%
Graphite Felt	0.00	13%	7%
	-1.00	22%	97%
Nanofibers on Graphite Felt	0.00	0%	0%
	-1.00	46%	99%
ASI Carbon Fibers			
"pelletized"	0.00	-	0%
	-1.00	-	98%
"oxidized"	0.00	-	0%
	-1.00	-	99%
WVU "*Coal-Derived*" Foam			
"crushed"	0.00	-	0%
	-1.00	-	99%

TABLE IV: IMMEDIATE INDUSTRIAL APPLICATION

(REMOVAL OF NICKEL VIA ELECTROPLATING)

$2H_2O\ (l) + 2e^- \rightarrow H_2\ (g) + 2\ OH^-\ (aq)$ E° = -1.050 V

$Ni^{+2}\ (aq) + 2e^- \rightarrow Ni\ (s)$ E° = -0.453 V

MATERIAL	SURFACE CONDITION	pH	E_{CELL}, Volts	TIME, hours	% REMOVED
WVU Foams	bare-crushed	3.5	-1.2	2	0.00
		7.0	-1.2	2	0.00
			-1.4	2	68.7
			-1.6	2	**97.9**
	w/ Cu	3.5	-1.2	2	49.0
				4	57.3
		7.0	-1.2	2	**82.4**
				4	**89.1**
	w/ Hg	3.5	-1.2	2	44.3
				4	47.6
				6	50.6

TABLE V: DOE APPLICATION

REMOVAL OF STRONTIUM VIA ELECTROSORPTION

Sr^{+2} (aq) + $2e^-$ → Sr (s) E° = -3. 108V

$2H_2O$ (l) + $2e^-$ → H_2 (g) + 2 OH^- (aq) E° = -1.050 V

MATERIAL	CONDITION	pH	E_{CELL}, Volts	TIME, hours	% REMOVED
Nanofibers	bare	3.5	-1.00	2	5.90
				4	5.60
			-1.20	2	5.70
				4	3.20
WVU Foams	bare	3.5	-1.20	2	0.00
		7.0	-1.20	2	0.00
ASI	bare	7.0	-1.20	0.5	**34.4**
Nanofibers					
"pelletized"		11	-1.20	0.5	**52.8**
	MnO_2	11	-1.20	0.5	**68.0%**

TABLE VI: PROJECT COMPLETION

ENVIRONMENTAL POLLUTION CONTROL DEVICES BASED ON NOVEL FORMS of CARBON

Task #	WSB Code	Task Name	Duration	% Complete
1	1.0	Identify Surrogates for Radionuclides.	JAN	100%
2	1.1	Provide List of Surrogates.	FEB	100%
3	2.0	Characterize Carbonaceous Materials.	JAN-OCT	95%
4	3.0	Use Small Remediation Cell to Test Other Carbonaceous Materials.	JAN-OCT	95%
5	3.1	Assess Carbon Material's Ability to Remove Heavy Metals and Radionuclides	JAN-DEC	75%
6	4.0	Construct 3-inch Remediation Cell	JAN-APR	95%
7	4.1	3-inch Remediation Cell Constructed	APR	95%
8	4.2	Engineering Parametric Studies on 3-inch Cell	APR-DEC	20%
9	4.3	Derive Correlations	JUN-DEC	10%
10	5.0	Test Metal Removal and Recovery Schemes	JAN-DEC	50%
11	6.0	Obtain "REAL" Samples from Industry.	FEB-DEC	80%
12	6.1	Test Removal Schemes Using Small Cell on "REAL" Samples.	JAN-DEC	50%
13	6.2	Apply 3-inch Prototype to "REAL" Samples	AUG-DEC	25%
14	7.0	Analysis and Reporting		
15	7.1	Analysis and Data Reduction	JAN-DEC	60%
16	7.2	Reports and Manuscripts	JAN-DEC	75%
17	8.0	Closure	DEC	
18	9.0	Overall Work Complete	DEC	

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DEVELOPMENT OF INSTRUMENTAL METHODS FOR CHARACTERIZATION AND ANALYSIS OF NUCLEAR WASTES AND ENVIRONMENTAL CONTAMINANTS

METC Task No. 2.5

Quarterly Technical Progress Report
Reporting Period: 1 July 1996 - 27 September 1996

Work Performed Under Contract
No.: DE-FC21-92MC29467

for:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

by:
Dr. Vincent T. Remcho
Department of Chemistry
Post Office Box 6045
West Virginia University
Morgantown, WV 26506-6045

DISCLAIMER

ABSTRACT

Technetium 99 (^{99}Tc) and Iodine 129 (^{129}I), common constituents of nuclear wastes at Hanford, Washington, and other DoE sites, are important target analytes in both the tank wastes themselves and in site aquifers. Characterization and chemical speciation of both of these radionuclides in tank wastes and aquifers therefore represents an important DoE research goal. The work described herein focuses on the development of new analytical strategies for the chemical speciation and quantitation of ^{99}Tc and ^{129}I in simulated nuclear waste tank and aquifer matrices. These tasks are being accomplished through the development of new methodologies in the area of capillary electrokinetic separations. Capillary zone electrophoresis (CZE) and ion exchange capillary electrokinetic chromatography (CEC) methods are being developed for each of the target ions. The CZE studies center on the determination of optimum separation parameters including: selection of operating buffer composition and ionic strength, optimization of buffer pH, selection of operating potential and injection conditions, and development of detection strategies (including UV, indirect fluorescence, and radiometric detection). The CEC studies focus on the development of packed capillary columns for ion exchange separations. This approach should provide a higher analyte loading capacity and therefore improve detection limits. Here, the novel detection strategies employed will include indirect fluorescence detection for the determination of small quantities of ^{99}Tc and ^{129}I, and radiometric detection for the analysis of ^{99}Tc. It is expected that these developments will lead to rugged, reproducible, cost-effective means for determining ^{99}Tc and ^{129}I forms and levels in various matrices.

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EXECUTIVE SUMMARY

The focus of this research effort is the analysis of technetium 99 (^{99}Tc) and iodine 129 (^{129}I), common constituents of nuclear wastes at Hanford, Washington, and other DoE sites. These species are important target analytes in both the tank wastes themselves and in site aquifers. Characterization and chemical speciation of both of these radionuclides in tank wastes and aquifers therefore represents an important DoE research goal.

This work focuses on the development of new analytical strategies for the chemical speciation and quantitation of ^{99}Tc and ^{129}I in simulated nuclear waste tank and aquifer matrices. Capillary zone electrophoresis (CZE) and capillary electrokinetic chromatography (CEC) methods are being developed for each of the target ions. The CZE studies center on the determination of optimum separation parameters including: selection of operating buffer composition and ionic strength, optimization of buffer pH, selection of operating potential and injection conditions, and development of detection strategies (including UV, indirect fluorescence, and radiometric detection). In the first quarter, a reliable CZE method for separation of iodide (I^-) and iodate (IO_3^-) was developed. In this the second quarter, a detector was developed for the indirect fluorescence measurement of iodide, iodate, and perrhenate (ReO_4^-) [a surrogate for pertechnetate (TcO_4^-)], and the CZE method previously developed was extended to include perrhenate.

Later in the project (in the third and fourth quarters of this first project year), the CEC studies will focus on the development of novel packed capillaries (rather than open tubular, self-assembled monolayer modified capillaries) for anion exchange separations. This approach should provide even higher loading capacities and therefore vastly improved detection limits. Here, the novel detection strategies employed will include indirect fluorescence detection for the determination of small quantities of ^{99}Tc and ^{129}I, and radiometric detection for the analysis of ^{99}Tc.

It is expected that these developments in the field of microelectrokinetic separations will lead to rugged, reproducible, cost-effective means for determining ^{99}Tc and ^{129}I forms and levels in various matrices. Ultimately, these newly developed tools will be applied in the analysis of other target analytes.

1.0 INTRODUCTION

The development of several new tools for chemical analysis is proposed for the qualitative and quantitative determination of two important target analytes in Hanford site tank wastes and in site aquifers. Innovations in capillary zone electrophoresis and open tubular capillary electrokinetic chromatography will yield powerful methods for the separation of ^{99}Tc and ^{129}I from other components of simulated tank wastes and aquifer matrices. The use of novel detection strategies, including indirect fluorescence detection and radiometric detection, with the two separation methods will provide for the determination of trace quantities of ^{99}Tc and ^{129}I in small (pL to nL) volume samples, thus limiting human exposure to hazardous wastes. These advanced technology tools are expected to have commercial value, as evidenced by the interest and support of two firms which are active in the environmental analysis and remediation industry: Selective Environmental Technologies, Inc. (Selentec), a small business headquartered in Atlanta, GA; and Battelle Northwest, a research institute and US Department of Energy Contractor with offices in Richland, WA. The interest of Selentec in the project is indicated by the attached letter of endorsement. Battelle's interest is indicated by the provision of laboratory facilities, equipment, and personnel in several stages of the project. The support of these two entities bodes well for the success of the project and for the possibility of transfer of the technology to the marketplace after sufficient development.

2.0 PURPOSE

Technetium 99 (^{99}Tc) and Iodine 129 (^{129}I), common constituents of nuclear wastes at Hanford, Washington, and other DoE sites, are important target analytes in both the tank wastes themselves and in site aquifers. [1] Characterization and chemical speciation of both of these radionuclides in tank wastes and aquifers therefore represents an important DoE research goal. The work proposed herein has as its purpose the development of new analytical strategies for the chemical speciation and quantitation of ^{99}Tc and ^{129}I in simulated nuclear waste tank and aquifer matrices. These tasks will be accomplished through the development of new methodologies in the area of capillary electrokinetic separations (both capillary zone electrophoresis (CZE) and capillary electrokinetic chromatography (CEC)) and the use of novel detection strategies for the determination of small quantities of ^{99}Tc and ^{129}I. It is expected that these developments will lead to a rugged, reproducible, cost-effective means for determining ^{99}Tc and ^{129}I forms and levels in various matrices.

The proposed studies address DoE needs in the focus area of *Crosscutting Technologies and Supporting Programs*, specifically in the sub areas of both *Innovative Technologies/Supporting Programs* and *Industry Programs.*

This project will foster the development of a strong research partnership between the US Department of Energy's Pacific Northwest Laboratory and the Department of Chemistry at West Virginia University. Prof. Remcho's research program in microscale separations will make a unique contribution to the US DoE effort to measure, remediate, contain, and monitor wastes produced at the Hanford Reservation over the past half-century. This contribution will be facilitated and augmented through Dr. Ballou's research program at Battelle-Northwest. Dr. Ballou has directed environmentally relevant research projects for the US DoE for over 20 years, and his unique expertise and laboratory facilities will both support the project and ensure a consistent focus on Hanford needs. This collaborative approach offers the following benefits:

1. the infusion of new ideas and technology into the Hanford waste cleanup effort;
2. an enhancement of West Virginia University's research effort in environmental analysis and remediation;
3. reduced research and development costs for DoE; and
4. the development of a strong and mutually beneficial bond between the US DoE's Pacific Northwest Laboratory and West Virginia University.

The high efficiency and high peak capacity typical of capillary electrokinetic separation techniques [2], coupled with the high selectivity of radiometric detection and high sensitivity of indirect fluorescence detection, will provide a powerful set of tools for characterization and chemical speciation of Hanford waste tank constituents. The technology developed in this initial research effort will be extended and applied to other analytes and matrices in follow-on studies.

3.0 BACKGROUND

Instrumental methods are being developed for the determination of ^{99}Tc and ^{129}I in simulated nuclear waste tank and aquifer matrices. The use of simulated matrices in method development provides a wide margin of safety by minimizing/eliminating exposure to radioisotopes and by eliminating the need to transport radioactive species. The use of simulants also allows for rapid method development because of the increased simplicity of working with a non-radioactive, well-defined matrix. These combined benefits result in savings in both time and expense.

The target analytes, iodine and technetium, are primarily present in solution as iodide, iodate, and pertechnetate anions. These anions are the focus of the initial research. In the interest of safety and practicality, a surrogate stable isotope of iodine(^{127}I) is being employed in developing both CZE and CEC methods for iodine determination. ^{127}I, end product of the mass 127 decay chain, is also a component of tank waste and is present in a concentration similar to that of ^{129}I. ^{99}Tc will be handled in trace quantities using the unique facilities and expertise available at Battelle-Northwest.

4.0 METHODOLOGY

The separation technologies to be employed in conducting the work include both CZE and CEC. CZE is conducted by introducing a small (~10 nL) sample into the open end of a 50 to 100 μm ID fused silica capillary, about 50 cm in length, whose ends are immersed in buffer-filled reservoirs. The sample is introduced either electrokinetically, by applying ca. 50 V/cm for 2-10 seconds; or hydrodynamically, by applying 3-10 psi for 2-10 seconds, while the inlet buffer reservoir is replaced with the sample reservoir. A potential gradient of 100-500 V/cm is then applied along the axis of the capillary, resulting in the separation of analytes according to differences in their charge and size. In addition to the electrophoretic movement of analytes induced by their charge, species are swept along the capillary via electroosmosis: bulk transport of solution in the capillary. Electroosmosis arises from the electrochemical double layer at the charged surface of the capillary when shear forces are generated by application of the high potential. Detection of the separated analyte zones is usually achieved using a UV detector, with a segment of the capillary itself serving as the optical flow cell. Here, the polymeric coating of the fused silica capillary is abraded or burned to produce a clear fused silica surface with excellent UV and visible optical transmission characteristics.

CEC is a promising new technique for achieving microscale separations. [3] Essentially, CEC represents the intersection of capillary electrophoresis and open tubular liquid chromatography. As such, it provides the unique benefits of each of these two techniques while overcoming some of the limitations of each. CEC is conducted in either packed or open fused silica capillaries (~10 to ~ 150 μm ID). The stationary phase may be either covalently attached to the fused silica capillary or formed as a self-assembled monolayer of small molecules (for open tubular CEC) or immobilized on a solid support particle which is then packed into the capillary (conventional CEC). In all other respects, the instrumentation employed in CEC is identical to that used in CZE.

The detection strategies employed will include: 1) UV detection for development of a CZE method for the determination of iodine; 2) indirect fluorescence detection for sensitivity optimization in the determination of iodine and technetium by both CZE and CEC; and 3) radiometric detection for the determination of ^{99}Tc by both CZE and CEC.

5.0 WORK PERFORMED THIS PERIOD

5.1 Development of CEC Methods for Analysis and Speciation.

0.01 M stock solutions of NaI, $NaIO_3$, and $NaReO_4$ (Sigma Chemical Co., St. Louis, MO, USA)were prepared in distilled, deionized water for use in method development. Serial dilutions of these were made in the appropriate buffer solutions as necessary for each stage of the experimental work. Sodium phosphate was obtained from Sigma Chemical Co. (St. Louis, MO, USA). The 5 mM pH 2.6 mobile phase was prepared by dissolving sodium phosphate in distilled deionized water. Solutions of the test probes were prepared in distilled deionized water. Unless otherwise noted, 75 μm ID fused silica capillary tubing (Polymicro Technologies, Phoenix, AZ, USA), 365 μm in OD, was used in all of the work described herein. All work was carried out using an ATI-Unicam (Boston, MA, USA) Crystal 310 CE system. Separations were conducted with the anode at the outlet reservoir due to the fact that $\mu_{ep} > \mu_{eof}$ for all of the analytes studied.

5.1.1 Analysis and Speciation of Ionic Iodine Species.

Work in this period focused on the separation of the iodine target species. ^{129}I and ^{127}I are most often present in solution as iodide (I^-) and/or iodate (IO_3^-). It was found that these two species were easily separable by ion exchange capillary electrokinetic chromatography in standard operating buffer solutions. This technique entailed the axial application of a 100 to 500 V/cm potential gradient to the separation capillary using a high voltage power supply. The platinum electrodes used in this method were immersed into two small reservoirs filled with aqueous buffer solutions, one at each end of the fused silica capillary in which the separation is conducted. A window for optical detection was burned near an end of the capillary using a fiber optic fusion splicer (Alcoa-Fujikura, Atlanta, GA, USA). UV detection was employed in initial studies. Both I^- and IO_3^- absorb in the low UV (~200 nm), allowing for non-radioactive (^{127}I) iodide and iodate to be determined by CZE-UV when working at moderately high analyte concentrations.

5.1.2 Analysis and Speciation of Ionic Rhenium and Technetium Species.

Technetium is most often present in solution as pertechnetate (TcO_4^-) ion. Since no stable isotope of technetium exists, the close and non-radioactive chemical homolog of technetium, rhenium, is being used initially to significantly simplify the experimentation.

Perrhenate (ReO_4^-) ion is being used to determine appropriate operating and detection conditions. These conditions will then be validated first with standard solutions of ^{99}Tc and then with simulated tank waste solutions containing ^{99}Tc. ^{99}Tc is a beta emitter, and thus should be detectable at relatively low levels using a commercially available Raytest Steffi™ radiometric detector (Raytest Inc., Wilmington, DE). [4] This detector has been delivered, and will be used in the fourth quarter of the project period at the Pacific Northwest National Laboratory (work planned for the third quarter was postponed as the detector was modified to provide optimum sensitivity for the weak β particles emitted by ^{99}Tc). Use of this sensitive, highly selective detector will minimize exposure to radioactive sources and should provide the trace level detection capability necessary for determination of ^{99}Tc in tank waste simulant and aquifer samples.

Initial work on perrhenate analysis has begun. Detection is by UV-Vis at 190 nm and buffer solutions are as described in section 5.1.1, above. This should allow for analysis of iodide, iodate, and perrhenate in a single step.

5.2 Development of Ion Exchange CEC for Analysis of Rhenium, Technetium, and Iodine.

5.2.1 Packing of Capillary Columns.

Work in this quarter largely focused on the development of an ion exchange CEC prrotocol. The first step in the development process is the production of capillary columns packed with an ion exchange sorbent. In accomplishing this goal, fused silica capillaries (Polymicro Technologies Incorporated, Phoenix, AZ, USA) with inner diameters of 75 µm were cut to a length of 60 cm. Frits were installed in the outlet end of the column as follows:

- 0.01 g silica packing material (Macherey-Nagel, D-5160 Duren, Germany) was mixed with 100 µl water glass (The PQ Corporation Valley Forge, PA, USA) and 10 µl formamide (Aldrich Chemical Company, Inc. Milwaukee, WI, USA), producing a homogeneous mixture;

- the capillary was tapped end downward into the mixture, thus forcing some of the mixture into the end of the capillary;

- once the capillary was filled to about 1 mm, the mixture was sintered into place by aligning the non-sintered frit with the electrodes of an arc fusion splicer (Fujikura, Japan) and heating the mixture for 1 second;

- after sintering, the capillary was baked in an oven for 2 hours at 120°C.

The columns were slurry packed by using a procedure similar to that described by Borra and others for preparing capillary columns of larger inner diameter (100-300 µm)[5]. A slurry was prepared in a ratio of 80:1 (ml:g) slurry liquid/packing material. The slurry liquid was a sodium phosphate mobile phase (5 mM, pH 2.6). Nucleosil (Macherey-Nagel, D-5160 Duren, Germany) 5µm SB packing materal, a strong anion exchanger, was used in producing the packed capillaries. The slurry was ultrasonicated for 15 minutes and transferred to a reservoir. The reservoir was connected to a pump which was operated in a constant pressure mode at 3500 PSI. While packing took place, the slurry reservoir was placed in an ultrasonic bath to minimize settling out of the slurry (Figure 2). The particles going into the column were observed with a zoom stereomicroscope (ISCO Inc. Lincoln, NE, USA) periodically throughout the packing process. Once the desired length of the column was filled, the pressure was maintained for 30 minutes and an outlet frit was made by using a fiber optic fusion splicer to sinter a portion of the packing material. Following this, the pump was turned off and the pressure allowed to slowly decrease for another 30 minutes. The column was then removed and flushed with sodium phosphate mobile phase (5 mM pH 2.6) at 3500 PSI and a narrow (0.5 mm) detection window was made immediately adjacent to the outlet frit, again using the fusion splicer.

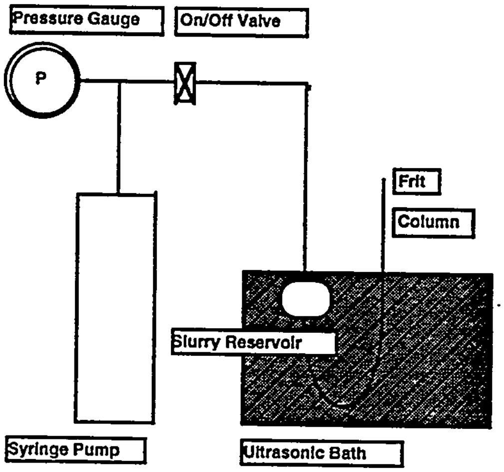

Figure 1: Apparatus used for slurry packing fused silica capillaries.

5.2.2 Assembly of the Chromatographic System.

An ATI-Unicam (now Thermo-CE, Franklin, MA, USA) model 300 CE-system was used to carry out the chromatographic and electrophoretic runs. Detection was performed on column by use of a linear instruments model 200 UV absorbance detector at 190 nm. In order to prevent the nucleation of gas bubbles, the instrument was modified in-house to operate at up to 100 PSI across the column. Samples were injected electrokinetically into the packed capillary by placing the sample vial at the anodic end of the capillary. The mobile phase vial was then replaced at the anodic end and the operating voltage was applied. Mobile phases were degassed before use by ultrasonication for 20 minutes.

6.0 RESULTS AND DISCUSSION (Current Period)

6.1 Development of CEC Methods for Analysis and Speciation: Analysis and Speciation of Ionic Iodine Species & Ionic Rhenium and Technetium Species.

Work in this quarter focused on development of a viable ion exchange CEC system, to be refined and improved on in the fourth quarter. A packed capillary format was utilized in order to provide the maximum sample loading capacity and thereby yield optimal limits of detection.

6.2 Development of Ion Exchange CEC for Analysis of Rhenium, Technetium, and Iodine.

6.2.1 Separation of Standard Mixtures of Iodide, Iodate and Perrhenate.

Both capillary zone electrophrosis (CZE) and ion exchange capillary electrochromatography (IE-CEC) were used to separate the mixture of iodide, iodate and perrhenate. Representive examples of the chromatograms obtained with IE-CEC and the electrophoregrams obtained with CZE are shown in figures 2 and 3. The three components are easily separated using both techniques. There is, however, a striking difference in selectivity. With CZE, the iodate is eluted after perrhenate, while with IE-CEC, the iodate is eluted before perrhenate. The number of theoretical plates per meter for iodide is shown in Table I.

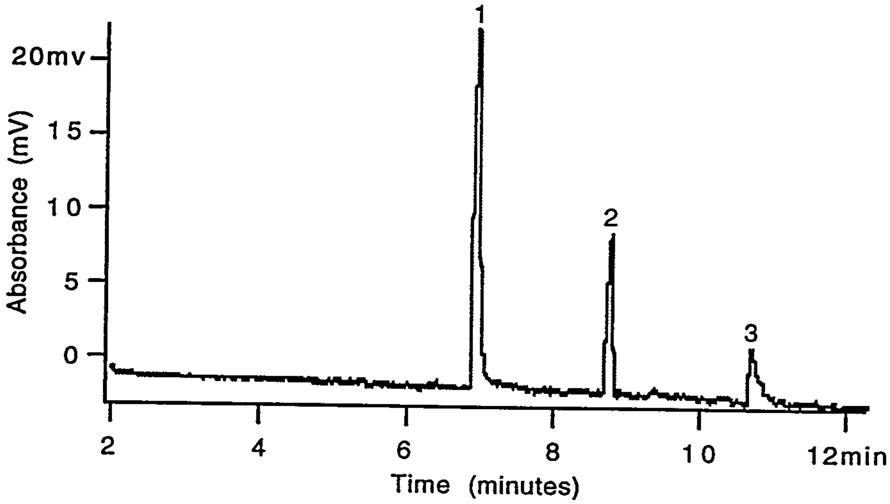

Figure 2: Separation of a mixture of iodide, iodate and perrhenate on an IE-CEC column containing Nucleosil 5µm SB (Macherey-Nagel, D-5160 Duren, Germany). L_{Tot}=60 cm, L_{Bed}=40 cm, L_{Det}=40 cm, i.d. =75 µm. Applied potential: -30 kV. UV-absorption at 190 nm. Mobile phase: 5 mM phosphate buffer (pH 2.6). Solutes: (1) iodide, (2) iodate, (3) perrhenate.

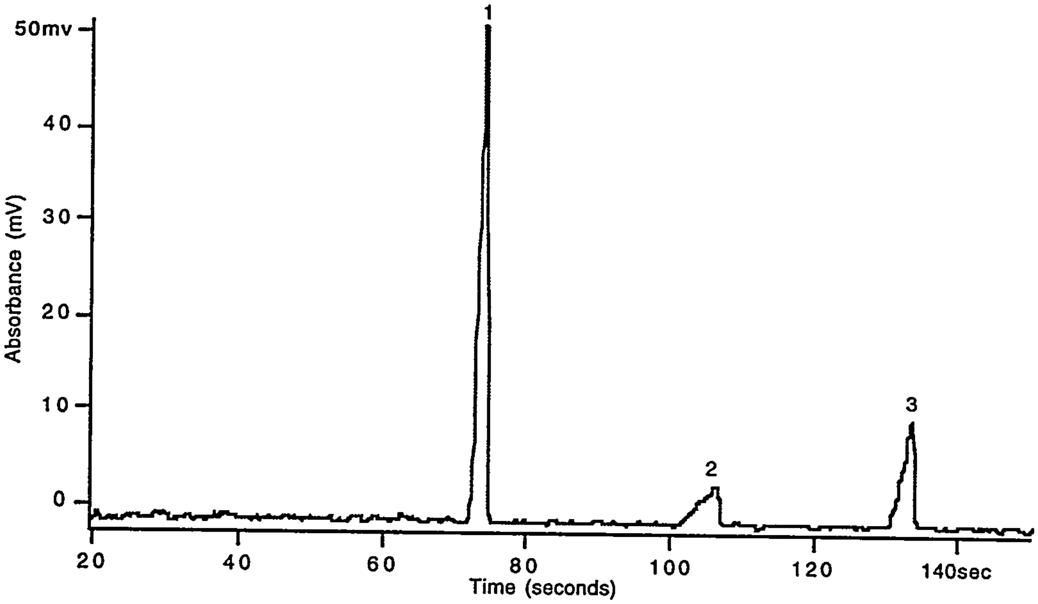

Figure 3: Separation of a mixture of iodide, iodate and perrhenate on an open tubular capillary. L_{Tot}=60 cm, L_{Det}=40 cm, i.d. =75 µm. Applied potential: -30 kV. UV-absorption at 190 nm. Mobile phase: 5 mM phosphate buffer (pH 2.6). Solutes: (1)iodide, (2) perrhenate, (3) iodate.

The high efficiency of IE-CEC is demonstrated by the reduced plate heights (h) listed in Table I. These reduced plate heights are within the range of values for h calculated for other electrochromatographic separations of compounds and are smaller than reduced plate heights obtained in HPLC, supporting the conclusion that the efficiency of electrically driven chromatography is higher than that of pressure driven chromatography.

Table I: Column efficiency (measured using iodide), plate height and reduced plate height.

method	N	N/m	H(µm)	h
IE-CEC	115000	287500	3.48	0.70
CZE	18500	46250	21.62	4.32

6.2.2 Determination of Limit of Detection.

In order to obtain the limit of detection for IE-CEC and CZE, analytical calibration curves for iodide were constructed. Very dilute solutions of sodium iodide (10^{-7} M) were employed. Chromatograms and electrophoregrams were obtained for various injection times. The calibration curve is a plot of signal (area), vs. amount of analyte injected (Figure 4). Limit of detection of IE-CEC for iodide is about 2.5×10^{-16} mol. Limit of detection of CZE for iodide is about 4.5×10^{-15} mol, which is about 20 times higher than that of IE-CEC.

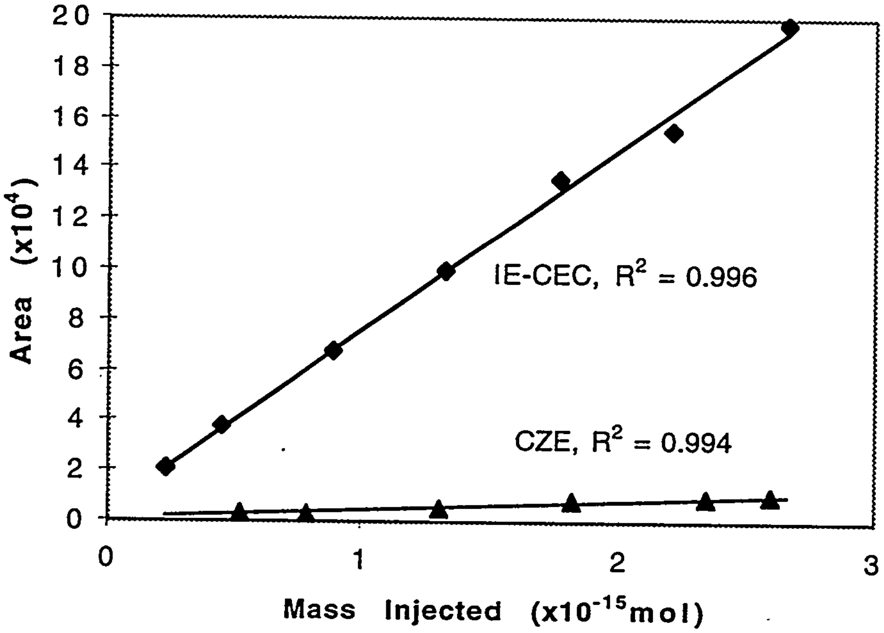

Figure 4: Calibration curves for iodide quantitation by CZE and IE-CEC. Each data point represents the mean of three runs.

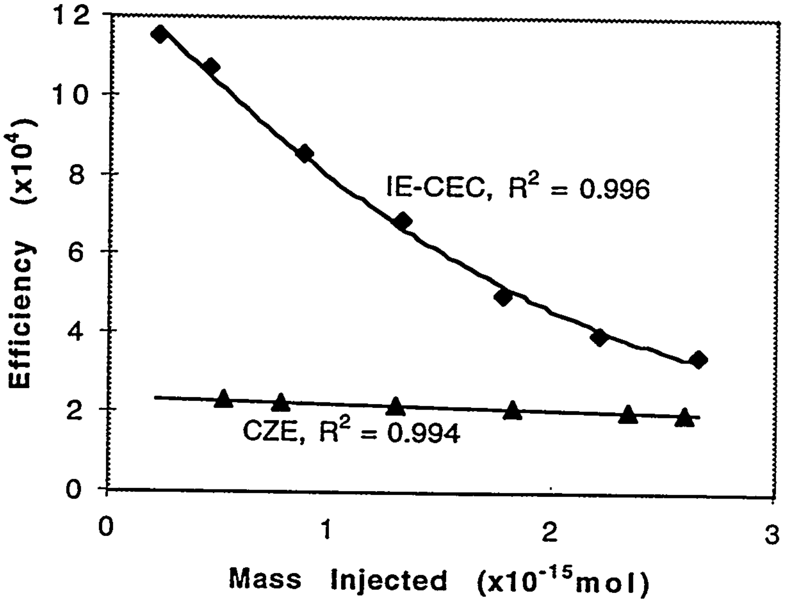

Figure 5: Efficiency as a function of sample load for CZE and IE-CEC. Each data point represents the mean of three runs.

6.2.3 Column Efficiency.

IE-CEC offers much higher efficiency than CZE for small sample loads. This is most likely due to a chromatofocusing effect in which the analytes, prepared in a weaker eluent than the mobile phase, are concentrated in a fine band at the head of the column as the sample is introduced. As injection time increases, efficiencies for both IE-CEC and CZE decrease, but that of IE-CEC decreases at a greater rate because of attenuation of the focusing effect.

6.2.4 Qualitative Reproducibility (t_R)

To study the reproducibility of a single IE-CEC packed capillary column, ten consecutive runs were performed for the analysis of sodium iodide and independently acetone using a capillary of the following dimensions: L_{Tot}=60 cm, L_{Bed}=40 cm, L_{Det}=40 cm, i.d. =75 µm. Figures 6 and 7 illustrate the reproducibility of the retention time of iodide ion and acetone, a neutral marker of the electroosmatic flow velocity.

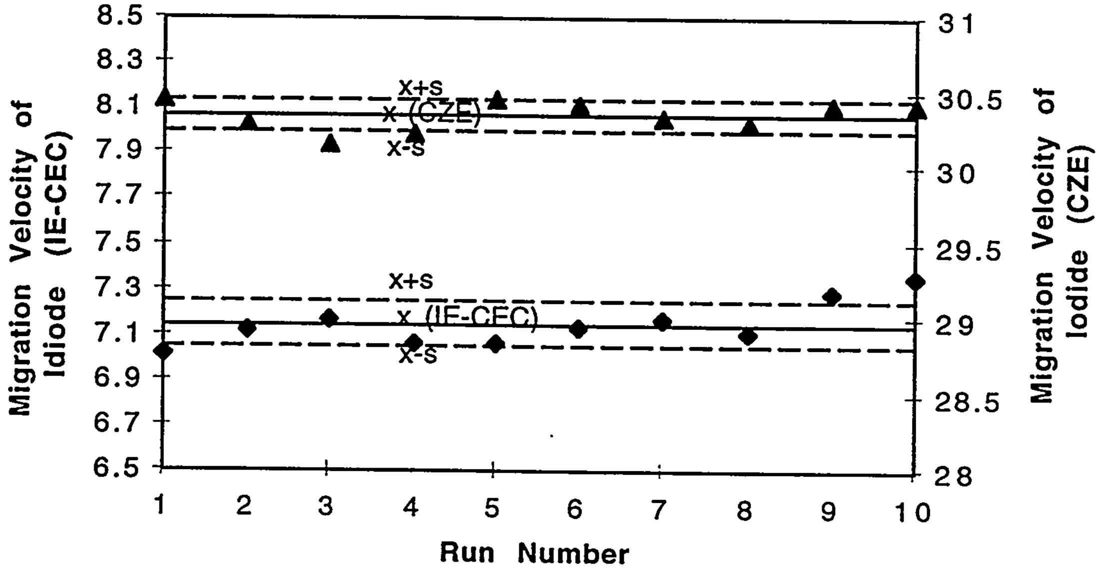

Figure 6: Retention/migration time reproducibility for the iodide ion using both separation techniques (CZE and IE-CEC).

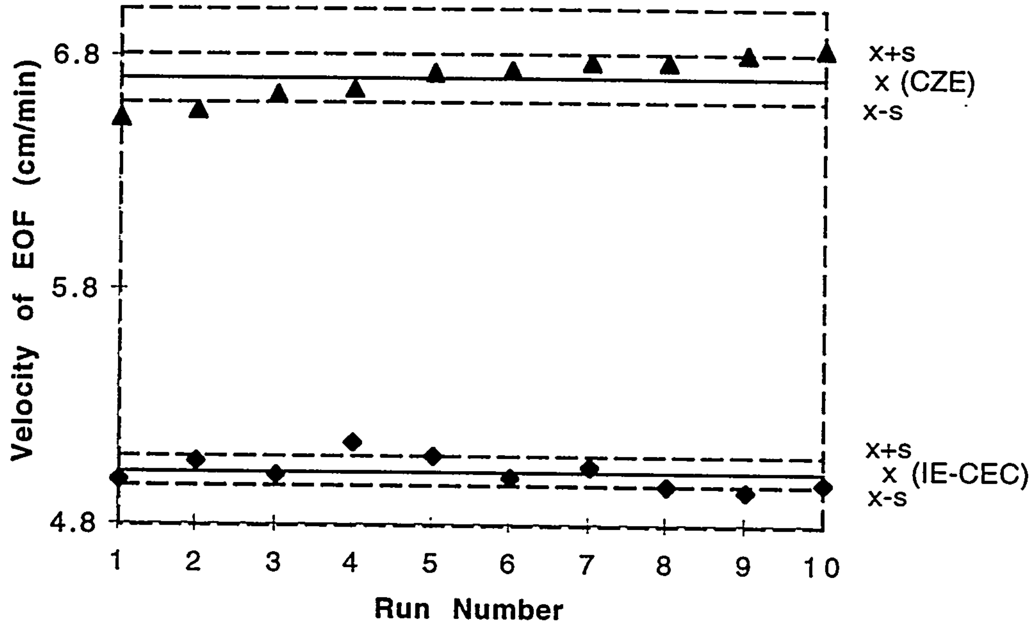

Figure 7: Electroosmotic flow rate reproducibility for each of the separation techniques studied (CZE and IE-CEC). Flow velocities were measured using acetone as a neutral marker.

7.0 CONCLUSIONS (Current Period)

We have demonstrated the feasability of IE-CEC as an emerging new technique for use in capillary-based separations. The method can be used routinely in the laboratory to achieve reproducible and reliable separations of ionic compounds. Relative to CZE, IE-CEC is capable of yielding higher efficiency and higher sample capacity in the separation of ionic compounds. The fluorescence detector constructed earlier inthe project year will be used in the fourth quarter in an effort to push limits of detection even lower. The project is on schedule and results continue to be encouraging.

8.0 WORK PLANNED FOR NEXT PERIOD

Experimentation with radiometric detection of ^{99}Tc will commence in the fourth project quarter. This work will be conducted at Battelle in conjunction with Dr. N.E. Ballou. In addition, the applicability of indirect laser-induced fluorescence detection to iodide, iodate, and perrhenate analysis by CEC will be studied. This technique will allow for the possibility of simultaneous analysis of ionic iodine and a technetium surrogate (Re) to be explored. Initial ^{99}Tc work will be conducted using the facilities and expertise of Battelle-Northwest, until such time that any necessary preparations and authorization are arranged for work to be conducted at WVU.

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PRODUCTION AND EVALUATION OF BIOSORBENTS AND CLEANING SOLUTIONS FOR USE IN DECONTAMINATION AND DECOMMISSIONING

METC Task No. 2-6A

Quarterly Technical Progress Report
Reporting Period:

July 1,1996 to September 30,1996

Work Performed Under Contract
No. DE-FC21-92MC29467

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Institute of Gas Technology
1700 South Mount Prospect Road
Des Plaines, Illinois 60018

DISCLAIMER

ABSTRACT

The objectives of this project are to prepare and to evaluate biosorbents and cleaning solutions that may be useful in the decontamination and decommissioning (D&D) of U.S. Department of Energy facilities. In this collaborative project, the Institue of Gas Technology will make available to researchers at West Virginia University (WVU) biosorbents and cleaning solutions that are useful for D&D activities, but require filtration/liquid processing in order to be used in a practical process. Filtration tests will be performed by WVU.

During the past quarter commercial surfactants were evaluated to find a suitable replacement for the specially formulated IGT-FF52 surfactant which functions well as a cleaning solution to solubilize organic contaminants. Several candidates of promising commercial surfactants were identified in preliminary foam stability tests performed in the presence of 25% ethanol. A sample of one of the commercial surfactants which performed the best, Witcamide 85, was sent to the West Virginia University for filtration tests. Additionally metal binding tests were performed with several biosorbent preparations and the results of those tests will be reported in the next quarterly report.

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1.0 Introduction

The decontamination and decommissioning (D&D) of the Department of Energy facilities can result in the production of large volumes of wastewater containing metals, radionuclides, organic contaminants, and particulate material. Treatment of these wastewater streams is highly problematic because of their complexity and toxicity. Treatments appropriate for the removal of particulate material, such as filtration, are generally ineffective for the removal of soluble contaminants in D&D wastes such as metals and radionuclides. Techniques commonly used for the removal of metals and radionuclides from wastewater include the use of ion exchange resins; however, particulate material and organic contaminants present in D&D waste are incompatible with the use of ion exchange columns because of plugging/fouling. Biosorbents are biologically derived substances that can be used to sorb metals/radionuclides in a manner analogous to ion exchange resins, and biosorbents may be inexpensive in comparison with ion exchange resins.

The approach that is being tested in this project is the use of biosorbents, in a powdered form, added directly to D&D wastewater for the removal of metals and radionuclides. The use of biosorbents as powders rather than as ion exchange columns avoids the plugging/fouling problems associated with ion exchange columns. Also the powdered biosorbent should provide better contact and therefore more rapid metal/radionuclide binding than with ion exchange columns or immobilized biosorbent columns. D&D wastewaters require filtration in order to remove particulates and the same filtration step can be used to remove biosorbent particles and metals/radionuclides bound to them. It is expected that this approach will simplify and decrease the cost of treatment of D&D wastewater.

A related issue in D&D activities is the use of effective cleaning solutions, and the ultimate fate of used/spent cleaning solutions. The Institute of Gas Technology (IGT) has developed novel surfactant formulations that are highly efficient in the removal of organic and inorganic contaminants from solids. Ordinarily surfactants are discarded in spent D&D solutions. However, these surfactants can inhibit the subsequent treatment of these solutions and if not completely destroyed can contribute to the mobility of contaminants and thereby pose an environmental risk. This project will examine the possibility of using filtration to remove surfactants from D&D solutions, and possibly aid in the recovery/reuse of those surfactants, as well as aid in the treatment of D&D wastewater.

2.0 Purpose

The purpose of this project is to evaluate the use of biosorbents in conjunction with filtration to treat D&D wastewater. An additional purpose of the project is to evaluate the removal of surfactants from D&D solutions.

3.0 Background

IGT has experience in the production and use of biosorbents, and in the production and use of surfactants for removing contaminants from solids. The West Virginia University (WVU) has experience in filtration and possesses a bench-scale model of a novel centrifugal filtration device developed by SpinTek Corporation. IGT and WVU are collaborating on this project to combine their areas of expertise to develop improved processes for the treatment of D&D wastewater.

4.0 Methodology

Bacterial Strains and Culture Media

Escherichia coli (ATCC 23724) was grown in nutrient broth (Difco) while *Zoogloea ramigera* 115 (ATCC 25935) was grown in nutrient broth supplemented with 25 g glucose per liter. Cultures were incubated at 30°C and the production of exopolysaccharide by *Z. ramigera* was monitored spectrophotometrically, and by measuring the viscosity of the medium. *Z. ramigera* cultures were harvested when viscosities exceeding 600 centipoise were achieved.

Immobilization of Dried Biomass

Biomass (cells plus exopolysaccharides if present) was harvested by centrifugation at 10,000 X g for 10 minutes and dried under vacuum in a rotary evaporator at 65°C. The dried biomass was ground to a uniform fine powder using a mortar and pestle. Dried, powdered biomass was used as is for metal-binding experiments.

Analytical Procedures

Atomic Absorption Spectrophotometry (AAS) (Model: 403, Perkin-Elmer Co.) Inductively Coupled Plasmic Emission Spectroscopy (ICPAES) (Model: Atom Scan 25, Thermo Jaorell Ash Co.) and ion exchange chromatography (Model DX-100, Dionex Corp.) were used for metal concentration analyses. Samples were diluted to 0.1-1.0 ppm to ensure accurate analysis. To remove the interference from suspended particles, samples were centrifuged (600 rpm, 5 min) and filtered by a 0.45-µm filter before injection into the AAS, ion exchange chromatography, or ICPAES apparatus.

Metal Adsorption Studies

Batch studies were performed using 100 mg of biomass in a 250-ml Erlenmeyer shaker flask containing 100 ml of 10 to 100 ppm metal solution (adjusted to pH 6.0 or 4.0 using HCl) and shaken for 15 minutes to 24 hours at a speed of 160 rpm at room temperature. Biomass was removed by centrifugation, the supernatant was decanted, filtered, and metal ions were then analyzed by AAS, ion exchange chromatography, or ICPAES.

Surfactant Solutions

The surfactant IGT-FF52 was prepared according to proprietary procedures. Commercial surfactants were obtained from Witco Corporation, Shell Chemicals, and Tomah Chemicals. These surfactants are described in greater detail in the attached appendix.

5.0 Work Performed This Period

During the past quarter commercial surfactant samples were evaluated for their ability to replace the "home made" IGT-FF52 surfactant for use in decontamination solutions to solubilize organic contaminants. Preliminary tests identified several commercial products which appear to be appropriate and a sample of one of the best products, Witcamide 85, was sent to WVU for filtration tests. Additionally, metal binding tests were performed using several biosorbent preparations. These items fulfill key milestones of the project and provide material needed by WVU to complete their portion of the project.

6.0 Results and Discussion

The objective of the work during this quarter has been to identify commercial surfactant formulations which will replace the home-made IGT formulation (IGT-FF-52). The superior performance of the IGT-FF52 surfactant to solubilize/remove organic contaminants results from its unique ability to function well in the presence of high concentrations of ethanol (20% to 50%). Ethanol destabilizes most surfactants and is used as a foam breaker; however, IGT-FF52 functions well in ethanol yielding reasonably stable foams. The combination of the IGT-FF52 surfactant and ethanol as a cosolvent results in superior ability to solubilize organic contaminants. If a practical process is to be developed based on the use of ethanol-surfactant mixtures the identification of commercial surfactants that can substitute for the specially formulated IGT-FF52 will allow better availability of products for scale-up activities and products with known costs. Surfactant samples like amines (primary, secondary, amphoteric etc.), amides, ethoxylated alcohols, ethoxylated amines, anionics etc. from a few manufacturers like Witco Corp., Shell Chemicals and Tomah Chemicals were obtained. Due to the requirement of a unique interaction between the surfactant and the ethanol (present in the solution) for film stability, an extensive search was conducted to identify a few potential surfactant mixtures. The details of the various surfactant compositions obtainable from the literature are given in the Appendix.

About twenty-five different surfactant mixtures were tested for their applicability to substitute for IGT-FF52 in decontamination solutions. In this report, the surfactants will be referred to by their abbreviations which have been defined in the appendix. Tests for all the surfactant formulations were carried out using a 25% (w/w) (i.e., 31.25% v/v) ethanol solution, at a total surfactant concentration of 4.78% in the solutions.

A Bartsch test (or shake test) was done for the above prepared samples. This test provides a simple method from which information on the behavior of surfactants under dynamic conditions and the stability of foam can be extracted. A key characteristic of IGT-FF52 is its ability to form reasonably stable foams in the presence of ethanol. This is somewhat unusual as ethanol normally destabilizes surfactants and can be used to break foams. Therefore testing to determine the ability to form stable foams in the presence of 25% ethanol was performed as a way to rapidly evaluate the suitability of a large number of commercial products and combinations of products. In the Bartsch test, a given volume of surfactant solution is taken in a graduated cylinder and agitated to form the foam, whose various properties can then be studied. The initial height of the foam after agitation is an indicator of the dynamic behavior of the surfactants, the lower is the dynamic surface tension, the higher is the height. In regards to long periods of standing times, only the most stable foams will be left and the less stable foams will collapse. The rate of liquid drainage from the foam can also be used study the contribution of the adsorbed layer in stabilizing the foam. Hence, such an experiment is a quick way to asses the suitability of a particular surfactant formulation. In a preliminary experiment, 10 ml of the formulation (5 cm in height) was taken in a graduated cylinder and agitated in a standardized manner for 15s to produce the foam. The height of the top of the foam and the level of the liquid at the bottom of the foam were monitored as a function of time. The total height of the foam and the liquid at the end of 9.5 hours were also noted for all the samples. After identifying a few potential surfactant formulations from this preliminary test (with 10 ml of the sample), another shake test was done using 50 ml of the surfactant solution, and then compared with the results of IGT-FF-52. Surfactant solutions were freshly prepared and left overnight before being used in the experiments. The time provided was enough to dissolve the surfactants in the solvent.

The aim of this investigation was to replace the "home-made" surfactant IGT-FF-52 with a commercially available product. A brief summary of the results is presented below; the surfactant (mixture) used in each test is indicated followed by a brief note on its performance. Formulations which did not yield a reasonable stability were not pursued for further experimentation. The essential criteria for a potential formulation in this preliminary screening exercise was good foaminess and stability in the presence of 25% ethanol.

1) PA-16: Solubility in the solvent (25% ethanol solution used for all the tests) is less than 1% w/w. At higher concentrations, a turbid solution is formed which slowly phase separates. Contradictory to what was expected (strong hydrogen bonding between primary amine and ethanol leading to stable foams) the foams were unstable with a half-life of only about a minute. This extremely low stability makes this formulation unsuitable for foam mediated soil remediation applications.

2) PA-19: This single surfactant formulation manufactured by TOMAH yielded the same characteristics as the previous sample, and was also found unsuitable for soil remediation due to the extremely low stability of the foam.

3) DA-14 + Enordet (3:1): Good small bubbles are formed but the foam collapsed in under a minute. Vertical film drainage was also studied and did not reveal any convective cells in the film. This leads us to conclude that ethanol evaporation-induced film destabilization is not the cause for low stability. The film does not even reach the black-film stage; at rupture, a thick film is present. Thus, this formulation does not provide either adsorbed layer or micellar stabilization. The same is also true for a pure DA-14 solution.

4) DA-17 + Enordet (3:1): This mixed surfactant formulation yielded the same characteristics as the previous sample, and was also found unsuitable for soil remediation due to the extremely low stability of the foam.

5) Amphoteric-N (AN)+ Enordet (EN): The initial results were encouraging, with the half-life in the order of tens of minutes. Thus, a more detailed experiment was done by changing the ratio of the surfactants from 0 to 1. The results are listed in Table - I. The half-life for the foam formed with such a formulation is the highest when pure Amphoteric-N surfactant is used (between 2 to 4 hours). The initial foaminess is nearly the same for all the ratios checked. As the stability was not nearly as good as the other formulations (compard with Tables II and IV) this mixture was not studied further.

6) Varonic T-215 + Enordet (EN): The foam half-life is about one minute. Excessive alcohol evaporation from the foam lamellas causes film instability and hence rupture.

7) PA-1214 + Enordet (EN) (1:0 and 3:1): For both the ratios checked, the foam half-life was about 30s.

8) Witcamide-5085 (W-85) + Amphoteric-N (AN): The initial results were encouraging, with the half-life in the order of a few hours. Thus, a more detailed experiment was done by changing the ratio of the surfactants in the whole range from 0 to 1. The results are listed in Table 1. The greatest stability was obtained for W-85:AN of 3:1, with a half-life of more than 9.5 hours. The foaminess of this sample is slightly lower than the less stable ratios of the formulation. This particular ratio was also experimented in a 250 ml cylinder where the foam would experience a larger hydrostatic suction force due to the higher foam height. It was observed that both foaminess and foam stability are good.

9) PA-16 + AN (3:1 and 1:1): The half-life is only about 15 minutes for both the ratios, hence this surfactant mixture was not considered suitable for the application at hand.

TABLE - 1 : Results of Bartsch test of formulations (5) & (8), in 25 ml cylinders.

The initial volume of the liquid is 5 ml (when no foam is present). The table lists the combined height of the foam and the liquid.

AN/ (AN+EN)	H_i	$H_{1\text{-hr}}$	$H_{2\text{-hrs}}$	$H_{4\text{-hrs}}$	$H_{6\text{-hrs}}$	$H_{7.5\text{-hrs}}$	$H_{9\text{-hrs}}$
0.25	14.5	8	5.5	5	5	5	5
0.5	14.3	5.5	5.5	5	5	5	5
0.75	14.5	7.5	5.5	5	5	5	5
1.0	14.8	13.3	11.5	5.5	5	5	5
W85/(W85+ AN)							
0.25	14.3	14	14	12.3	10.3	9	7.8
0.5	15.1	6.5	6	5	5	5	5
0.75	12.5	12.5	12.2	12.2	12	11.8	11.8
1.0	10	10	10	10	9.8	9.8	9.8
W85/EN	14.4	14.3	13.7	13.5	13.3	13.1	11.5

10) DA-17 + AN (3:1): This has a half-life of about 5 minutes. Bubble pockets are formed in the foam structure as the lamella ruptures in the middle and the foam collapses in a very short time.

11) AN + PA-19: The initial results were encouraging (for ratios AN/PA-19 > 1), with the half-life in the order of few hours. For the ratios AN/PA-19 < 1, the solution was turbid and phase separated due to the insoluble nature of PA-19 in the solvent. Thus, a more detailed experiment was done by changing the ratio of the surfactants (AN/PA-19) in the region greater than 1. The results are listed in Table - 2. We see that the most stability and foaminess is obtained for AN:PA-19 of 4 (i.e. AN/(AN+ PA-19)= 0.8), with a half-life of more than 9.5 hours. This particular ratio was also experimented in a 250 ml cylinder, the results of which are tabulated in Table - 3.

NOTE: Both the above surfactants are manufactured by Tomah.

12) Witconol-100 (W-100)+ AN (3:1): W-100 (HLB about 13.8) has structure close to that of Enordet (HLB about 14.4) but is a little more hydrophobic as can be seen from the HLB numbers. The Half-life of this formulation was about 5 mins. Comparing this run with run 5, we see that Enordet seems to perform better in the 25% (w/w) ethanol solution. A myriad number of reasons can contribute to a surfactants failure, including polydispersity in the chemical product itself. W-100 seems to have an effect of destroying the foam.

13) PA1214 + Standapol ES-2 (1:1): This formulation had a half-life of only about 5 mins. It was expected that PA-1214 would prevent ethanol evaporation from the film (due to primary amine-ethanol hydrogen bonding) and standapol would increase the film stability by micelle formation, but even this formulation had a half-life of only about 5 mins.

TABLE - 2 : Results of Bartsch test of formulations (11) & (16)-(20), in 25 ml cylinders.

The initial volume of liquid is 5 ml (when no foam is present). The table lists the combined height of the foam and the liquid.

AN/(AN +PA19)	H_i	$H_{1\text{-hr}}$	$H_{2\text{-hrs}}$	$H_{4\text{-hrs}}$	$H_{6\text{-hrs}}$	$H_{7.5\text{-hrs}}$	$H_{9.5\text{-hrs}}$
0.6	14.3	14.3	8.7	6.5	6.3	6.3	6
0.7	14.8	11.7	11.7	8	8.3	8.3	8.2
0.8	14.8	14.5	14.3	14	14	13.8	13.5
0.9	14.9	14.7	13	12	5	5	5
W85/(W85+Neodol)							
Neodol 1-5	14.1	9.5	9	7	6.5	6	6
Neodol 1-7	11.5	8.5	8.3	7.4	6.6	6	5.5
Neodol 1-9	12.5	12.3	11.1	8.8	7.6	6.5	6
Neodol 25-12	15.3	12.6	11.5	10	9.2	8.3	6
Neodol 45-9	15	14.5	13.8	11	10.2	8.7	8

TABLE - 3 : Results of Bartsch test of formulations (8), (11), (19) & (20), in 250 ml cylinders.

The initial volume of the liquid is 50 ml (when no foam is present). The table lists the combined height of the foam and the liquid.

	H_i	$H_{5\ \text{mins}}$	$H_{10\ \text{mins}}$	$H_{15\ \text{mins}}$	$H_{20\ \text{mins}}$	$H_{25\ \text{mins}}$	$H_{30\ \text{mins}}$	$H_{35\text{mins}}$
AN/(AN+PA19)= 0.8	31	31	27	25	19.4	17	15	15
W85/(W85+AN)= 0.75	31	21	13	10	8.5	7	7	6.5
W85/(W85+N45) =0.75	31	23	22	19	17	16.6	16	15.5
W85/(W85+N25-12)=0.75	31	30	26	21	18.6	16	15	14
W85/(W85+E) =0.75	31	31	29.5	27	25	20	18	15.5
IGT-FF52	31	29	23.5	15.5	11	10	10	10

14) PA16 + Standapol ES-2 (1:1): A mixture of primary amine and a second surfactant were again checked in this formulation for their synergistic effect (if any) in stabilizing the foam. Since PA-16 prevented ethanol evaporation from the film (similar to the earlier formulation), standapol was used to increase the film stability by micelle formation, but even this formulation had a half-life of only about 15 mins.

15) PA19 + Standapol ES-2 (1:1): This formulation had a half-life of only about 20 mins. By increasing the hydrophobic chain length of the surfactant, the CMC of the surfactant would decrease and its micelle forming capacity would increase. In the last two formulations this chain length effect created a slight increase in the stability of the foam formed. Since PA-19 prevented ethanol evaporation from the film, standapol was used to increase the film stability by micelle formation, but this formulation had a half-life of only about 20 mins.

16) W-85 + Neodol 1-5 (3:1): The Neodol series of surfactants are ethoxylated alcohols, which are the new line of Shell products which have replaced their Enordet series. Hence the Neodol series was checked to compare performance with the best performing formulation which employs Enordet (W85/EN). This particular formulation had moderate foaminess and good stability. The results are tabulated in Table-2. The half-life was about 7 hours.

17) W-85 + Neodol 1-7 (3:1): The effect of changing the HLB (Hydrophilic Lipophilic Balance) of the second surfactant (in this case by increasing the ethoxylation) was studied. This formulation behaved in nearly the same manner as the previous formulation. It had moderate foaminess and good stability. The results are tabulated in Table-2. The half-life was about 7 hours.

18) W-85 + Neodol 1-9 (3:1): With a HLB of about 13.9, Neodol 1-9 is most hydrophilic of the Neodols' studied so far. The results are tabulated in Table-2. The half-life was about 7 hours.

19) W-85 + Neodol 25-12 (3:1): This is the same as the W85/EN formulation except that Neodol 25-12 is the new line of Shell product which has replaced their Enordet series. The foam had good foaminess and stability too. The half-life was greater than 7.5 hours. This formulation was also experimented in a 250 ml cylinder, the results of which are tabulated in Table-3. It is observed that both foaminess and foam stability are comparable to the W85/EN formulation.

20) W-85 + Neodol 45-7 (3:1): Neodol 45-7 (HLB about 11.7) is more hydrophobic than Neodol 25-12 (HLB about 14.4). This foam also had satisfactory foaminess and stability. The half-life was

greater than 7.5 hours. This formulation was also experimented in a 250 ml cylinder, the results of which are tabulated in Table-3. It was observed that both foaminess and foam stability are comparable to the W85/EN formulation.

21) W-85 + Witconol SN-90 (SN-90) (3:1): SN-90 is an ethoxylated alcohol manufactured by Witco, and is similar to the Neodol series of Shell. The results are tabulated in Table-4 (for 250 ml cylinder tests). Both the foaminess and foam stability are only moderate In the larger cylinder tests, the foam collapse is quite rapid, indicating that a tall column of foam of this formulation will not be stable and will collapse under its own hydrostatic pressure.

22) W-85 + Witconol SN-70 (SN-70) (3:1): This formulation has slightly better stability than the previous one (SN-70 is more hydrophobic than SN-90, due to two units of ethoxy units less in its structure). The results are tabulated in Table-4 (for 250 ml cylinder tests). This surfactant mixture has a satisfactory foaminess with a moderate stability. The stability is easily visible in the 250 ml cylinder test, where it is only moderately stable compared with the other samples.

23) W-85 + Witconol TD-140 (TD-140) (3:1): TD-140 is a relatively hydrophilic surfactant (HLB about 15.1) among those that were studied. The results are tabulated in Table-4 (for 250 ml cylinder tests) and one can see that this is a promising formulation. It had good foaminess and stability.

TABLE - 4 : Results of Bartsch test of formulations (21-24), in 250 ml cylinders.

The initial volume of the liquid is 50 ml (when no foam is present. The table lists the combined height of the foam and the liquid.

	H_i	$H_{5\ mins}$	$H_{10\ mins}$	$H_{15\ mins}$	$H_{20\ mins}$	$H_{25\ mins}$	$H_{30\ mins}$	H_{35mins}
W85/(W85+SN90)=0.75	29	29	13	11	10.8	10.8	10.6	10
W85/(W85+SN70)=0.75	30.5	28	25	25	20	17	15	15
W85/(W85+TD-140)=0.75	30.5	30.5	29.5	27	23	20.8	20.8	20.4
W85/(W85+2722) =0.75	30.5	30.5	27	27	26	25	20.6	20
W85/(W85+EN) =0.75	31	31	29.5	27	25	20	18	15.5
IGT-FF52	31	29	23.5	15.5	11	10	10	10

24) W-85 + Witconol-2722 (2722) (3:1): 2722 (polysorbate-80) is a food surfactant, and hence it is environmentally benign. The results are tabulated in Table-4 (for 250 ml cylinder tests). Both the foaminess and the stability are good; they are better than the W85/EN formulation. This formulation has potential to be applied environmental remediation.

As was stated earlier, the aim of this investigation was to replace the "home-made" surfactant IGT-FF-52 with a commercially available surfactant. The ratio of surfactants seems to affect the ethanol evaporation from the foam films due to the varying adsorbed layer compositions. The adsorbed layer interacted with the ethanol and lowered its evaporation from the film. At 25% (w/w) alcohol concentration, for most surfactant formulations, the foaminess and stability were the highest for the formulation having a ratio of amide/(amide+second surfactant) at about 0.75.

In the first few formulations (1 to 5) that were tested with primary amines (Tomah surfactants), it was found that the films were extremely unstable. The low life time can probably be attributed to a lack of any stabilizing effects from the micelles (from structural forces to the disjoining pressure isotherm) from those formulations.

The mixture of W-85 and AN, even though performed well in the small cylinder tests, did not do as well in the 250 ml cylinder Bartsch test. The amphoteric surfactant AN by itself was also quite effective in reducing ethanol evaporation from the foam films. When this (i.e. AN) was combined with the primary amines, it imparted good foaminess and stability to the foam even under large hydrostatic pressures. This was especially true for the formulation (11) of PA-19 + AN. The lower chain lengths of PA did not have such a good synergistic effect with AN and the foam life time was short. In this preliminary study, as the main emphasis was to identify a few formulations which performed well in the presence of an ethanol-based solvent, further detailed studies investigating the cause of the observed behavior have not yet been carried out. Such studies will be done in the next quarter with the aim of determining the specific ability of promising commercial surfactants to solubilize organic contaminants such as PAHs.

Comparing experiments between Witconol TD-100 (HLB=13.8) and Witconol TD-140 (HLB= 15.1) as the second surfactant, it was found that the more hydrophilic TD-140 performed much better than TD-100. Similar results could not be seen as distinctly with the Shell surfactants, where the lower HLB surfactants performed nearly as well as the higher HLB ones. This could be due to a number of reasons like the spread of the actual product composition from the average quoted one, other impurities in the surfactant products, etc. Neodols 25-12 and 45-7 performed nearly the same when used with W-85 and only marginally less than the W85/EN formulation, but much better than IGT-FF-52 in regards to foam stability and foaminess (Table-3).

The formulations 23 (W-85 + TD-140) and 24 (W-85 + 2722) performed the best among all the formulations considered. All the components are biodegradable, the foam has a high foaminess and good stability. In general then Witcamide 85 (W85) appears to perform well in these preliminary tests, and is the best candidate for a commercial product to replace IGT-FF52. Accordingly, samples of W85 have been sent to WVU for filtration tests and the ability of W85 to desorb PAH contaminants from soil will be tested next quarter.

7.0 Conclusions

The project is proceeding as scheduled.

The object of the investigation carried out this quarter was to replace the "home-made" surfactant IGT-FF-52 with a commercially available surfactant. Surfactant samples like amines (primary, secondary, amphoteric etc.), amides, ethoxylated alcohols, ethoxylated amines, anionics etc. from a few manufacturers like, Witco Corp., Shell Chemicals and Tomah Chemicals were obtained. Due to the requirement of a unique interaction between the surfactant and the ethanol (present in the solution) for film stability, an extensive search was conducted to identify a few potential surfactant mixtures. About twenty-five different surfactant formulations were tested for their applicability to form foam in 25% ethanol solutions. The ratio of surfactants (of the multicomponent system) affected the stability of the foam formed. At 25% (w/w) alcohol concentration, the foaminess and stability were the highest for the formulation having a ratio of amide/(amide+second surfactant) at about 0.75 (for most surfactant formulations).

In this preliminary study, the main emphasis was to identify a few formulations which performed well in the presence of an ethanol-based solvent; further detailed studies investigating the cause of the observed behavior will be carried out in the next quarter with the aim of obtaining data concerning the specific ability of candidate solutions prepared from commercial surfactants to desorb/solubilize organic contaminants. A sample of the commercial surfactant Witcamide 5085 (W85) was sent to WVU for use in filtration tests using the SpinTek ultrafiltration device.

The following surfactant formulations perform better than IGT-FF-52 and are comparable to or better than W85/EN formulation (Witcamide-5085 + Enordet 1215-12; 3:1) as regards foaminess and foam stability. The numbers in the brackets are the ratios of the two surfactants (the total surfactant concentration used was 4.78% w/w):

Witcamide-5085 (W-85) + Amphoteric-N (AN) (3:1)

AN + PA-19 (4:1)

W-85 + Neodol 25-12 (3:1)

W-85 + Neodol 45-7 (3:1)

W-85 + Witconol 2722 (W-2722) (3:1)

W-85 + Witconol TD-140 (TD-140) (3:1)

Filtration tests using surfactant solutions should proceed as planned as should the evaluation of biosorbents to bind metals in a simulated D&D wastewater.

8.0 Work Planned for Next Period

Metal binding experiments to test the ability of biosorbents to remove metals from simulated D&D wastewater have been performed and the results of those tests will be obtained and evaluated. Additionally, the ability of promising commercial surfactants such as Witcamide 85 will be evaluated in comparison with IGT-FF52 for their ability to desorb PAH contaminants from soil.

APPENDIX - I

LIST OF SURFACTANTS USED

This appendix lists the surfactants that were used in this study. The abbreviations used for the surfactants in the report are shown as bold text in the following list. Wherever the abbreviation is not mentioned, the complete surfactant name was referred to in the report.

SURFACTANTS FROM TOMAH

Primary Ether Amines ($R\text{-}O\text{-}(CH_2)_3\text{-}NH_2$) :

1) **PA1214** (R is about 12 to 14): Octyl/Decyl oxypropyl amine

2) **PA16** (R is about 16): isododecyl oxypropyl amine

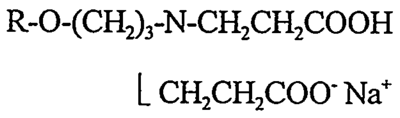
3) **PA19** (R is about 19): Linear alkoxypropyl amine

Ether Diamine ($R\text{-}O\text{-}[(CH_2)_3\text{-}NH]_2)\text{-}H$:

4) **DA14**: N-isodecyl oxypropyl-1,3 diamino propane

5) **DA17**: N-isotridecyl oxypropyl-1,3 diamino propane

6) Amphoteric-N (**AN**):



$$\begin{array}{l} R\text{-}O\text{-}(CH_2)_3\text{-}N\text{-}CH_2CH_2COOH \\ \qquad\qquad\qquad\;\; \llcorner\, CH_2CH_2COO^- Na^+ \end{array}$$

(R is 12-15 chain linear hydrocarbon)

SURFACTANTS FROM WITCO

1) Witcamide-5085 (**W-85**): (Oleic diethanol amide modified)

toxic ingredients: 15% diethanol amine

Sp. density: 0.99

2) **Varonic T-215**: (Ethoxylated tallow amine)

N, N, bis(polyoxyethyl-15) unstaurated tallow amine

Description: Amines C14-C18 and C16-C18 unsat

conc.: >99%

Remarks: Emulsifiers

3) Witconol SN-90 (**SN-90**): Ethoxylated alochol

$R_{10\text{-}12}(EO)_9$; HLB about 13

4) Witconol SN-70 (**SN-70**): Ethoxylated alochol

$R_{10\text{-}12}(EO)_7$; HLB about 11.3

5) Witconol TD-100 (**TD-100**): Ethoxylated alochol

$R_{13}(EO)_{10}$; HLB about 13.8

6) Witconol TD-140 (**TD-140**): Ethoxylated alochol

$R_{13}(EO)_{14}$; HLB about 15.1

7) Witconol 2722 (**2722**): Polysorbate 80 (Sorbitanoleate ethoxylated)

SURFACTANTS FROM SHELL

All Neodol surfactants are ethoxylated alcohols and biodegrade easily. They have the following genneric structure,

$$R\text{-}O\text{-}(CH_2CH_2O)_nH$$

1) **Neodol 1-5**:

with C_{11} alcohol for R, n=5; HLB= 11.2

2) **Neodol 1-7**:

with C_{11} alcohol for R, n=7; HLB= 12.9

3) **Neodol 1-9**:

with C_{11} alcohol for R, n=9; HLB= 13.9

4) **Neodol 25-12**:

with R in the range of C_{12} to C_{15} alcohol , n=12; HLB= 14.4

5) **Neodol 45-7**:

with R in the range of C_{14} to C_{15} alcohol , n=7; HLB= 11.6previous two formulations.

6) Enordet 1215-12 (**EN**)

with R in the range of C_{12} to C_{15} alcohol , n=12; HLB= 14.4

USE OF SPINTEK CENTRIFUGAL MEMBRANE TECHNOLOGY AND SORBENT/CLEANING SOLUTIONS IN THE DECONTAMINATION AND DECOMMISSIONING OF DOE FACILITIES

METC Task No. 2.6B

Quarterly Technical Progress Report
Reporting Period: 6/31/96 - 9/30/96

Work Performed Under Contract
No.: DE-FC21-92MC29467

For:
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Brian E. Reed, Associate Professor
Wei Lin, Research Assistant Professor
Department of Civil and Environmental Engineering, West Virginia University
PO Box 6103, Morgantown, WV 26506-6103
304.293.3031 ext. 613, 304.293.7109 (Fax)

DISCLAIMER

ABSTRACT

The use of sorbents (biosorbents, powdered activated carbon) and cleaning agents (surfactants, chelating agents) to decontaminate surfaces such as pipes, walls, *etc.* represents a unique and cost-effective method for remediating DOE facilities. As envisioned, the sorbent or cleaning agents would be added to a liquid (most likely water) and the solution would be contacted with the contaminated surface. For example, a pipe that is contaminated with inorganic and organic contaminants can be contacted with a solution containing a surfactant (for organics) and a chelating agent (for inorganics). After removal of the surface-bound contamination by the cleaning agents, a solid sorbent can be added to the liquid to bind the cleaning agent-contaminant compound. A major problem associated with this approach is the efficient separation of the sorbent/cleaning agent from the liquid phase using conventional separation techniques.

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks set on a hollow rotating shaft inside a cylindrical housing. The waste stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate is forced through the membrane and is collected in the hollow shaft and discharged. The concentrate exits at the edge of the membrane packs. The centrifugal action continuously cleans the membrane surface decreasing cleaning chemical use and maintenance and increasing the volume of waste treated per area of membrane.

The objective of this Task 2.6B research is to investigate the feasibility of using centrifugal membrane technology to separate sorbents/cleaning agents used in the decontamination/decommissioning of DOE facilities. The result of this effort will be the development of an efficient cost-effective method for the decontamination/decommissioning of DOE facilities. An additional benefit of this research is the application of the experimental results to the treatment of other DOE wastes such as contaminated groundwater, mixed-waste process water, and existing waste residuals (*e.g.,* Hanford Tank Farm Waste). It is envisioned that the volume reduction factors for this technology will be high and a relatively low amount of residual requiring additional treatment will be produced.

Work on the project will be performed by West Virginia University's Department of Civil and Environmental Engineering (WVU-CEE) and the Institute of Gas Technology (IGT). The following tasks will be completed by WVU-CEE: 1) selection and procurement of membrane(s) and 2) bench-scale testing of centrifugal membrane process. The following tasks will be completed by IGT: 1) identification and formulation of synthetic wastes and 2) selection and evaluation of sorbents and cleaning solutions. WVU-CEE and IGT will submit separate quarterly reports. A joint proposal will be prepare to obtain funding for D&D research through existing DOE ROAs and PERDAs.

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EXECUTIVE SUMMARY

Three runs were completed to study the SpinTek system's effectiveness at separating several types of solids/macromolecules. A thickened sludge from a domestic wastewater treatment plant (used to simulate biosorbents) was used in the first run. Two concentrations of kaolinite (0.8 and 3.2%) were used in the second run to simulate a zeolite sorbent. A biosorbent, supplied by IGT, was studied in the third run. Five ionic strengths were studied in the IGT biosorbent run by adding dosages of $NaNO_3$ (0.01N, 0.05N, 0.1N, 0.5M, and 1.0N) to the biosorbent solution to test the durability of the ceramic membrane. Additionally, two runs were completed using an amide surfactant provided by IGT (IGT-FF52) to study the removal of micelles from the amide solution. In the future, this may aid in the treatment of actual D&D wastewater and may aid in the recovery/reuse of those surfactants.

A thickened waste activated sludge/distilled water mixture was treated for three hours in recycle mode using the SpinTek unit. The permeate flux varied over the first hour and leveled off at an average of 317 gal/ft^2 day over the last two hours of operation. Permeate turbidity ranged from 0.1 to 1.0 NTU when the thickened sludge was treated in this run. The average permeate turbidity in this experiment was 0.48 NTU. In the kaolinite run, the permeate flux increased with both time and total kaolinite concentration. The concentrate TSS increased corresponding with increasing kaolinite concentration. Additionally, the permeate turbidity was stable at an average of 0.37 NTU over the course of this run. In the biosorbent run, the permeate flux decreased as the ionic strength was increased. However, the permeate turbidity remained steady throughout each dose.

In the Surfactant Run, an amide surfactant provided by IGT (IGT-FF52) was treated using the SpinTek unit. Because no analytical method is available for measuring the micelle and dissolved amide concentration in the amide solution, turbidity and absorbance of amide solution at different concentrations were measured. A linear relationship between turbidity and concentration was observed, for both permeate and concentrate. A linear relationship was also observed when the adsorbance was investigated at different concentrations of permeate and concentrate. As a result, it was theorized that the turbidity was an indicator of micelle concentration in the amide solution. The adsorbance was determined to be an indicator of the dissolved amide content. The effects of system shut down on the permeate flux were determined using a PV-5k and a PV-10k membrane. For the PV-5k membrane, the flux decreased after each shut down period and was not restored when the system was restarted. The PV-5k membrane was then cleaned by running a cleaning solution of 30mL *Dawn* dishwashing detergent in 5 gallons of distilled water through the system and by hand cleaning with a combination of *Biz* and *Dawn*. The clean water flux was restored, but the permeate flux decreased when the amide solution was run through the system. When the PV-10k was examined under similar experimental conditions, the permeate flux decreased after each shutdown period, but was restored after being cleaned and restarted.

1.0 INTRODUCTION

The use of sorbents (biosorbents, powdered activated carbon) and cleaning agents (surfactants, chelating agents) to decontaminate surfaces such as pipes, walls, *etc*. represents a unique and cost-effective method for remediating DOE facilities. As envisioned, the sorbent or cleaning agents would be added to a liquid (most likely water) and the solution would be contacted with the contaminated surface. For example, a pipe that is contaminated with inorganic and organic contaminants can be contacted with a solution containing a surfactant (for organics) and a chelating agent (for inorganics). After removal of the surface-bound contamination by the cleaning agents, a solid sorbent can be added to liquid to bind the cleaning agent-contaminant compound. A major problem associated with this approach is the efficient separation of the sorbent/cleaning agent from the liquid phase using conventional separation techniques.

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks set on a hollow rotating shaft inside a cylindrical housing. The waste stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate is forced through the membrane and is collected in the hollow shaft and discharged. The concentrate exits at the edge of the membrane packs. The centrifugal action continuously cleans the membrane surface decreasing cleaning chemical use and maintenance, increasing the volume of waste treated per area of membrane, and increasing the contaminant/solids concentration in the residual (*i.e.* increases volume reduction factor). SpinTek's centrifugal membrane technology represents an improvement over conventional ultrafiltration technology and increases the number of waste streams that can be treated using membrane treatment.

2.0 PURPOSE

The purpose of this research is to investigate the feasibility of using sorbents (biosorbents, powdered activated carbon) and cleaning agents (surfactants, chelation agents) to decontaminate surfaces (*e.g.*, pipes) and then to separate the sorbents/cleaning solutions from the aqueous phase using the SpinTek centrifugal membrane technology. The result of this work will be an effective decontamination/decommissioning process that can be used for inorganic (*e.g.* radionuclides and heavy metals) and organic contaminants.

3.0 BACKGROUND

Background information contained in this report pertains only to membrane technology. The Institute of Gas Technology (IGT) will provide, in a separate report, the required background information on the development/use of sorbents/cleaning solutions.

SpinTek, a California-based company, has developed and patented a cross-flow technology that improves upon the conventional membrane process. The SpinTek system uses a series of flat, round membrane disks packs set on a hollow rotating shaft inside a cylindrical housing. The fluid stream enters the membrane chamber under pressure and is distributed across the membrane surfaces by centrifugal and hydraulic action. Permeate ("clean" liquid) is forced through the membrane and is collected in the hollow shaft and then discharged. The concentrate (liquid containing the contaminants) exits at the edge of the membrane packs.

With time, the membrane surface will become fouled resulting in a decrease in the permeate flow rate. To reduce fouling in conventional membrane systems, a large portion of the concentrate is recycled back to the membrane unit producing large liquid velocities near the membrane surface. The large velocities increase turbulence which reduces the thickness of the fouling layer. In centrifugal membrane systems, the rotation of the membrane disk induces the turbulence (*i.e.*, shear force near membrane surface) required to minimize the thickness of the fouling layer. In conventional membrane systems, maximum surface velocities of about 15 ft/s are possible while with centrifugal systems surface velocities of 60 ft/s are typical. As the concentrate thickens with treatment time, conventional systems are not able to maintain the high velocities because of the difficulty in pumping viscous material at large flow rates. Because the centrifugal membrane system does not rely on pumping to produce the required surface velocities, extremely concentrated wastes can be treated.

Membrane systems are typically operated in either the semi-batch mode or the batch mode. In the semi-batch mode, fresh waste is added to the system at a rate equal to the permeate production rate. In the batch-mode, which normally occurs at the end of semi-batch operation, no fresh waste is added to the system and the concentrate remaining from semi-batch operation is concentrated further ("batch down"). During batch-down, large concentration factors are achieved (permeate is still being produced reducing the concentrate volume).

All membrane technologies are volume reduction technologies because contaminants are not degraded or destroyed. Thus, an important measure of the efficiency of membrane technology is the concentration factor (CF). The hydraulic-based CF during semi-batch and batch-down operation are calculated using the following two equations:

Semi-Batch Operation

$$CF_{SB} = 1 + V_{perm}/V_{feed\ tank}$$

Batch-Down Operation

$$CF_{BD} = CF_{SB} \times [V_{feed\ tank}/(V_{feed\ tank} - V_{perm})]$$

where,

CF_{SB} = hydraulic-based concentration factor during semi-batch operation.
V_{perm} = volume of permeate produced, gal.
$V_{feed\ tank}$ = volume of the feed tank, gal.
CF_{BD} = hydraulic-based concentration factor during batch-down operation.

Concentration factors are expressed as 1X, 2X, *etc.*, and increase with treatment time. CFs in excess of 100X are obtainable with the membrane technology. Another measure of treatment efficiency is the permeate flux (volume of permeate/membrane area per time). The permeate flux for a given membrane is dependent on the waste's viscosity, solids content, and temperature. These waste characteristics can also change during treatment.

Membranes that are used for ultrafiltration are characterized by the molecular weight of a molecule that is not allowed to pass through the membrane. For example, the membrane used in this quarter's research had a molecular weight cut-off (MWCO) of 100,000. In theory, compounds having a molecular weight greater than the MWCO would be retained by the membrane and compounds with molecular weights less than the MWCO would pass through the membrane and be contained in the permeate. It should be noted that the MWCO designation is somewhat misleading because a molecule having a molecular weight less than the membrane's MWCO may still be retained by the membrane because its unique geometry.

4.0 METHODOLOGY

Centrifugal membrane technology will be tested using several sorbents/cleaning solutions. Several membrane types will be investigated. A 0.1um pore size ceramic membrane will be used in the thickened sludge, kaolinite, and biosorbent experiments and 5k and 10k MWCO polymeric membranes will be used in the amide experiments. For each amide experiment, the clean water flux (CWF) will be determined for the virgin membrane. The CWF serves as a baseline indicator to determine if the membrane is 1) permanently fouled and 2) sufficiently cleaned. For each waste-membrane combination, the SpinTek system will be operated in recycle mode. The following variables will be monitored during technology testing:

1. Membrane flux.
2. Temperature and pressure.
3. Turbidity of all liquids in each run and both turbidity and adsorbance in the amide runs.

Permeate flux, contaminant(s) concentration in the permeate, concentration factor, operational ease, and the membrane's physical/chemical resistance and cleanability will be used to judge the effectiveness of the process. Several runs will be conducted for each size membrane in the amide experiments. The thickened sludge, kaolinite, and biosorbent runs will examine the effects of ionic strength on the membrane's durability and the effects of simulated and actual biosorbent materials on the SpinTek system's operational parameters. Quality assurance/quality control practices, as outlined in US EPA's Test Methods for Evaluating Solids Wastes, will be followed. The use standard analytical methods (*i.e.,* ASTM, Standard Methods for the Measurement of Water and Wastewater, etc.) will also be followed.

5.0 WORK PERFORMED DURING THIS PERIOD

During the period 6/31/96 to 9/30/96, the following waste streams were treated using SpinTek's centrifugal ultrafiltration system: 1) IGT's biosorbent material, 2) thickened waste activated sludge, 3) kaolinite, and 4) IGT's amide-based surfactant,. For the biosorbent, waste activated sludge, and kaolinite, a 0.1 um ceramic membrane was used. For the amide-based surfactant, two polymeric membranes having a molecular weight cutoff of 5,000 and 10,000 were employed.

6.0 RESULTS AND DISCUSSION

In this chapter, results from the waste activated sludge, the kaolinite, and the biosorbent experiments and the amide experiments will be presented and discussed. Three experiments were conducted using a thickened sludge, kaolinite, and a biosorbent material. Two experiments were conducted using an amide-based surfactant. A schematic of the experimental apparatus is provided in Figure 1 (Figures are included at the end of the text portion of Section 6.0).

Thickened Waste Activated Sludge

Ten gallons of a thickened sludge/distilled water mixture were treated for three hours under the following conditions: vessel pressure = 42 psi, rotational speed = 1750 rpm, feed temperature = 110 °F, return flow = 0.8 - 1gal/min, operation mode = recycle, and initial TSS concentration = 0.1%. Prior to conducting this experiment, the thickened sludge was stored in the refrigerator for two days, to hinder the proliferation of live biomass in the sample. The permeate flux versus operation time is presented in Figure 2. The flux varied between 298 and 328 gal/ft^2 day over the first one hour and then reached a plateau at about 317±7 gal/ft^2 day. The feed tank temperature versus operational time is presented in Figure 3. The temperature was controlled, using a heat exchanger, at 110 ± 2°F. There was a drop in temperature to 108°F at 0.5 hours. A corresponding decrease in the permeate flux was observed at this point. The flux stabilized when the temperature was restored to 110 °F. Permeate turbidity versus operational time is presented in Figure 4. The turbidity ranged from 0.2 to 1.2 NTU, with its maximum reading occurring at 2.2 hours. The average turbidity over the course of this run was 0.48 ± 0.28 NTU.

Kaolinite

Ten gallons of a kaolinite/distilled water mixture was treated for five and an half hours under the following condition: vessel pressure = 42psi, rotational speed = 1750rpm, feed temperature = 110°F, return flow = 0.8-1gal/min, operation mode = recycle and kaolinite concentration 0.8% for the first two and an half hours and 3.2% for the balance of the experiment. The permeate flux versus operation time is presented in Figure 5. Permeate flux increased over the entire run, with a plateau region between 1 and 1.5 hours. The average flux was 717 ± 58 gal/ft^2 day for the first kaolinite concentration and 896 ± 28 for the second. The feed tank temperature versus operational time is presented in Figure 6. The feed tank temperature was maintained at 110 ± 2°F using a heat exchanger, as described earlier. The permeate turbidity versus operation time is presented in Figure 7. The turbidity was stable at an average of 0.37 ± 0.11 NTU over the course of the run. The maximum turbidity observed in this run was 0.61 NTU and the minimum turbidity was 0.21 NTU. The permeate and concentrate TSS versus operational time is presented in Figure 8. The permeate TSS remained steady throughout the run. The concentrate TSS ranged from its low of 5200 mg/L at the 0.8% kaolinite concentration to a high of 30,000 mg/L at the 3.2% kaolinite concentration. Theoretically, there should have been a four-fold increase in concentrate TSS when the kaolinite concentration was increased from 0.8 to 3.2%. However, an increase of six times the 0.8% TSS was measured.

IGT's Biosorbent

Five and a half gallons of a biosorbent/distilled water mixture was treated for 25 hours. The ionic strength was changed by adding different dosages of $NaNO_3$ (0.01N, 0.05N, 0.1N, 0.5N and

1.0N). The system was run in recycle mode for four hours at each dose with the operating parameters detailed above. The flux versus operational time is presented in Figure 9. The permeate flux decreased as the ionic strength increased. The greater ionic strength provided more ions to neutralize the charge of the electric double layer around the biosorbent surface. As a result, biosorbent particles had more opportunities to bind with each other. This may also provide additional opportunities for the biosorbent in solution to combine with biosorbent particles on the gel layer. This may have increased the gel layer and as a result, decreased the flux. The average permeate flux versus $NaNO_3$ dosage is presented in Figure 10. The average flux was: 201gal/ft day at 0.01N, 184gal/ft^2 day at 0.05N, 179gal/ft^2 day at 0.01N, 165 gal/ft^2 day at 0.5N and 152 gal/ft^2 day at 1.0N. The feed tank temperature versus operation time is presented in Figure 11. The feed tank temperature was maintained at 110 ± 2°F using a heat exchanger, as described earlier. The turbidity versus operational time is presented in Figure 12. The turbidity at each ionic strength remained steady at 0.2 NTU.

Amide-Based Surfactant - 5000 MWCO Polymeric Membrane

A 3.96 gallon (15L) solution of 1% (wt.) IGT-FF52 amide in deionized water was treated for 5 days using a PV-5k membrane under the following conditions: vessel pressure = 42psi, rotational speed = 1750rpm, feed temperature = 100 ^{0}F and 110 ^{0}F, return flow = 1 - 1.2gal/min and operation mode = recycle. The operational procedure for this run is summarized in Table 1. Prior to beginning this run, a CWF of 581gal/ft^2 day was measured for the virgin 5k polymeric membrane. After the run started, concentrate and permeate samples were taken and diluted at different dilution factors to measure their turbidity and absorbance. A linear relationship was observed between turbidity and relative concentration for both concentrate and permeate. A similar relationship exists between adsorbance and relative concentration. These data are presented in Figures 13 through 16, respectively. It was determined that the turbidity was an indicator for micelle concentration in the amide solution and adsorbance was an indicator of the dissolved amide concentration in the amide solution.

During the run, the feed tank temperature was first maintained at 100 ^{0}F until the flux was stable then the feed tank temperature was then increased to 110 ^{0}F. The feed tank temperature versus operational time is presented in Figure 17. The permeate flux versus operational time is presented in Figure 18. The flux increased when the temperature was raised. After the flux stabilized at 110 ^{0}F, the system was shut down overnight with the amide solution remaining in the system to observe the effects of system shutdown on membrane fouling. When system was restarted, the permeate flux was not restored at either 100 ^{0}F or 110 ^{0}F. The system was then shut down again and cleaned by a running cleaning solution (30 mL of Dawn dishwasher detergent in 5 gallons of distilled water) through the system. A CWF of 287gal/ft^2 day was measured. The flux was still not restored when the system was restarted. The system was shut down again and the membrane was taken out after flushing and cleaning manually with a sponge soaked in a solution of 36.2 g Biz detergent and 6 ml of Dawn dishwasher detergent in 1 gallon of tap water. A CWF of 481gal/ft^2 day was measured. However, the permeate flux still was not restored after restarting the system. The average flux and turbidity at each temperature on each day is presented in Table 2. The permeate and concentrate turbidity versus operational time is presented in Figure 19. The permeate and concentrate turbidity decreased over the first hour the system was running and then remained stable. The explanation for this is that the shear force in the membrane housing destroyed some of the micelles in the amide solution when the system was started and then reached equilibrium for the rest of the run.

Amide-Based Surfactant - 10000 MWCO Polymeric Membrane

A 3.96 gallon (15L) solution of 1% (wt.) IGT-FF52 amide in deionized water was treated for 3 days using a PV-10k membrane under the following conditions: vessel pressure = 42psi, rotational speed = 1750rpm, feed temperature = 110 ^{0}F, return flow = 1-1.2gal/min, and operational mode = recycle. A summary of the experimental procedure is presented in Table 3. Prior to beginning this run, a CWF of 459gal/ft^2 day was measured for the virgin 10k polymeric membrane.

The system was started and the feed tank temperature was allowed to increase to 110 ^{0}F. Feed tank temperature versus operational time is presented in Figure 20. The permeate flux versus operational time is presented in Figure 21. After the permeate flux had stabilized for 12 hours, the system was shut down and cleaned by running cleaning solution (30ml *Dawn* dishwasher detergent in 5 gallons of distilled water). A CWF of 357gal/ft^2 day was measured. The permeate flux was restored after the cleaning. Then the system was shut down with amide solution remaining in the system overnight. When the system was restarted, the flux was restored within one hour. The turbidity, adsorbance of both the concentrate and permeate was measured frequently. The turbidity and adsorbance versus operational time is presented in Figure 22. The turbidity of permeate decreased during the first day of the run and stabilized over the second and third days. The adsorbance of the permeate remained stable for the whole run. The concentrate turbidity and adsorbance remained stable for the course of the run. The average flux and turbidity for each day of the run is presented in Table 4.

The permeate flux of PV-10k was much lower than that of PV-5k, although the PV-10k has a greater MWCO. However, the average permeate turbidity of PV-10k was higher than that of PV-5k. It is hypothesized that since the MW of IGT-FF52 amide micelle is close to the MWCO of PV-10k membrane (IGT correspondence), micelles tended to either pass through the membrane or become lodged in the pore. For the PV-5K membrane, the micelles could not enter the pores because of the size differential thus, the permeate turbidity was lower and the flux higher than those observed with the PV-10K membrane.

Table 1.
Summary of Experimental Procedure for Surfactant Run 1

Date	Aug. 23	Aug. 24	Aug. 26	Aug. 27	Aug. 28
Comments	System was run for 10 hours then shut down without cleaning	System was run for 8 hours then shut down without cleaning	System was run for 5 hours then shut down and cleaned with cleaning solution	System was run for 6 hours then shut down and membrane was cleaned manually	System was run for 4 hours then shut down and amide solution was drained.

Table 2.
Summary of Permeate Results for PV-5k Membrane

Permeate	Aug. 23		Aug. 24		Aug. 26		Aug.27		Aug. 28	
	100^{0}F	110^{0}F	100^{0}F	110^{0}F	100^{0}F	110^{0}F	100^{0}F	110^{0}F	100^{0}F	110^{0}F
Flux,gal/ft^2-d	329±5	336±8	235±3	268±6	170±5	181±1	120±3	122±1	131±6	-
Turbidity, NTU	0.59± 0.11	0.55± 0.07	0.65± 0.10	0.71± 0.07	0.78± 0.04	0.78± 0.04	0.20± 0.01	0.25± 0.05	1.33± 0.25	-
Turbidity Removal(%)	99.6	99.7	99.6	99.6	99.6	99.6	99.9	99.9	99.3	-

Table 3.
Summary of experimental procedure for Surfactant run 2

Date	Aug. 29-Sep.1	Sep. 3	Sep. 4
Comments	System was run for 35 hours then shut down and cleaned with cleaning solution	System was run for 5 hours then shut down and not cleaned	System was run for 9 hours and then shut down and cleaned

Table 4.
Average of permeate flux and turbidity for PV-10k membrane

	Average ± standard deviation		
	Aug. 29	Sep. 3	Sep.4
flux(gal/ft^2 -d)	157±15	182±14	162±3
turbidity(NTU)	1.94±0.73	0.85±0.09	0.87±0.15
turbidity removal (%)	98.9	99.5	99.5

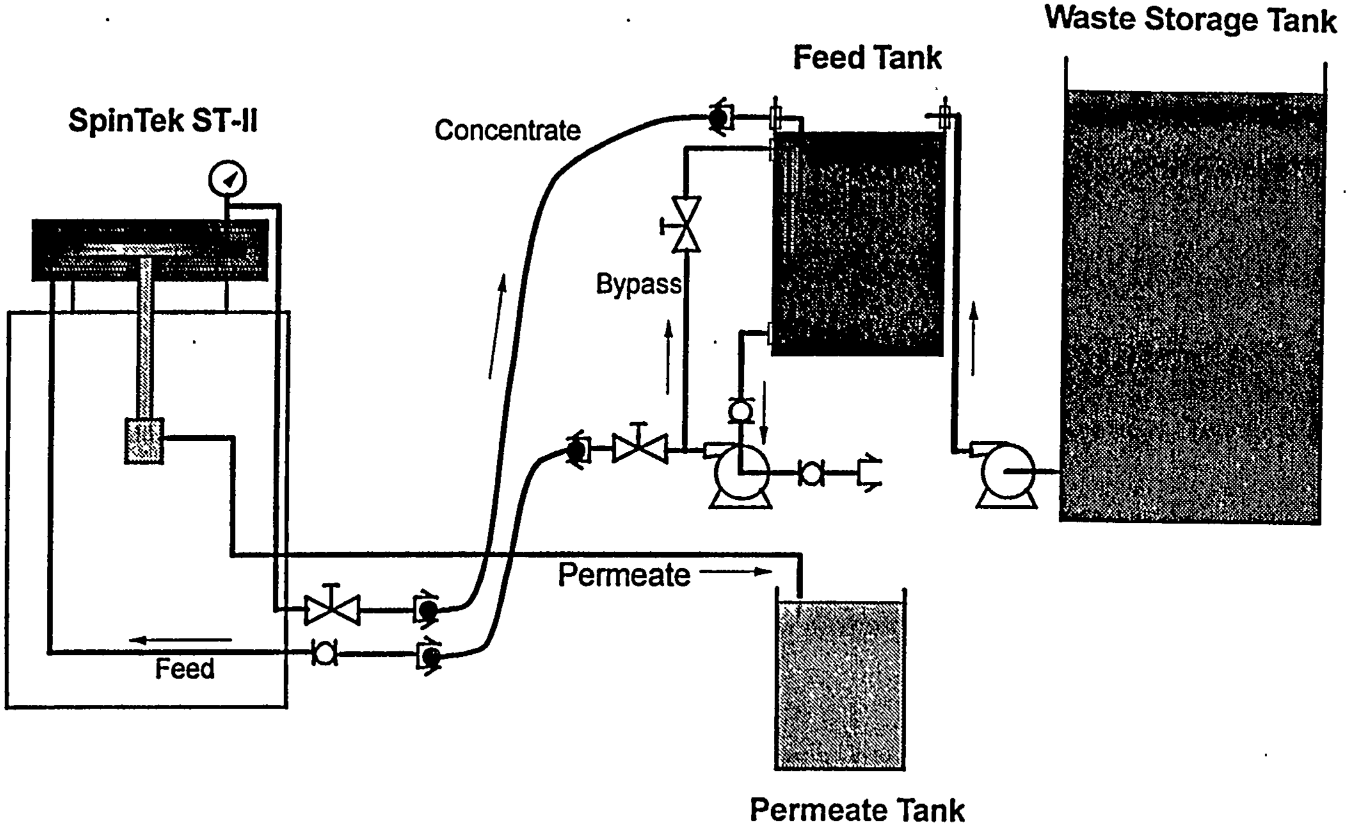

Figure 1. Schematic of Experimental Apparatus.

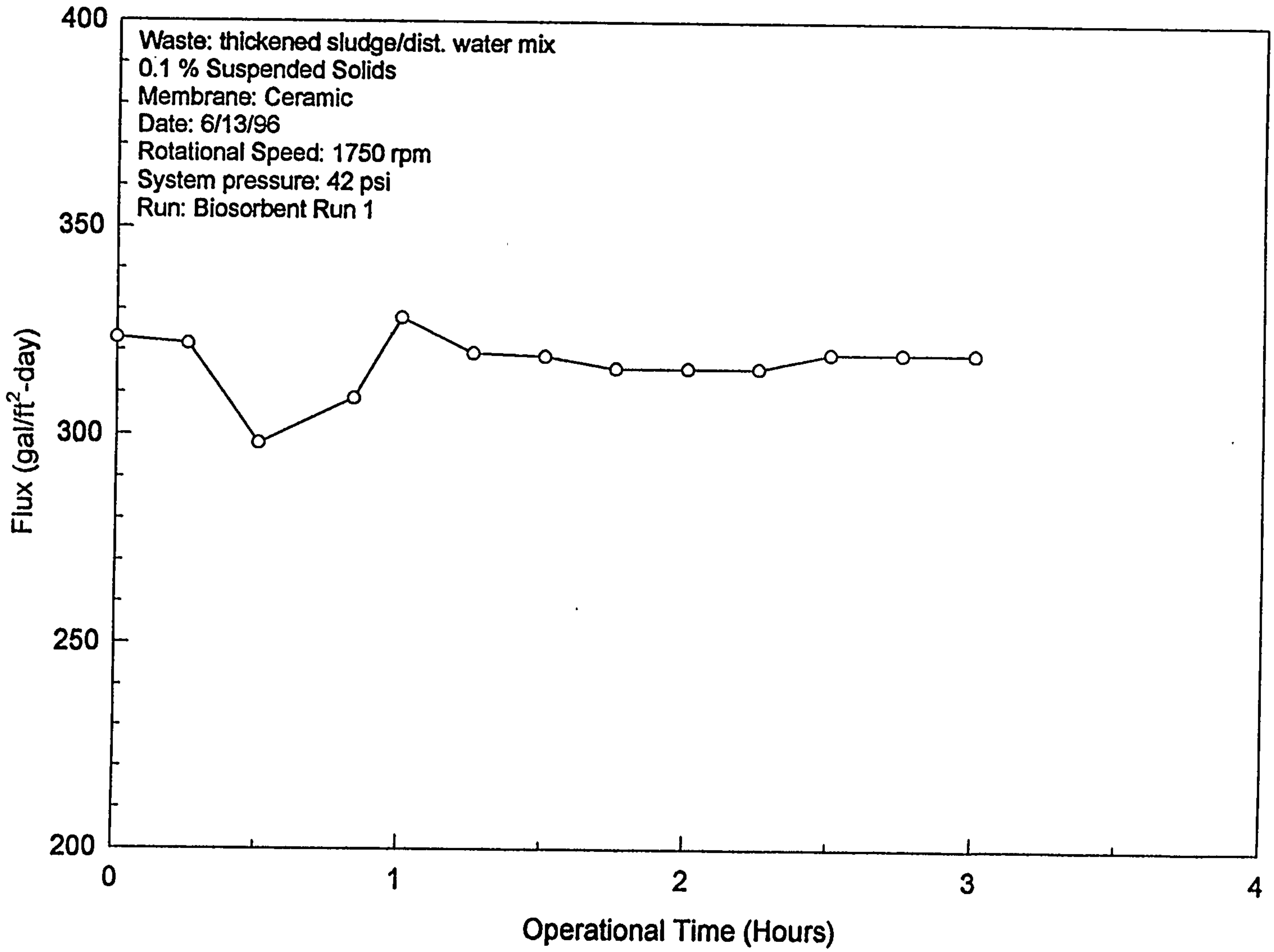

Figure 2. Permeate Flux Versus Operational Time For Thickened Waste Activated Sludge Run.

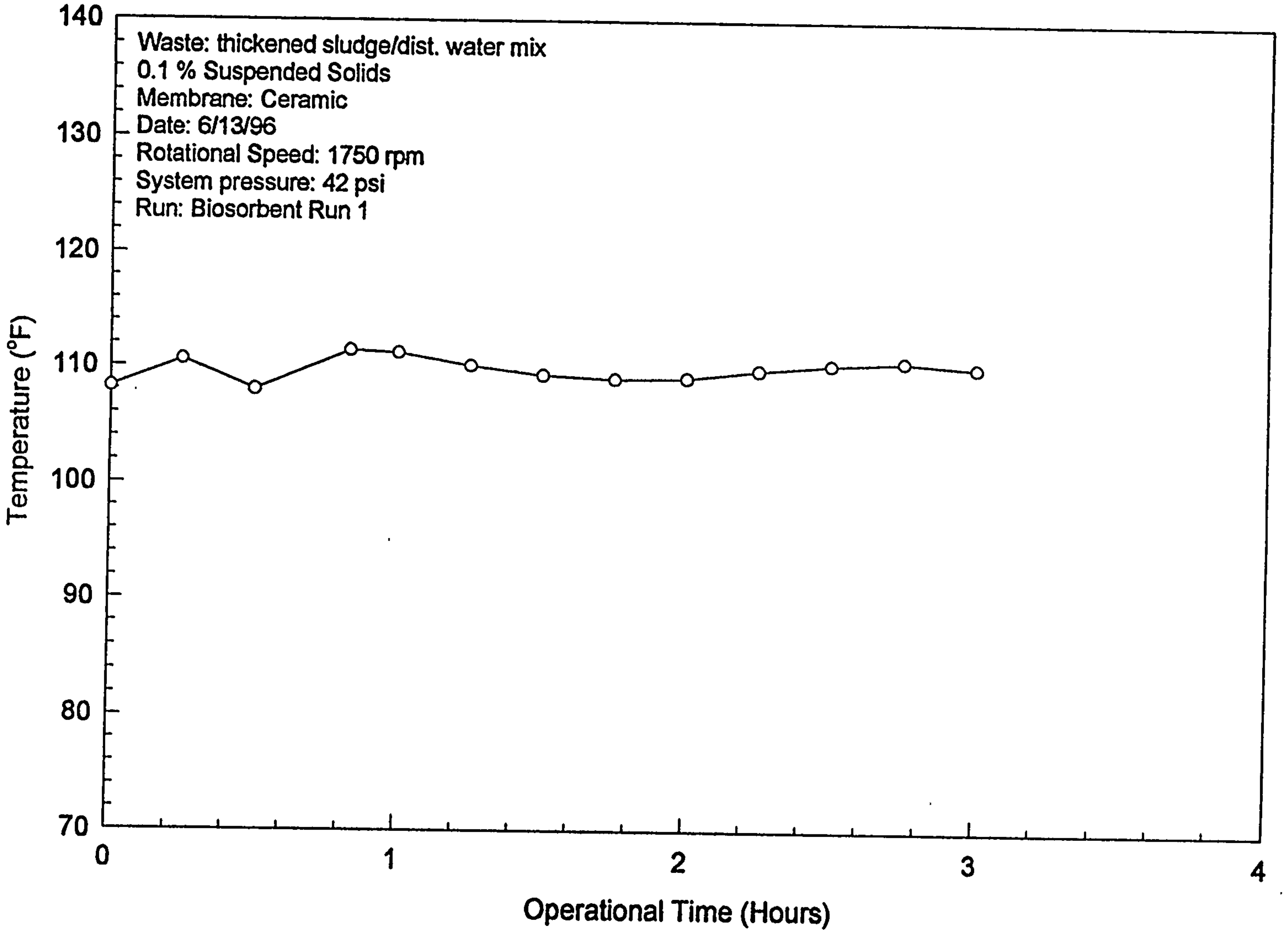

Figure 3. Feed Tank Temperature Versus Operational Time For Thickened Waste Activated Sludge Run.

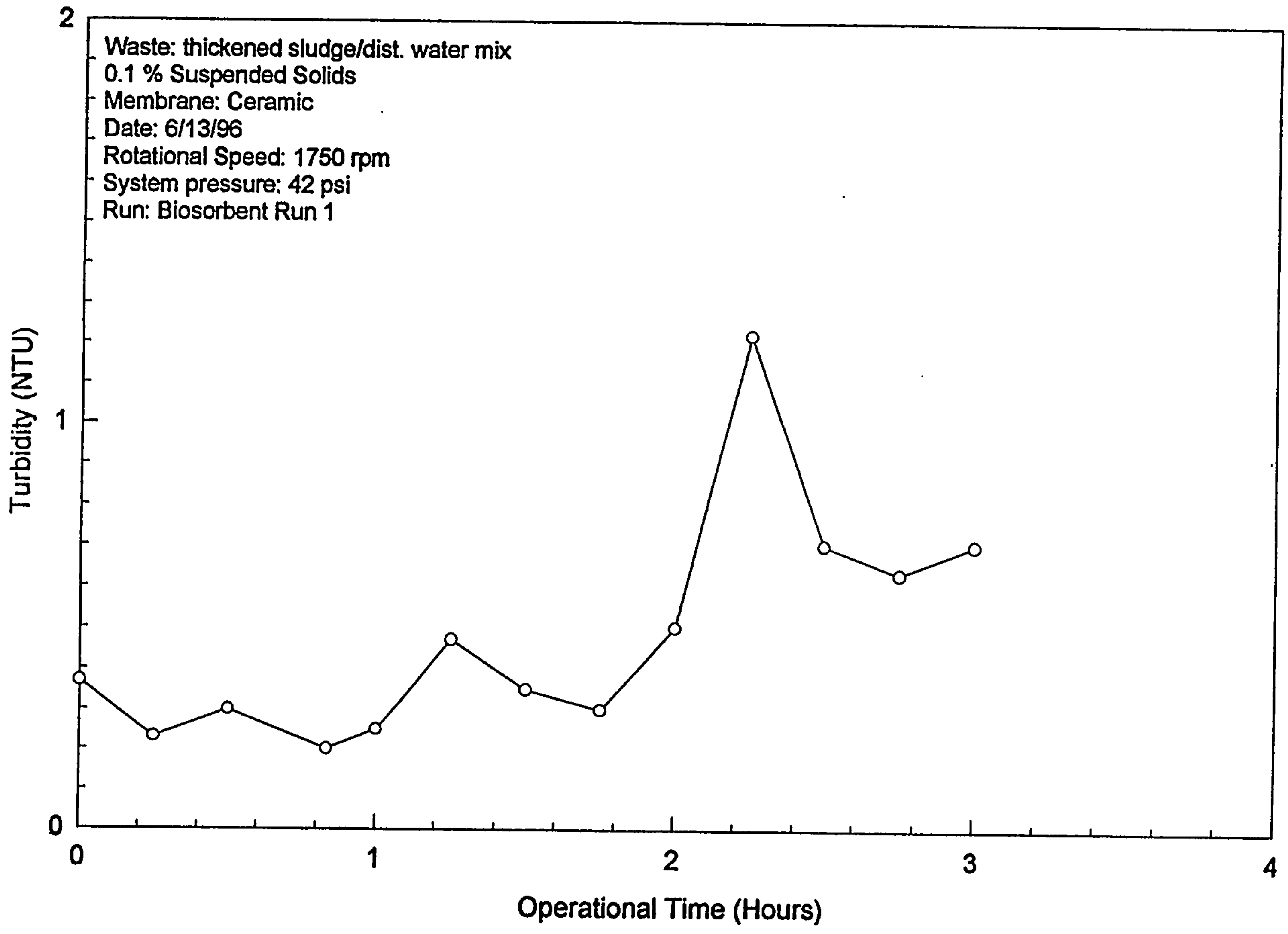

Figure 4. Permeate Turbidity Versus Operational Time For Thickened Waste Activated Sludge Run.

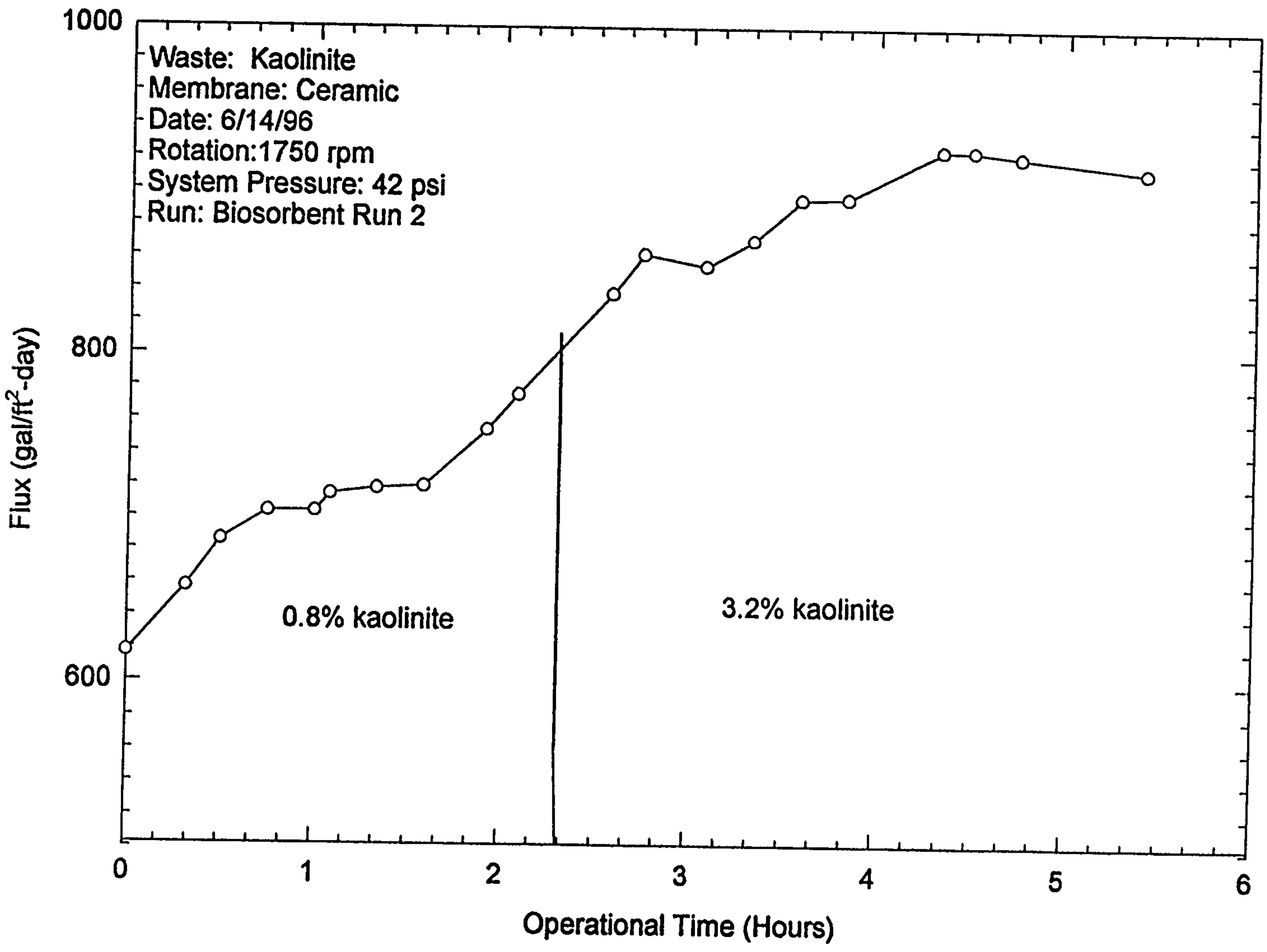

Figure 5. Permeate Flux Versus Operational Time For Kaolinite Run.

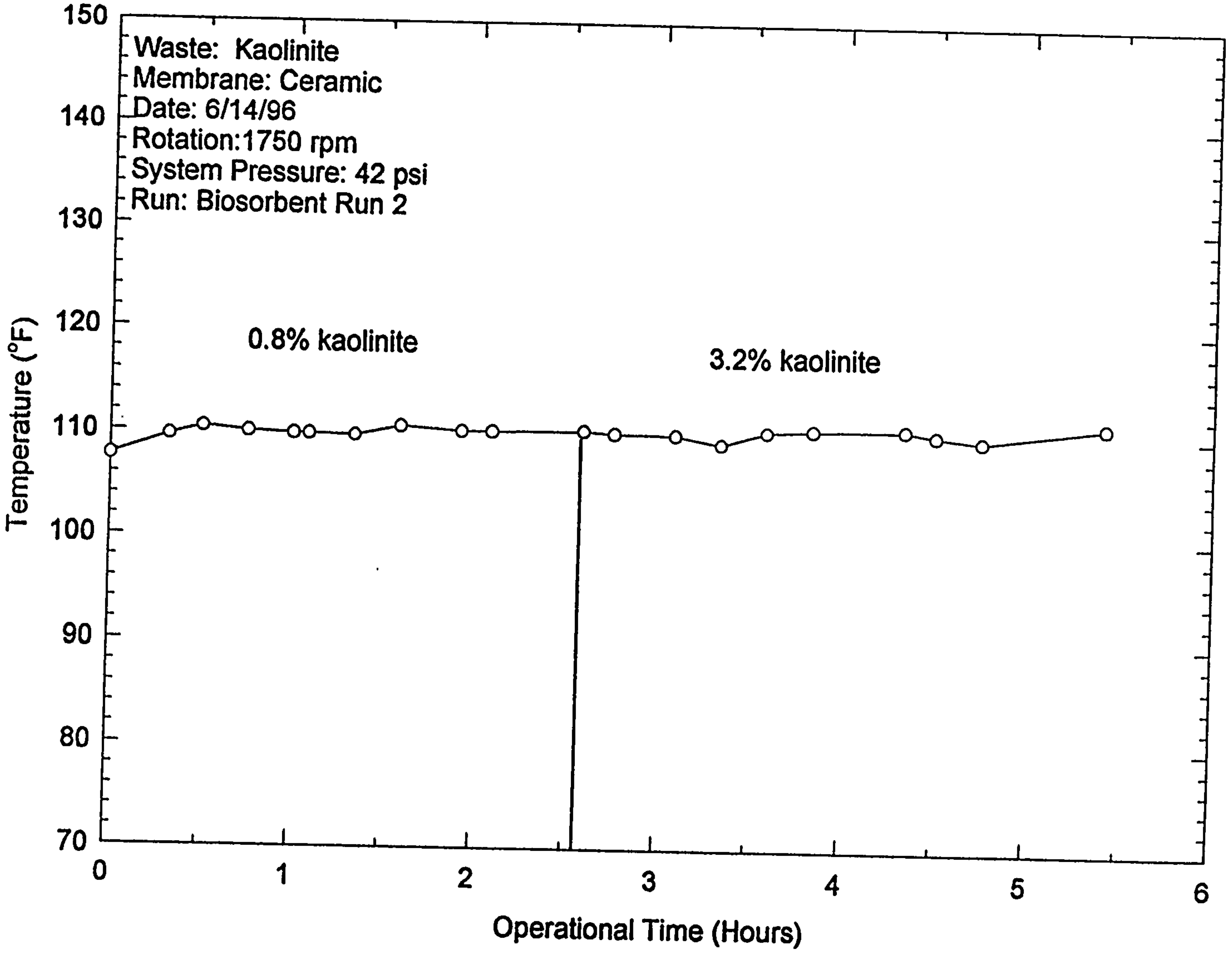

Figure 6. Feed Tank Temperature Versus Operational Time For Kaolinite Run.

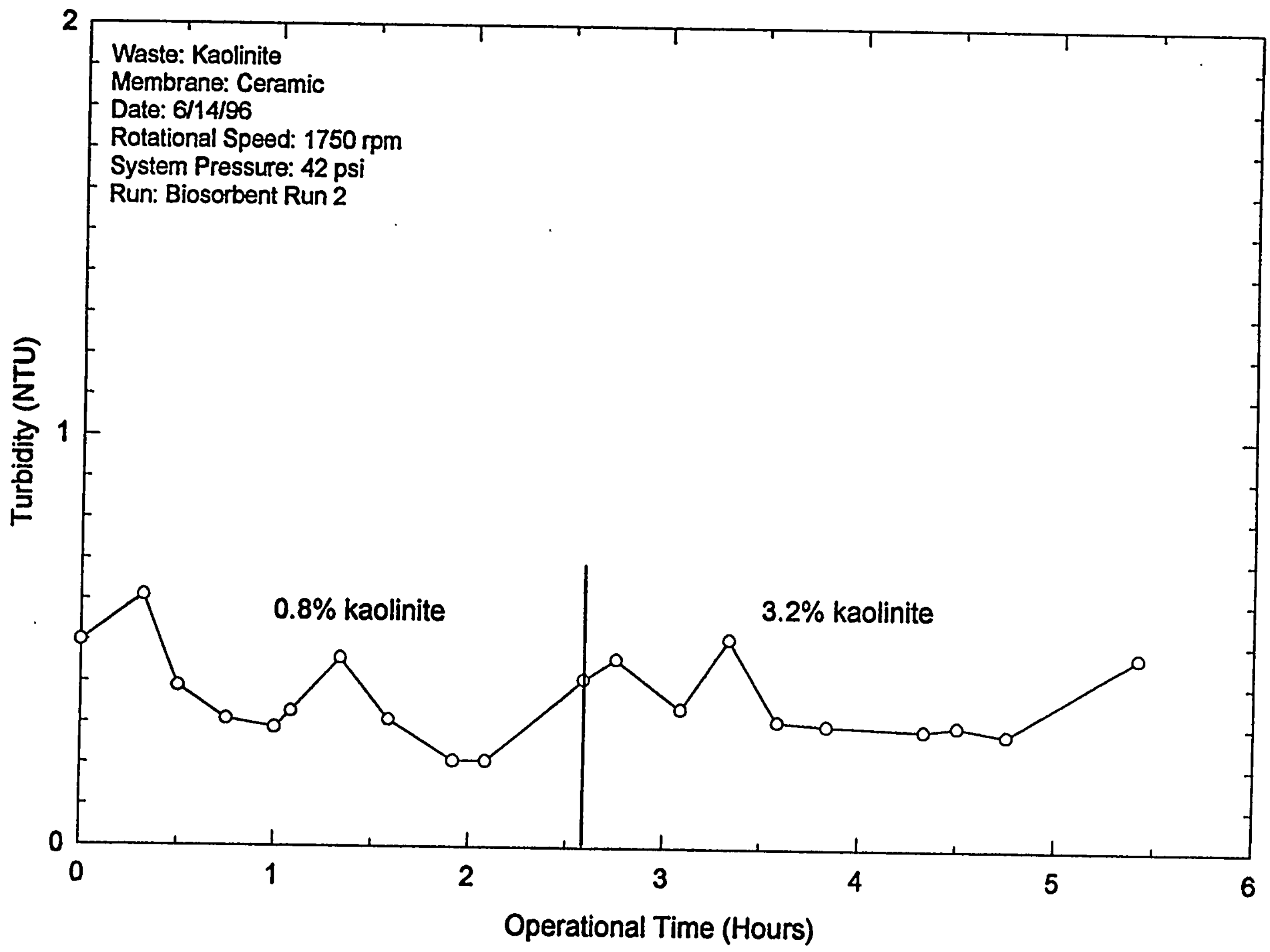

Figure 7. Permeate Turbidity Versus Operational Time For Kaolinite Run.

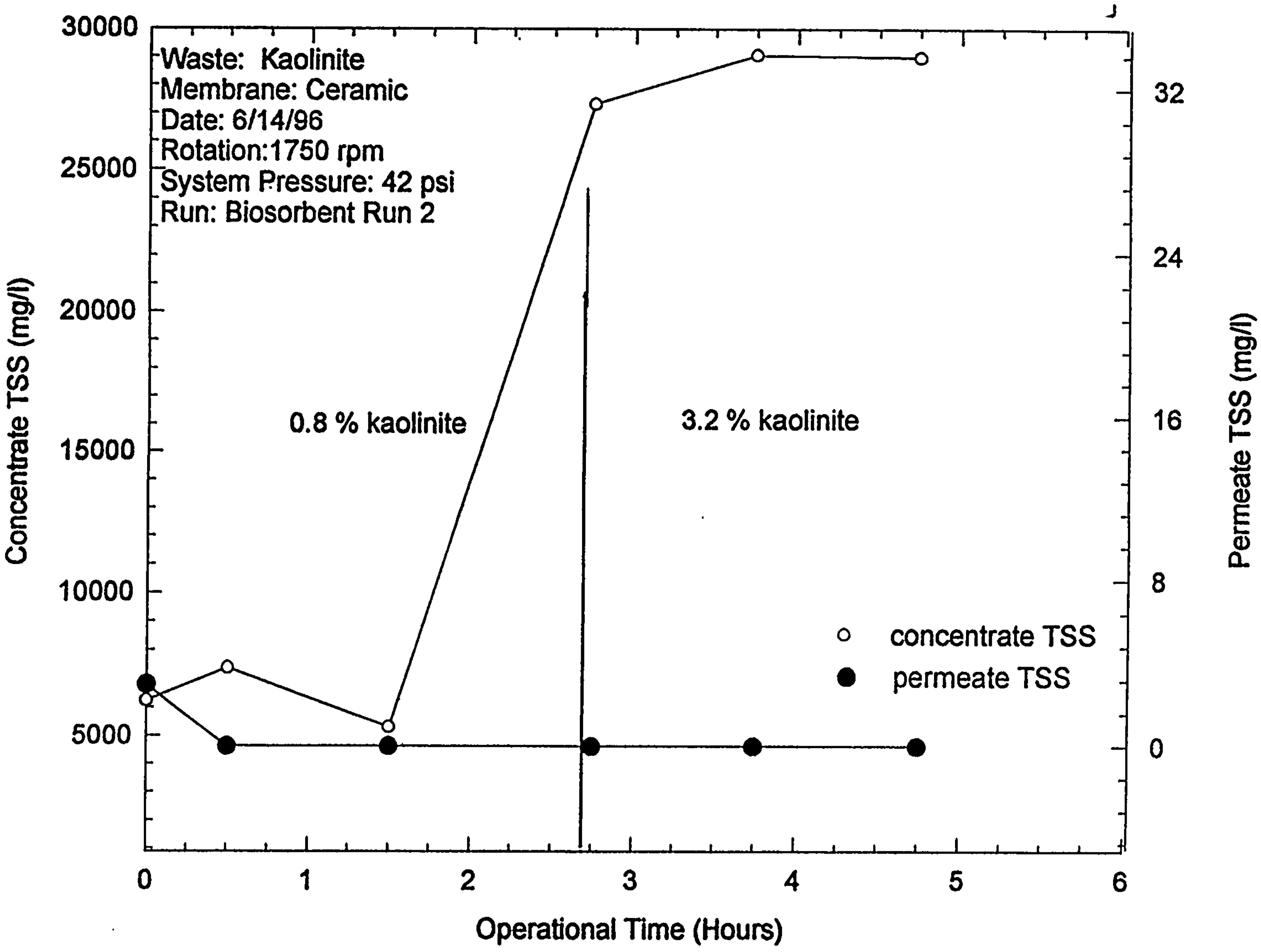

Figure 8. Permeate and Concentrate TSS Versus Operational Time For Kaolinite Run.

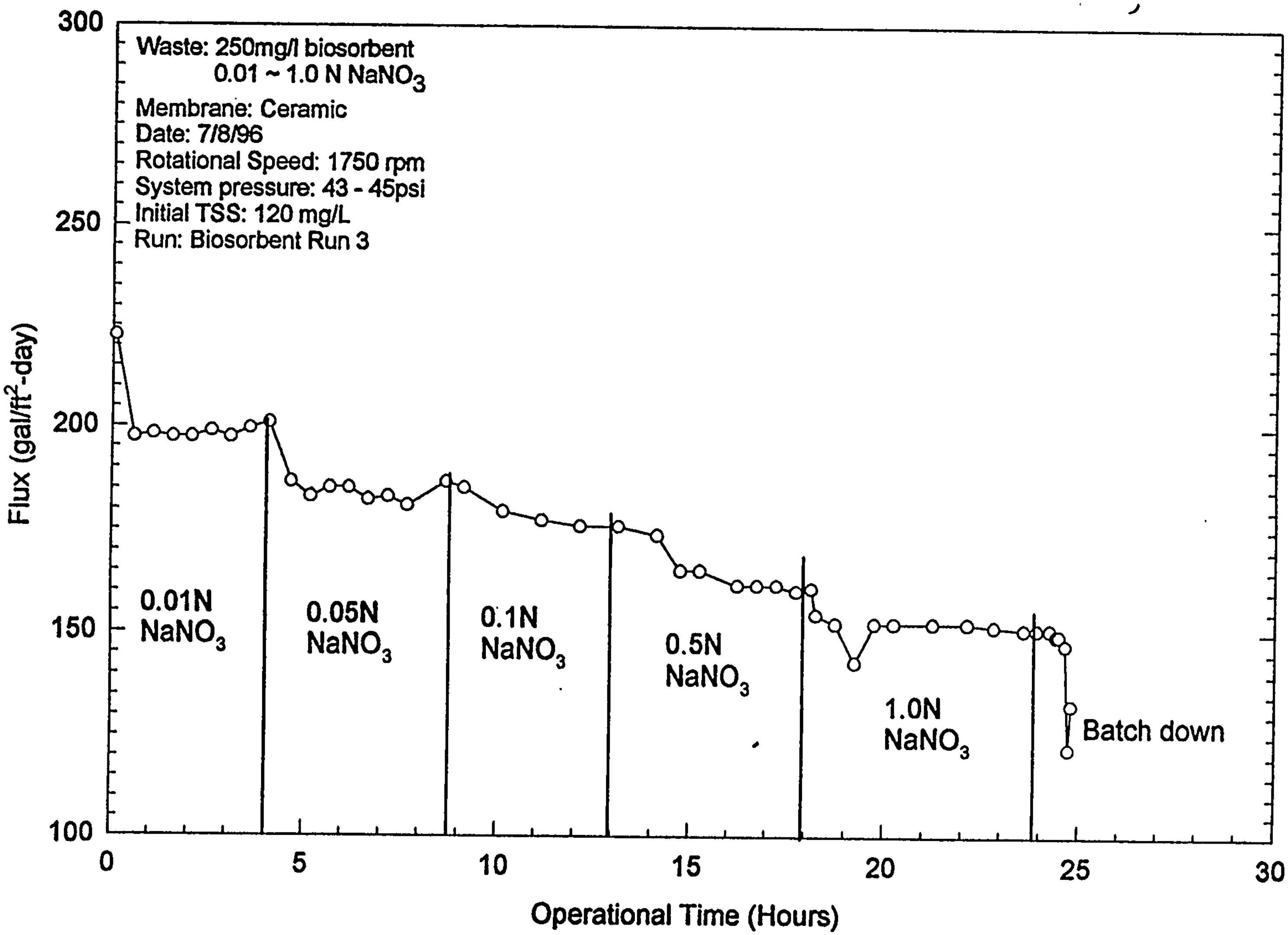

Figure 9. Permeate Flux Versus Operational Time For Biosorbent Run.

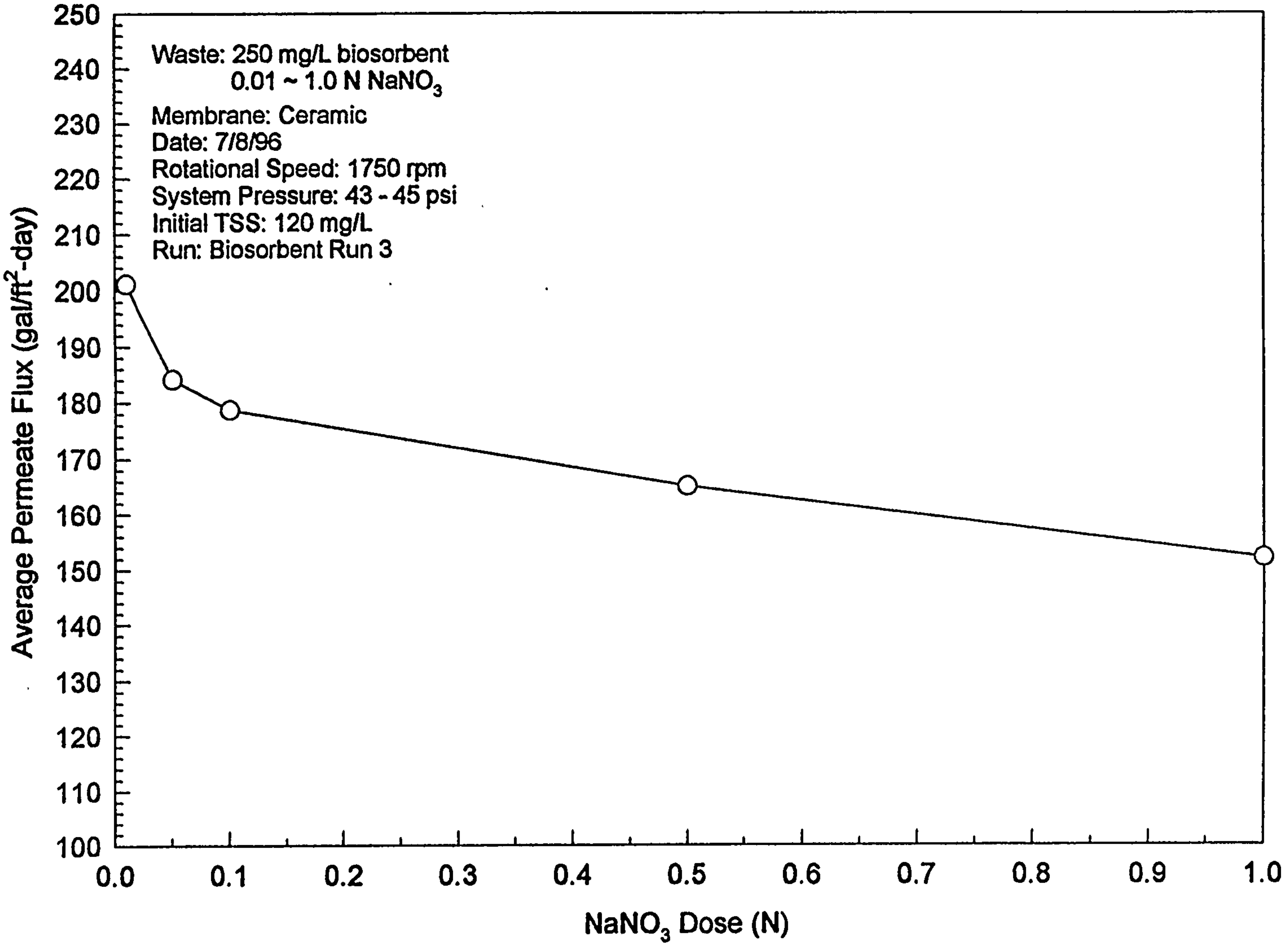

Figure 10. Average Permeate Flux Versus $NaNO_3$ Dosage For Biosorbent Run.

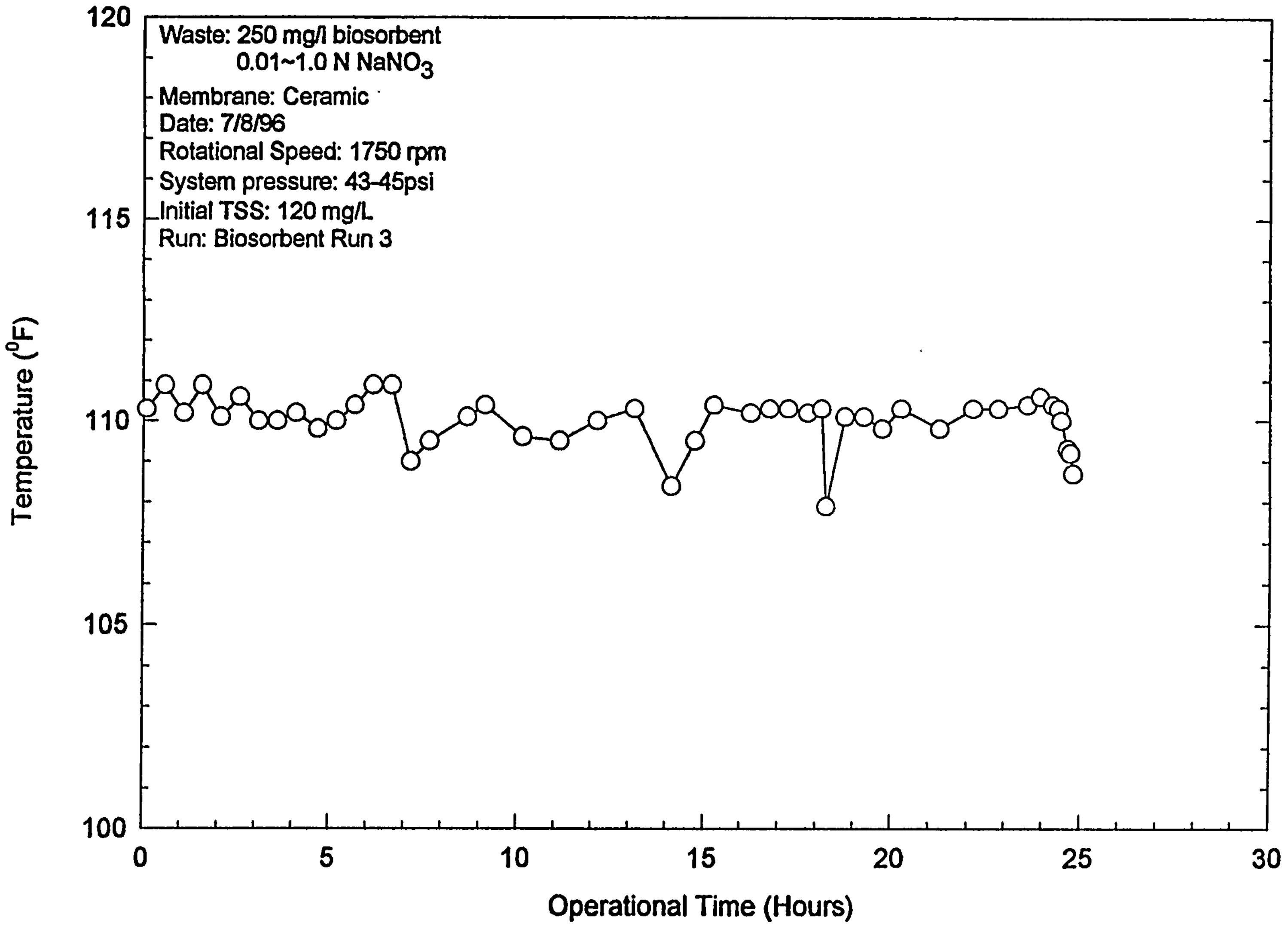

Figure 11. Feed Tank Temperature Versus Operational Time For Biosorbent Run.

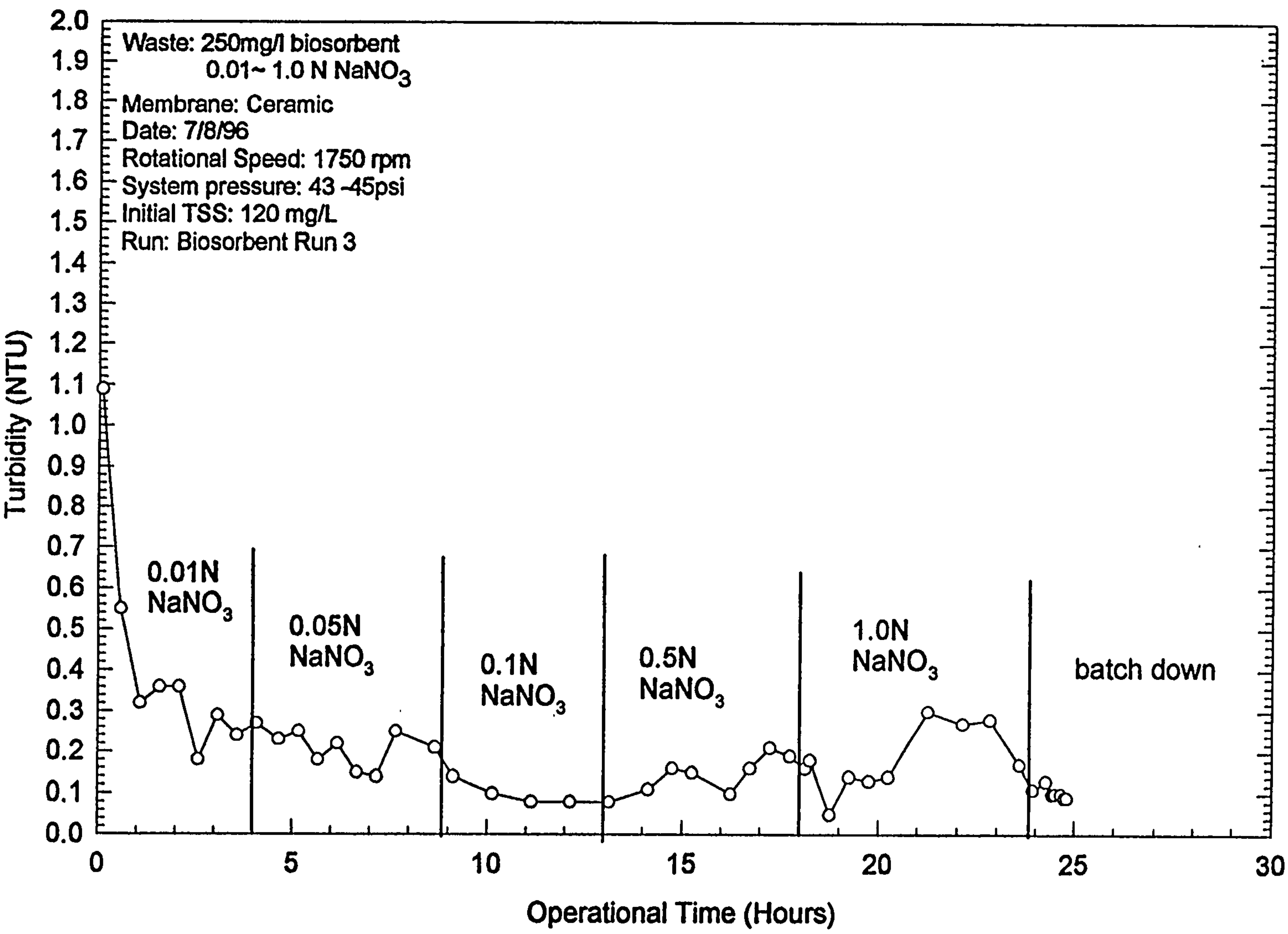

Figure 12. Permeate Turbidity Versus Operational Time For Biosorbent Run.

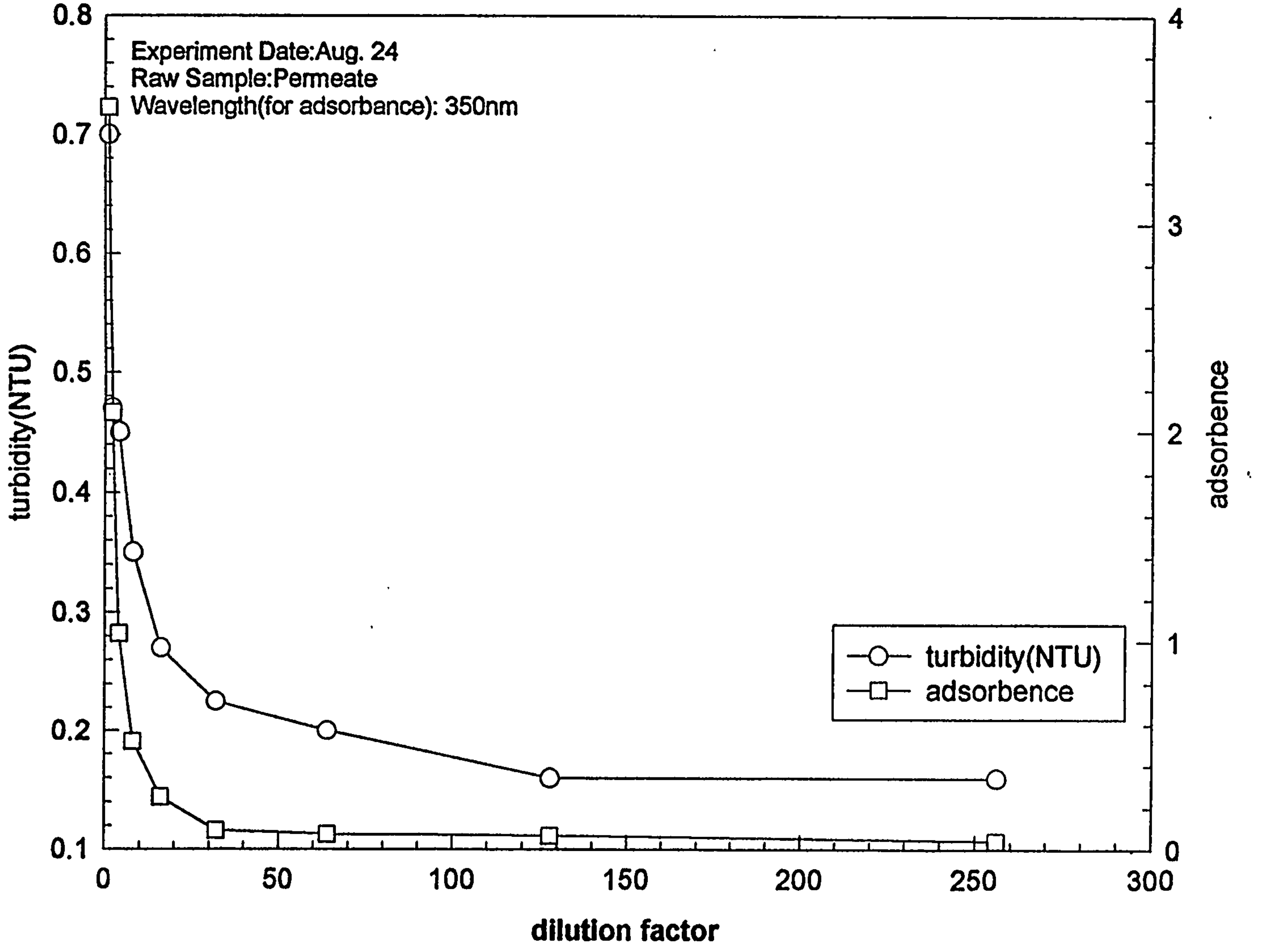

Figure 13. Permeate Turbidity and Adsorbance Versus Dilution Factor For Surfactant Run 1

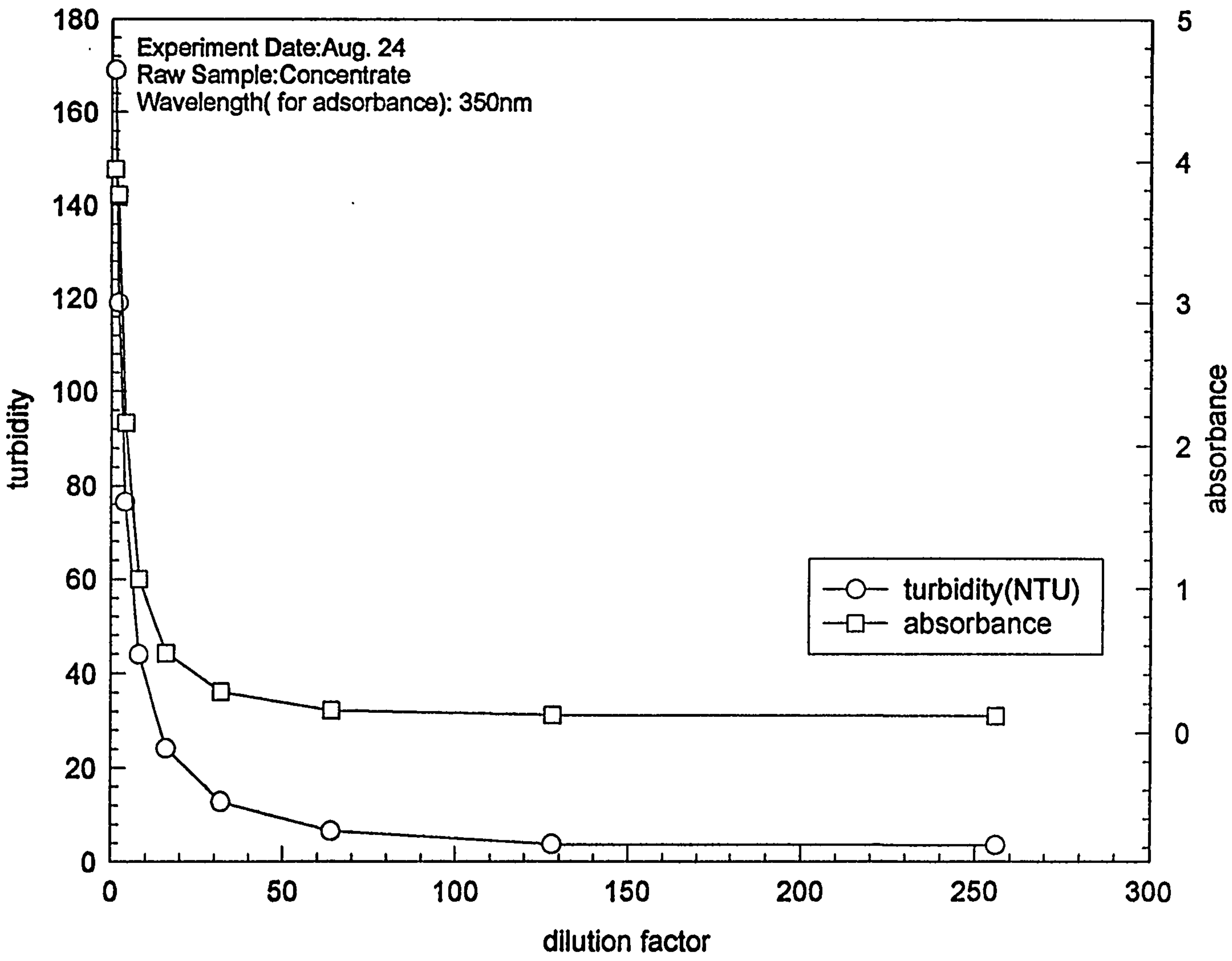

Figure 14. Concentrate Turbidity and Adsorbance Versus Dilution Factor For Surfactant Run 1.

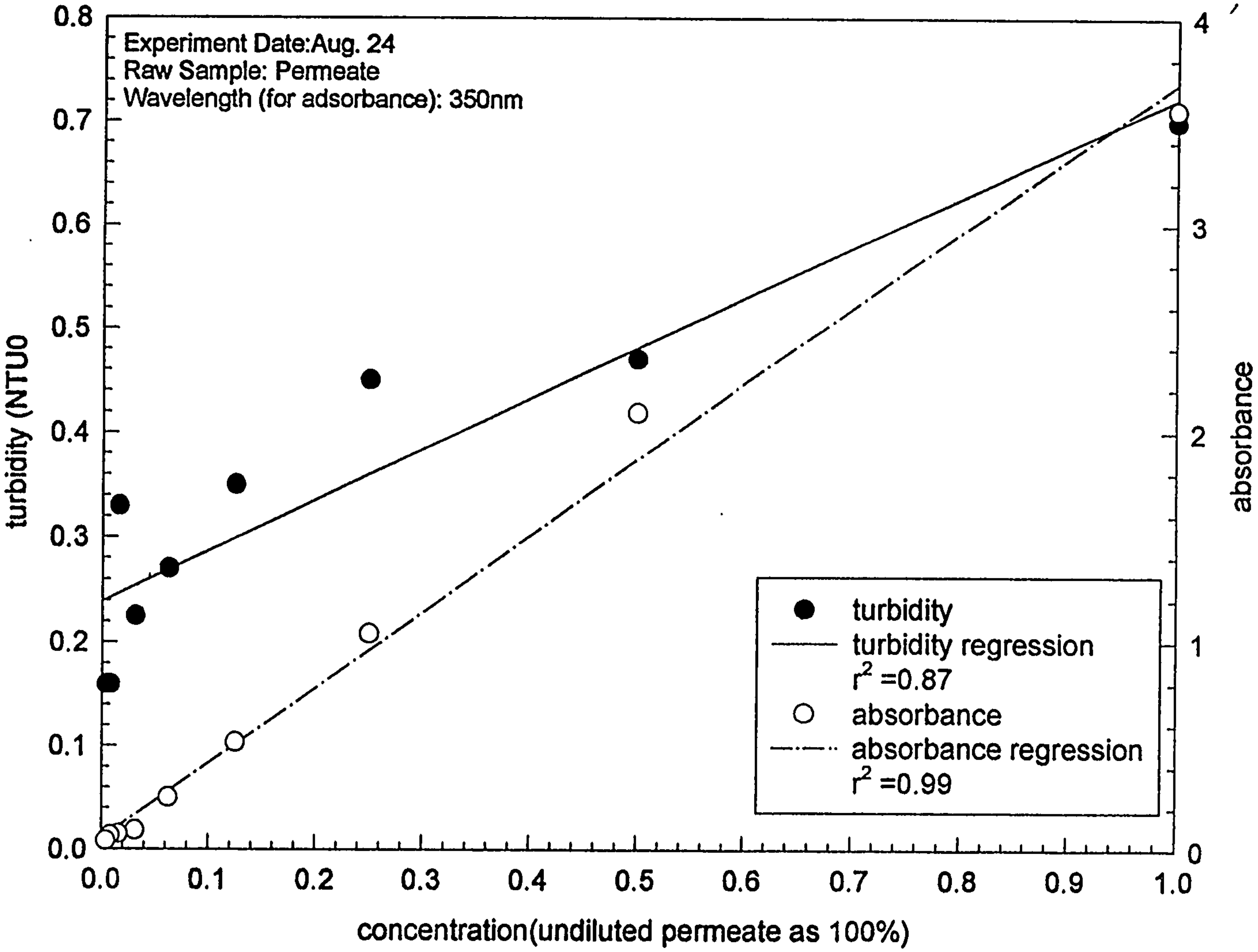

Figure 15. Linear Regression of Permeate Turbidity and Adsorbance Versus Amide Concentration For Surfactant Run 1.

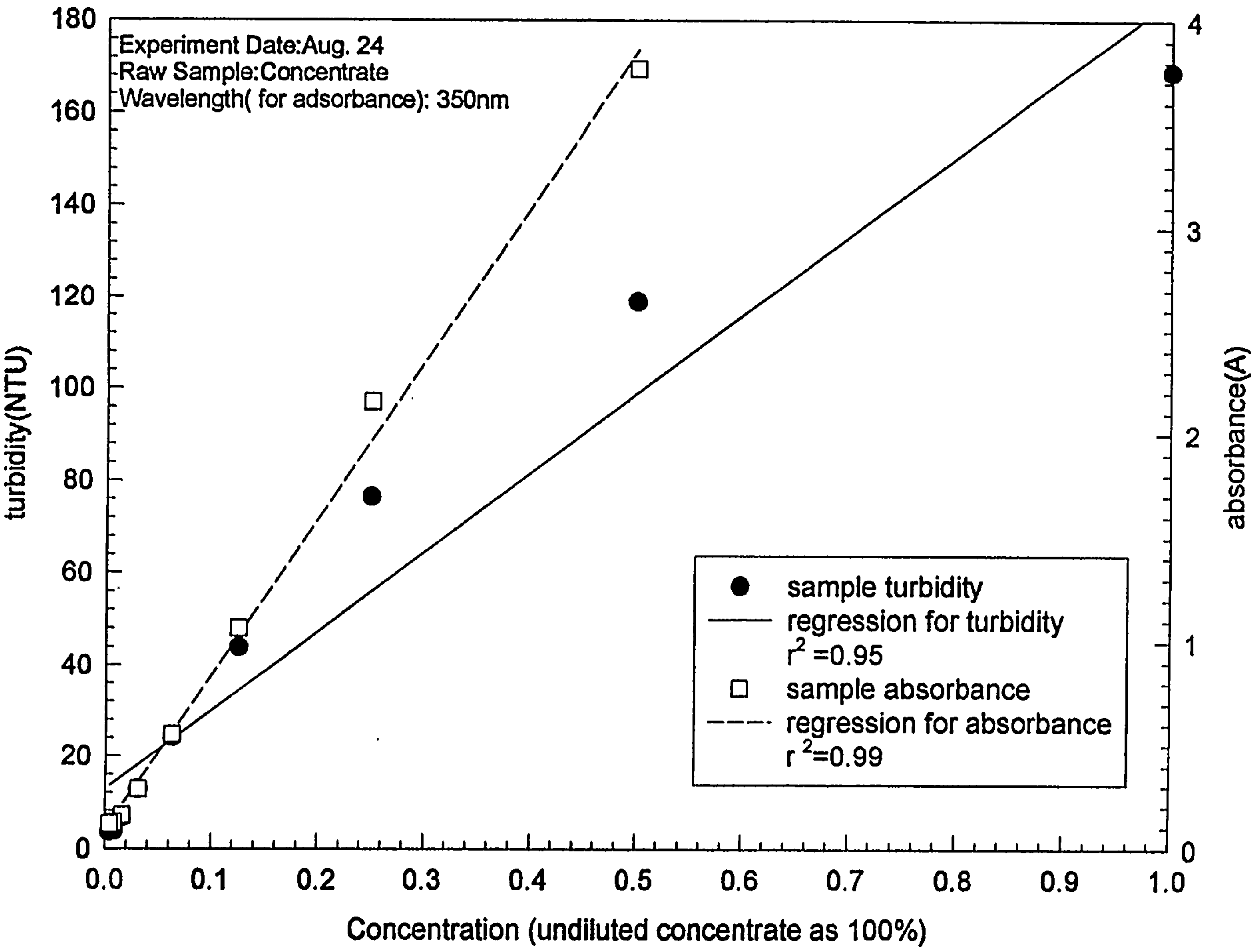

Figure 16. Linear Regression of Concentrate Turbidity and Adsorbance Versus Amide Concentration For Surfactant Run 1.

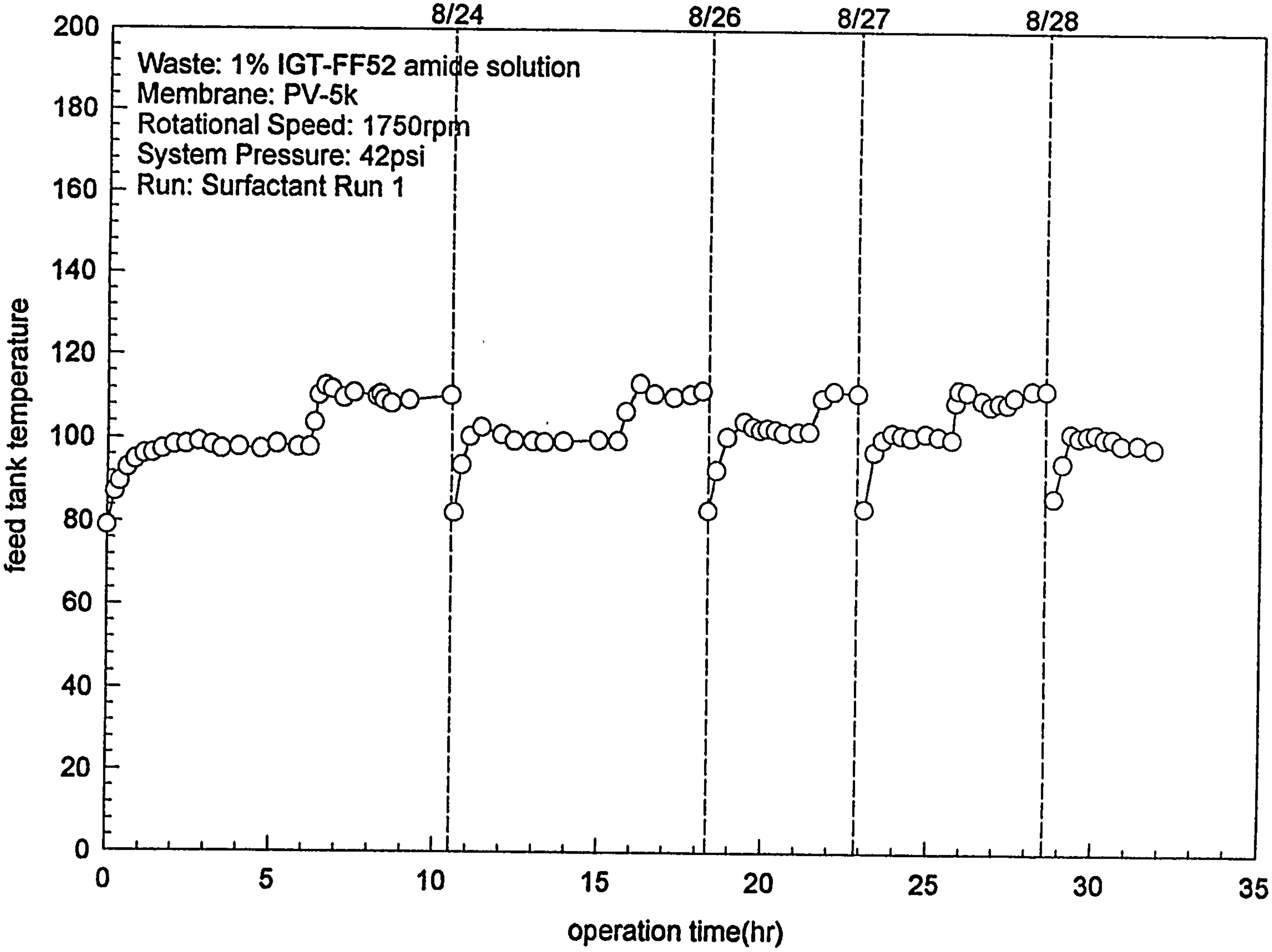

Figure 17. Feed Tank Temperature Versus Operational Time For Surfactant Run 1.

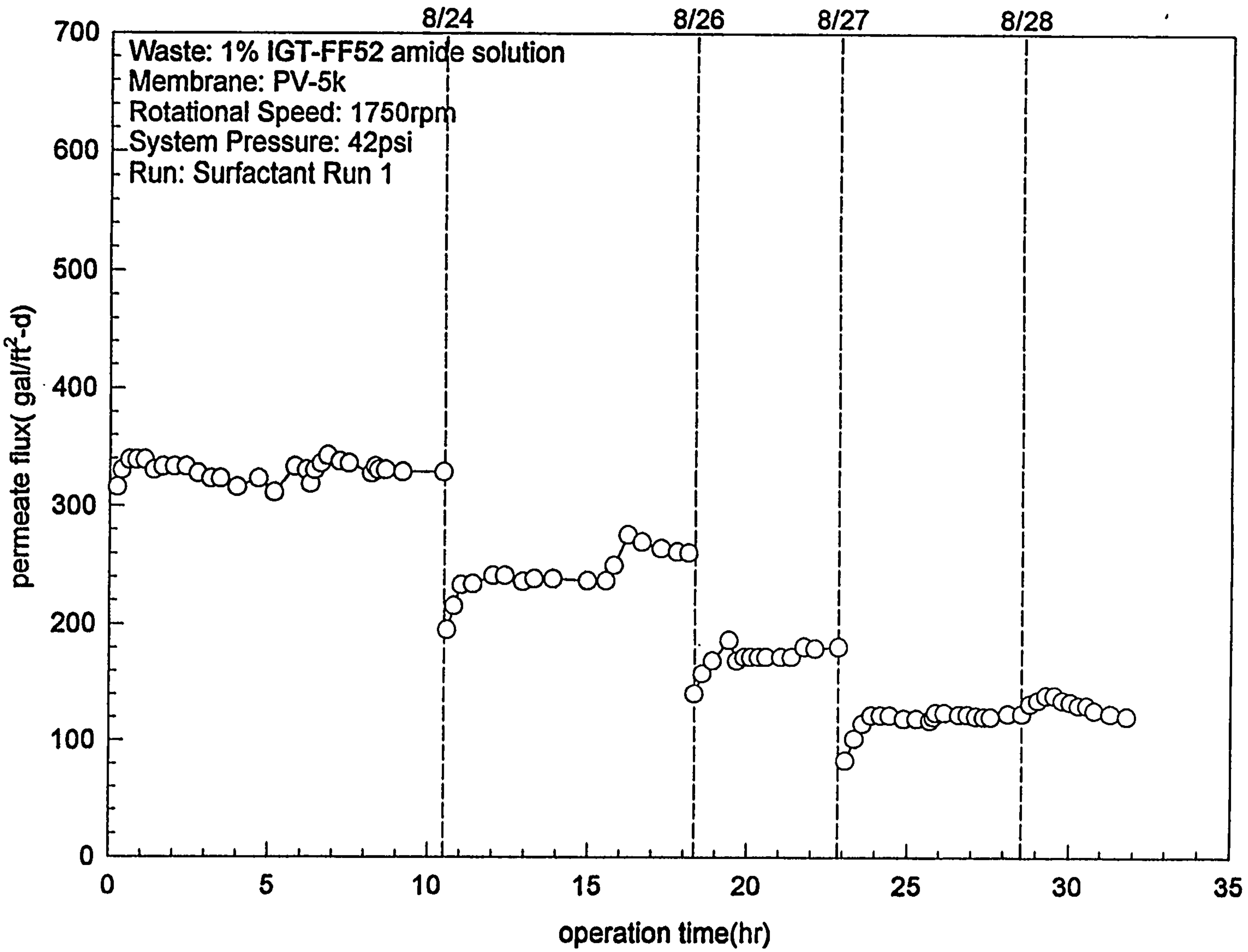

Figure 18. Permeate Flux Versus Operational Time For Surfactant Run 1.

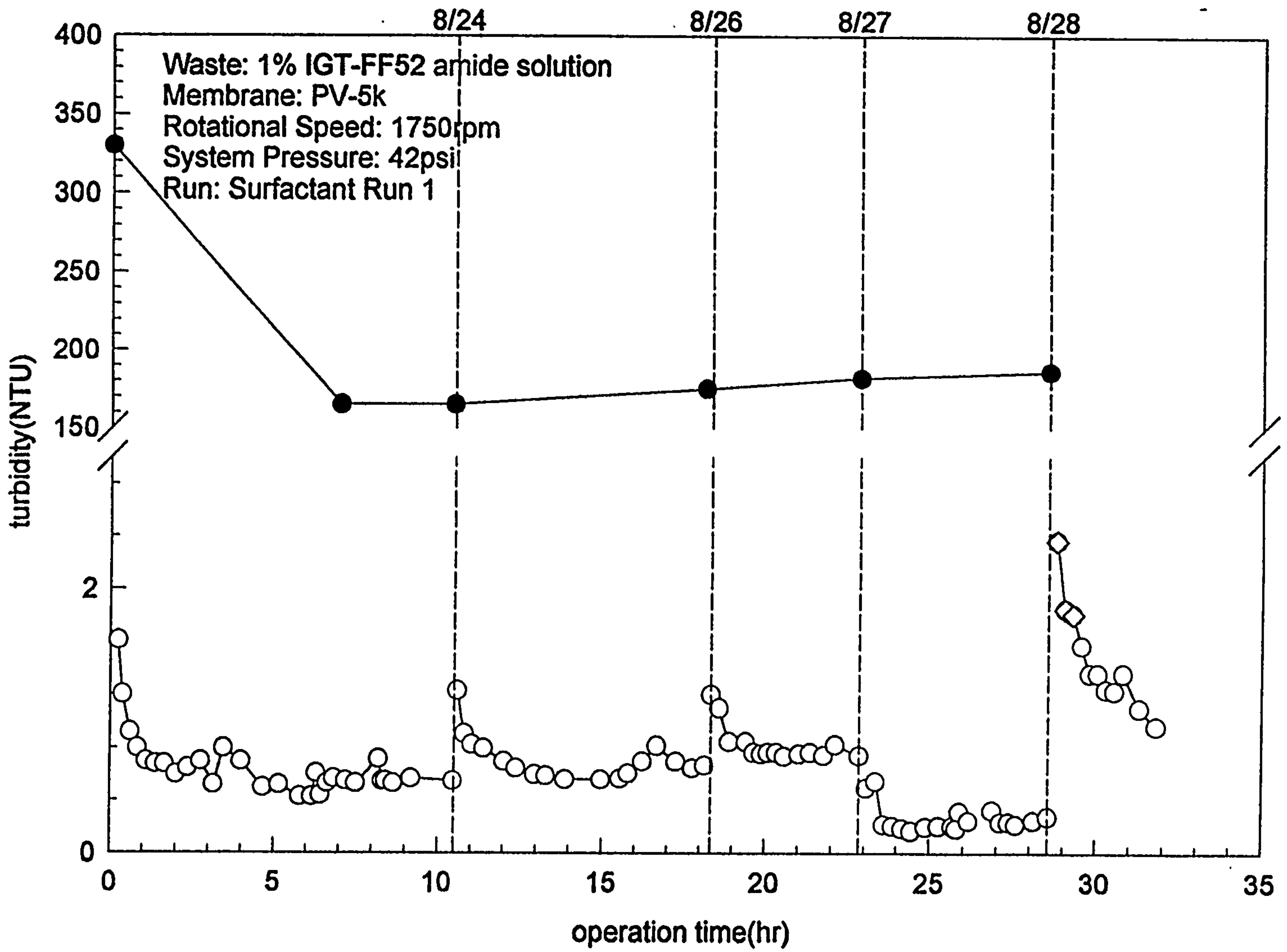

Figure 19. Permeate and Concentrate Turbidity Versus Operational Time For Surfactant Run 1.

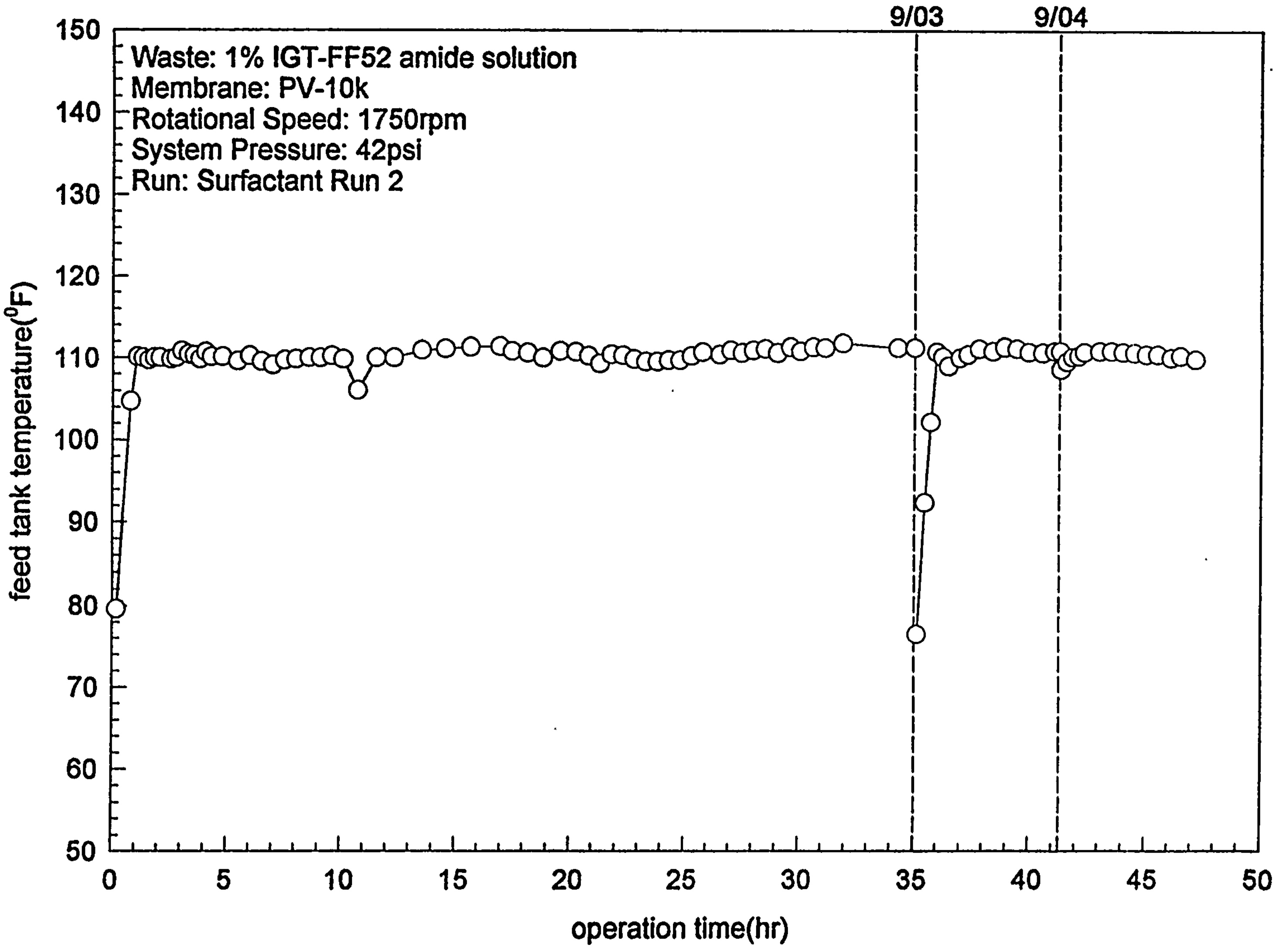

Figure 20. Feed Tank Temperature Versus Operational Time For Surfactant Run 2.

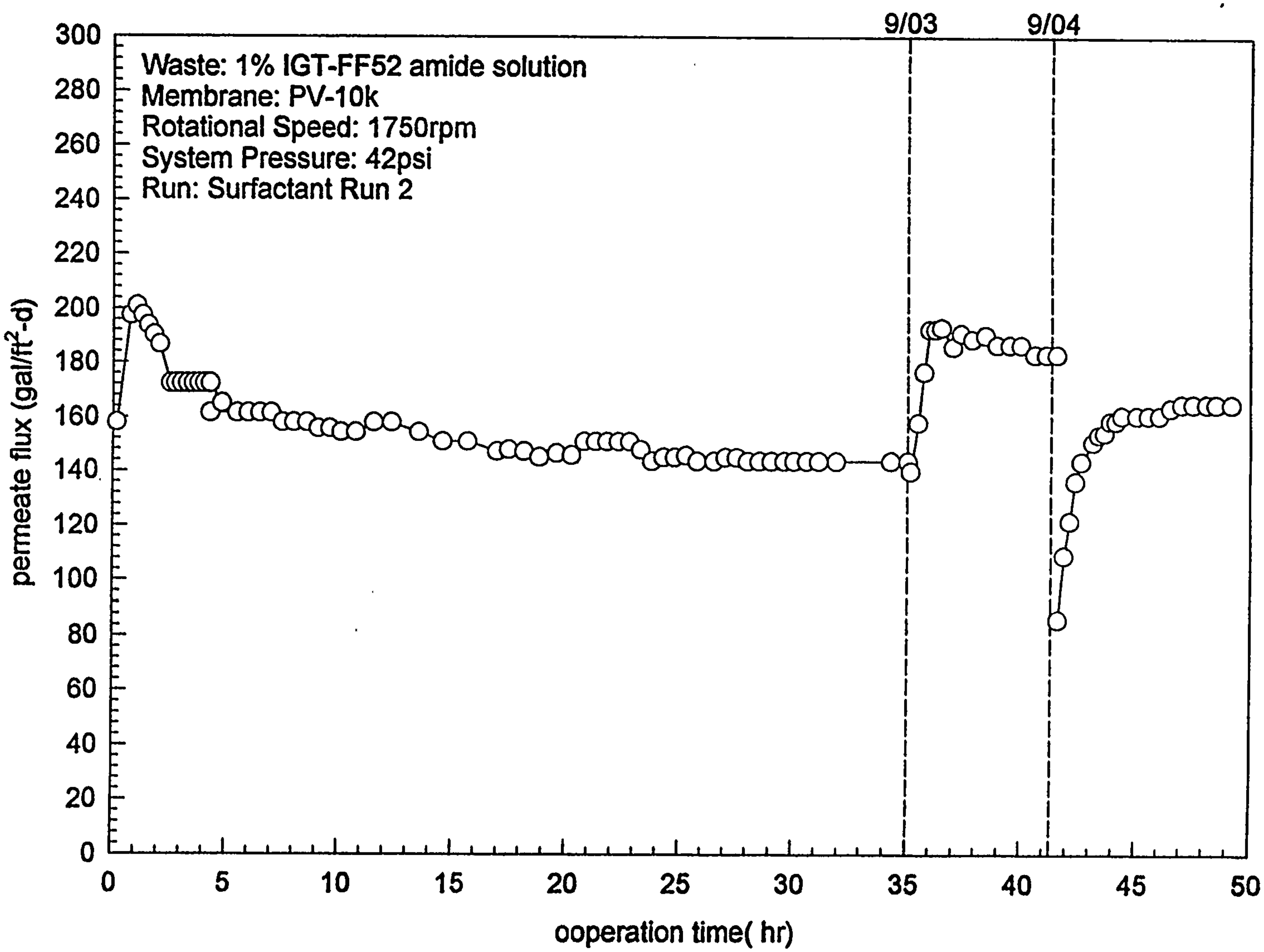

Figure 21. Permeate Flux Versus Operational Time For Surfactant Run 2.

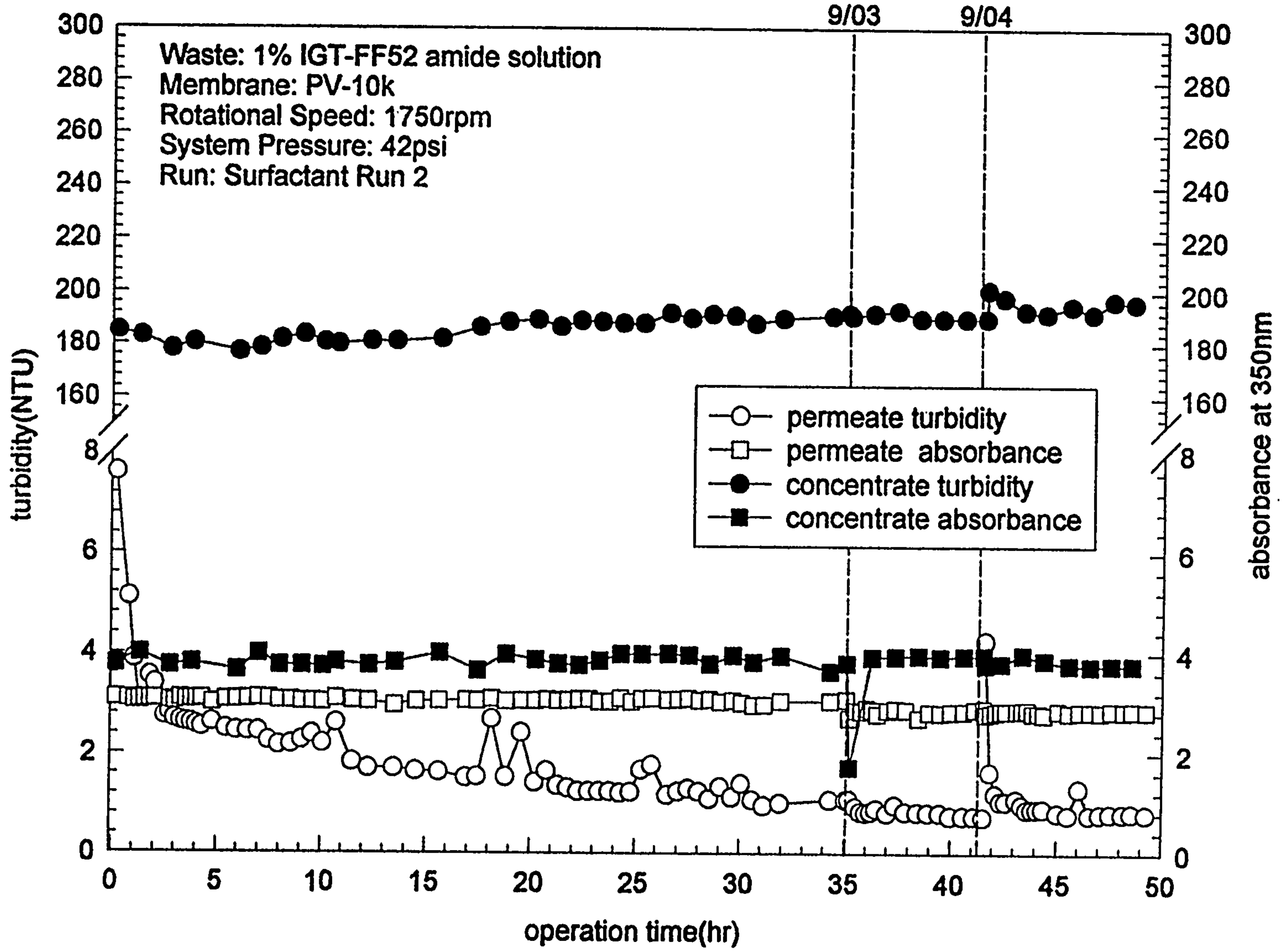

Figure 22. Permeate Turbidity and Adsorbance Versus Operational Time For Surfactant Run 2.

7.0 CONCLUSIONS

The SpinTek centrifugal ultrafiltration unit's experimental parameters (flux, temperature, and turbidity) were studied for treating a thickened activated sludge solution. The permeate flux and temperature remained steady. The permeate turbidity increased over time. The SpinTek unit retained the kaolinite, a simulated zeolite sorbent, in the concentrate stream. The increased kaolinite concentration was reflected by an increase in concentrate TSS. Additionally, a flux increase was observed with both time and kaolinite concentration. When the effects of ionic strength were examined, it was determined that the permeate flux decreased as the ionic strength of the concentrate solution was increased. The SpinTek system also successfully separated a biosorbent from the permeate stream using a 0.1 um ceramic membrane.

Turbidity was determined to be an appropriate indicator of micelle concentration in the IGT-FF52 amide solution. Adsorbance was found to be an indicator of the dissolved amide concentration in the amide solution. The PV-5k membrane was more effective than PV-10k in treating the IGT-FF52 amide (with higher turbidity removal and higher permeate flux). However, the 5k membrane was easily fouled and an effective cleaning method has not been developed.

8.0 WORK PLANNED FOR NEXT QUARTER

Several surfactants will be separated from the aqueous phase in the next quarter. The selection of the surfactants will be based on their use at the proposed soil flushing demonstration at DOE's RMI site in Astabula, Ohio.

9.0 TECHNICAL/ADMINISTRATIVE DIFFICULTIES

There were significant no technical/administration difficulties.