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Development Studies for a Novel Wet Oxidation Process:

Phase III Topical Report

Work Performed Under Contract No. DE-AC21-92MC29107

For

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Morgantown Energy Technology Center
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Abstract

DETOX^{SM} is a catalyzed wet oxidation process that destroys organic materials in an acidic water solution of iron at 373 to 473 K. The solution can be used repeatedly to destroy great amounts of organic materials. Since the process is conducted in a contained vessel, air emissions from the process can be well controlled. The solution is also capable of dissolving and concentrating many heavy and radioactive metals for eventual stabilization and disposal.

The Phase III effort for this project is fabrication, assembly, and installation of the DETOX^{SM} demonstration unit, preparation of documentation and training to meet site requirements for operation, followed by system run-in and shakedown testing of the unit prior to demonstration testing.

The Title III design was completed and the unit was fabricated according to standards set forth by OSHA, EPA, the American Petroleum Institute (i.e., chemical and petroleum industry standards), and the ASME B-31.3 Piping Code requirements as agreed to in preliminary design meetings with primary stakeholders. The unit was assembled in three modules and two trailers and then shipped to the TNX facility at the Savannah River Site in September and October of 1996. On-going site integration tasks were addressed while delays in installation arose due to funding sources and costs. In March of 1997, Delphi was authorized to proceed with the installation of the unit, making electrical and mechanical connections necessary to operate the unit. All installation tasks were completed in August of 1997.

Results of an Operational Readiness Review conducted in August 1997 verified that Delphi's procedures and documentation met the necessary requirements to operate the unit at SRS. Completion of the final checklist of WSRC requirements was then addressed including the Owner's Independent Inspection Report, verifying all components of the unit met B-31.3 standards. Final hydraulic and pneumatic tests were completed in November to satisfy the B-31.3 requirement. During the month of December, the control system and heating and cooling systems were then prepared to initiate system startup and conduct the run-in tests. Shakedown tests were combined with the Phase IV tasks due to DOE funding constraints, i.e., tests formerly planned to be conducted with oil and solid materials were deferred to Phase IV and combined with similar testing.

Once run-in testing has been completed, the primary objectives of Phase III will have been achieved and the project will be ready to proceed to demonstration testing (Phase IV).

Acknowledgments

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Background

Large volumes of hazardous, toxic, and/or radioactive wastes have been generated at Department of Energy (DOE) operational sites and in many areas of the private sector. These wastes may contain variable amounts of hazardous and/or toxic organic compounds and hazardous and/or toxic inorganic materials, such as heavy metals, and radioactive elements.

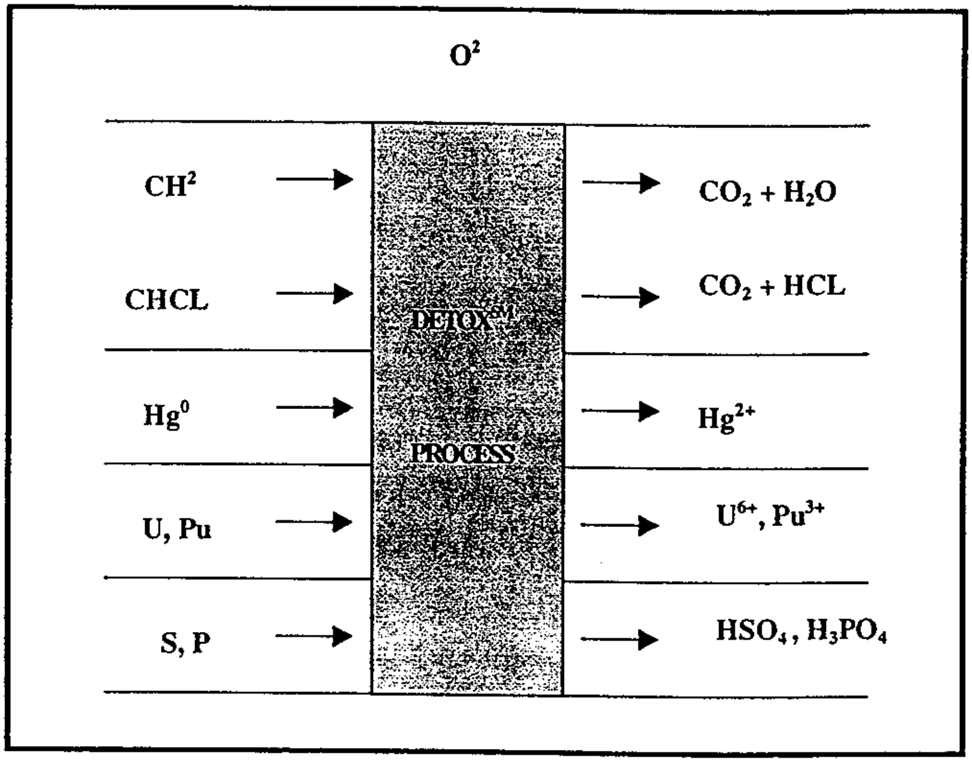
The problem of waste that is both hazardous and/or toxic, and radioactive (often designated "mixed" waste) at Department of Energy sites is particularly acute, since there are few, if any, non-incineration methods approved for its treatment and the Federal Facilities Compliance Act (FFCA) mandates the development of effective methods of dealing with mixed waste at government facilities.

The work reported herein has resulted from PRDA DE-RA21-92MC28245, Research and Development for Environmental Restoration and Waste Management Technologies, Sub-Research Area 3.05–Innovative Chemical and Biological Reactor Systems.

The technology proposed for development to meet the Department of Energy's needs was the DETOX^{SM} process, a patented chemical treatment that oxidizes organic compounds to simple products. Figure 1 shows reactants and products in the process. Treatment is performed in an acidic water solution using oxygen gas as the oxidant.

Due to the novel combination of catalysts used in the solution, the process can oxidize most organic compounds at moderate pressure and temperature. The acidic water solution is capable of dissolving great amounts of many toxic and radioactive metals. Since treatment is performed in a water solution, the process is not especially sensitive to the water content of the waste. The process will also accept a large range of particle sizes and waste types, and is not especially sensitive to variations in waste content.

Figure 1: The DETOX^{SM} Process



Although DETOX^{SM} had shown promising results in testing on some organic and infectious waste types, the process had never been applied to complex hazardous and mixed wastes, which may contain a variety of organic compounds and metals.

The Phase I effort of this project determined the practical range of application for the process in treatment of multi-component wastes and performed a conceptual engineering study for the process in application to Department of Energy wastes.

In the Phase II effort, a survey of potential demonstration sites and development of a Title II engineering design of the demonstration unit was completed.

Objectives

The objectives of the Phase III effort were to fabricate, assemble and install the demonstration unit, to prepare documentation and complete training programs necessary to meet site requirements, and to conduct system checks and functionality testing sufficient to begin formal testing.

Project Description

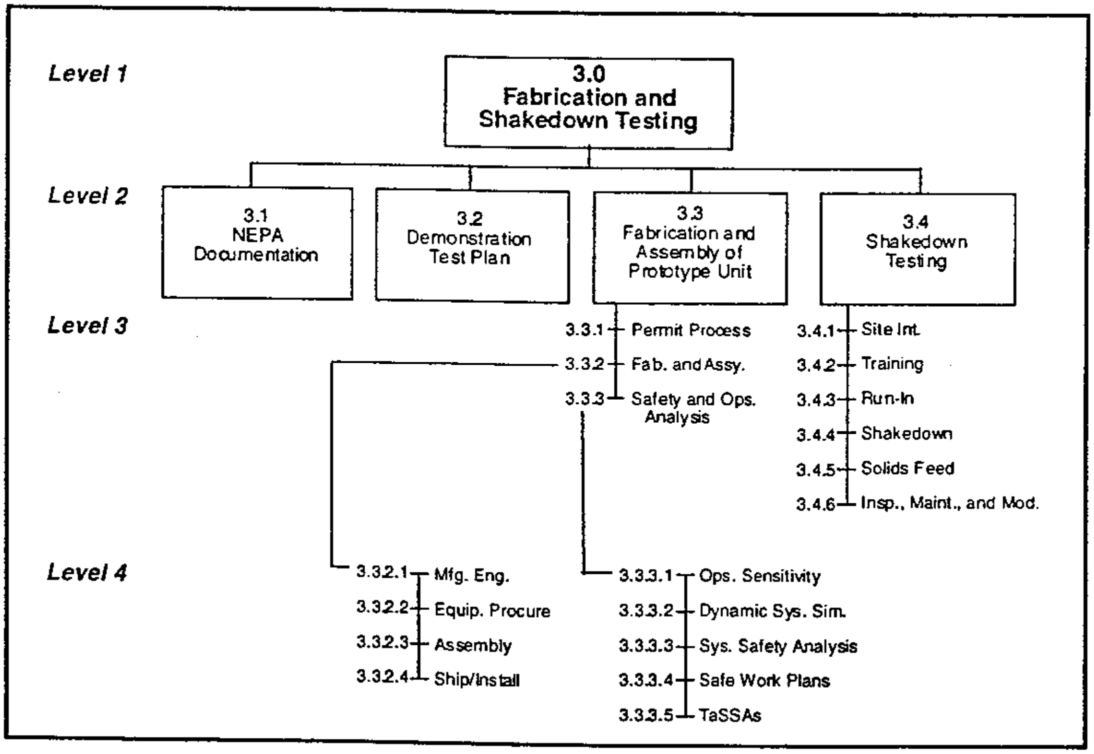
Development of the DETOX^{SM} process is planned to proceed in four phases. In Phase I, laboratory tests established the range of applications for the process and a conceptual design (Title I) was performed. In Phase II, a site selection survey has been conducted and the design of the demonstration unit completed through Title II level (sufficient for costing, but not including detailed shop drawings). In Phase III (initiated in March 1995), detailed design (Title III) and construction of the prototype has been performed. The unit has been delivered, installed and tested to ensure proper functionality, as specified. In Phase IV, the demonstration unit will be operated in the field to evaluate its performance on surrogate wastes.

Phase III Task/Test Plan

Phase III of the project has been organized into a four-task effort consisting of (1.) preparation of NEPA documentation for Phase IV, (2.) drafting of a Demonstration Test Plan, (3.) Fabrication and Assembly of prototype unit, and (4.) Shakedown Testing.

Each task is shown along with any of its subtasks in the Work Breakdown Structure, Figure2.

Figure 2 *Work Breakdown Structure*



Each of the tasks are delineated in Table 1 below as to their description, responsible party and the deliverable or milestone characterizing the completion of the task.

Table 1: Tasks Outline

Task No	Task Description	Responsible Party	Objective
3.3.	Fabrication and Assembly	Delphi	Unit delivered, installed, permits issue, safe operating plans and studies complete
3.3.1	Permit Process	Delphi	Authorization to operate issued by SCDHEC
3.3.2	Fabrication and Assembly	Delphi	Unit delivered
3.3.2.1	Manufacturing Engineering	Jacobs Applied Technology, Inc.	Title III Design Package
3.3.2.2	Equipment Procurement	Jacobs Applied Technology	Purchase of components and parts for total system
3.3.2.3	Assembly	Jacobs Applied Technology	Assembly of Modules 1,2,3, control trailer, and MCC
3.3.2.4	Ship and Install	Jacobs Applied Technology	Transport and location of unit in 673-T at SRS. Connection of electrical and mechanical systems.
3.3.3	Safety and Operations Analysis	Delphi	Operating limits, engineering model, safe work plans and procedures
3.3.3.1	Operations Sensitivity Analysis	Delphi	To determine operating limits and characterize secondary waste streams
3.3.3.2	Dynamic System Simulation	University of New Mexico, Dept of Chemical Engineering	Engineering Model
3.3.3.3	System Safety Analysis	Sandia National Laboratories	Safe Operating Limits
3.3.3.4	Safe Work Plans	Delphi Research, Inc.	Site specific safety plans and procedures
3.3.3.5	TASSA's	Delphi Research, Inc.	Site specific safety plans and procedures
3.4	Shakedown Testing	Delphi Research, Inc.	Defensible demonstration test data
3.4.1	Site Integration	Delphi Research, Inc.	Readiness Review and authorization to operate by SRS

Table 1: Tasks Outline Cont'd

Task No	Task Description	Responsible Party	Objective
3.4.2	Training	Delphi Research, Inc.	Certified Operations Staff
3.4.3	Run-in	Delphi Research, Inc.	Functionality Testing
3.4.4	Shakedown Testing	Delphi Research, Inc.	Verification of feed systems and operational conditions
3.4.5	Solids Feed Tests	Delphi Research, Inc.	Solid Feed System verification
3.4.6	Inspection, maintenance, and modification	Delphi Research, Inc.	Verification that unit is operationally sound for Phase IV testing.

Results

Task 1 – Information required for the National Environmental Policy Act

Work performed under this task resulted in the issuance of a formal Notice of NEPA Approval for Phase IV. The final NEPA documentation for the DETOX^{SM} demonstration at SRS consisted of two parts: An Environmental Evaluation Checklist (EEC) and a Specific and Supplemental Information package. The EEC is a detailed checklist that seeks to ascertain the degree of potential impact to the environment posed by the proposed project effort. The Specific and Supplemental Information package provides detailed and specific information needed to fully support the answers given on the EEC. Both of these documents were then evaluated to determine whether further documentation and evaluation of environmental impact (i.e., Environmental Assessment or Environmental Impact Statement) were warranted. The EEC and the Specific and Supplemental Information package were reviewed by Westinghouse Savannah River Company (WSRC) and the DOE-Savannah River Operations Office. Their conclusion was that the DETOX^{SM} demonstration project at SRS was covered as a Research, Development, and Demonstration under a site-wide Categorical Exclusion (CX), as set forth in the DOE NEPA implementation regulations. Therefore, no further NEPA documentation would be required to proceed into Phase IV of the DETOX^{SM} demonstration.

Task 2 - Drafting of the Demonstration Test Plan

Work performed under this task consisted of drafting a single document that described the technology, demonstration objectives, regulatory compliance, health and safety issues, and a management plan for the demonstration. The Demonstration Test Plan was established to be fundamental to the project, since primary stakeholders (host sites, the contractor and its fabricator, DOE Mixed Waste Focus Area (MWFA) and DOE-FETC) are required to approve and sign the document before demonstration testing can proceed. The Demonstration Test Plan is now in its final form.

Task 3 - Fabrication and Assembly of the Demonstration Unit

Design revisions were made to the Title II unit design generated in Phase II to accommodate the HAZOP requirements and recommendations, to meet site specific requirements, and to further enhance system performance and reduce costs. Major components were fabricated and the unit assembled at Jacobs Applied Technology, Inc.'s fabrication facility in Orangeburg, SC.

The unit was transported and delivered to the TNX facility for placement and installation. Other activities included in this task were safe operating studies, dynamic system simulation studies, and development of safe operating plans and procedures. In addition, studies were undertaken to address metals solubility, physical characteristics of the DETOX^{SM} solution, filtration characteristics, and stabilization of the solid residue containing heavy metals. Information obtained from these studies was used to specify materials of construction and to develop chemical engineering models and specifications for design of the demonstration unit.

Task 4 - Shakedown Testing

One of the chief subtasks in this effort was to perform site integration functions. Identification and compliance with site requirements regarding training, safety, operating requirements, and management issues related to site labor agreements were all addressed under this task.

Installation and hook-up of the unit is recognized as one the most significant factors which was not fully addressed in planning. Approximately six months of delay was experienced due to resolution and funding of this issue. However, once agreed upon, the installation was performed by Delphi and proceeded fairly smoothly.

A milestone in the shakedown testing phase was the Operational Readiness Review, which was conducted by WSRC. Review of documentation and field interviews to verify that all operational personnel were fully trained and prepared to safely operate the system were performed by WSRC. Delphi was given authorization to proceed with the startup of the unit, when it had been verified that all site requirements for operation of the unit had been met.

System checks and start-up of the unit are in progress. Run-in tests are scheduled to conclude in January of 1998. As a result of the meeting on December 3, 1997, subtasks 3.4.4 and 3.4.5 to perform shakedown testing using oil and solid combustible materials, respectively, have been consolidated with tests of surrogate materials to be conducted in Phase IV. Therefore, Phase III shakedown testing with organics has been combined with Phase IV performance testing.

Completion of this task will conclude the Phase III effort.

The schedule showing the planned and actual completion of tasks is shown in Appendix I.

Section 1

Introduction

1.1 Problem Statement

Complex multi-component hazardous and mixed wastes and remediates are a common problem at Department of Energy (DOE) operational sites and in many areas of the private sector. These materials can contain variable amounts of hazardous and/or toxic organic compounds and hazardous and/or toxic inorganic materials such as heavy metals, and radioactive metals. The hazardous, toxic, and/or radioactive materials may be dispersed in other organic materials, sludges, soils, or water solutions.

The problem of mixed waste at Department of Energy sites is particularly acute, since there are few if any methods for its treatment and the Federal Facilities Compliance Act (FFCA) mandates the development of effective methods of dealing with mixed waste at government facilities.

What is needed is a robust, fairly omnivorous process for destroying a wide variety of organic waste components, while concentrating the inorganics and radioactive metals in a matrix for ultimate recovery and/or disposal. The process should use conventional technology to minimize development and application time, and it should minimize air and water emissions, operate safely, and be cost effective.

1.2 Objective

The objective of this Project is to develop a catalytic wet oxidation process (DETOX^{SM}), which utilizes a concentrated, acidic solution of iron chloride and cocatalysts, for the treatment of multi-component wastes and remediates. Successful development will provide a versatile treatment method, which can destroy hazardous organics in a variety of matrices while simultaneously concentrating many metals in the process solution for recovery and/or disposal.

The overall objective for Phase III is to assemble and prepare the demonstration unit for full scale testing and to certify personnel for safe operation of the unit during testing. Necessary steps to achieve this objective involve the design, fabrication and assembly of the demonstration unit, its transportation to, placement, and installation at SRS, and the performance of functionality and run-in tests necessary to verify that the unit is capable of performing testing on surrogate materials in Phase IV. Development of procedures, plans, and training methodologies which qualify Delphi personnel to operate and maintain the system through the demonstration period are secondary objectives that must be met in order to meet the overall objectives of Phases III and IV.

While the process may not provide stand-alone treatment of all applicable waste types, it holds promise of being able to provide significant volume reduction of mixed wastes and remediates, and conversion of mixed waste types to radioactive-only wastes. Some of the process residues from future hazardous and mixed waste treatment operations would require additional treatment, such as immobilization, before ultimate disposal. Other residues, such as excess water and neutralized condensate brine from the process, should be non-hazardous, but under current law would require delisting before release. In the demonstration testing at SRS, effluent condensate from the unit will be disposed of via the TNX wastewater treatment facility. WSRC will dispose of all plant trash and non-hazardous materials. WSRC will also dispose of all RCRA hazardous wastes and will be reimbursed for all reasonable costs by Delphi as a direct cost under the FETC contract.

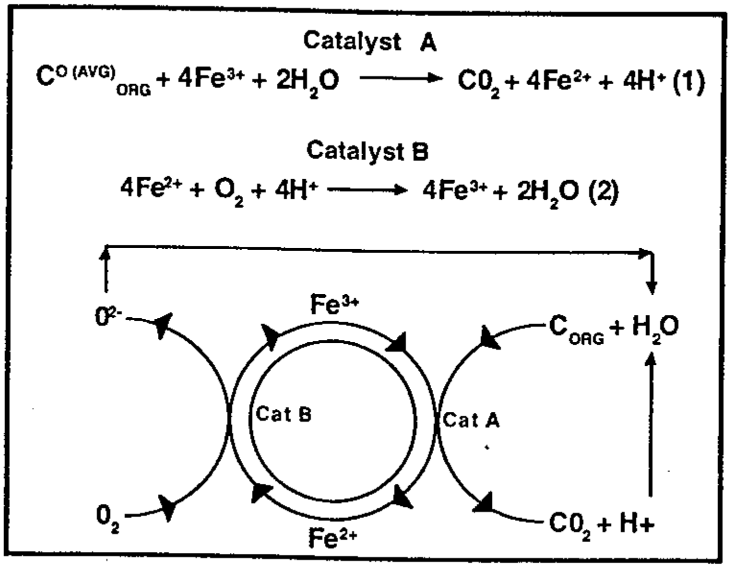
1.3 Process Description

A patented process, DETOX^{SM} is a combination of iron ions and homogeneous oxidation cocatalysts (typically platinum and/or ruthenium ions) in an acid solution. Ferric iron in aqueous hydrochloric acidic solution oxidizes a wide range of organics to simple products (i.e., CO_2, H_2O, HCl) at temperatures of 373 K to 523 K. Figure 3 illustrates the process chemistry.

The typical DETOX^{SM} solution composition is about 60% by weight ferric chloride ($FeCl_3$) and 40% by weight HCl in water, with small amounts of dissolved platinum and ruthenium cocatalysts. Ferric iron is responsible for the oxidation of the organic compounds, and is reduced to ferrous iron in the reaction. The ferrous iron is returned to the ferric state by reaction with oxygen. Thus, the iron ions act ultimately as a catalyst, being cycled between the ferric and ferrous states.

Extensive previous testing performed by Delphi Research, Inc. both privately and under contract to the U.S. Air Force, the New Mexico Research and Development Institute, EG&G Rocky Flats/Kaiser-Hill, LLC, and Los Alamos National Laboratory has established the ability of ferric iron to oxidize organic compounds, the effects of temperature on the reaction rate, and the advantages of using the cocatalysts.

Figure 3: *The DETOX^{SM} Chemistry*



Catalyst A

$$C^{0\,(AVG)}_{ORG} + 4Fe^{3+} + 2H_2O \longrightarrow CO_2 + 4Fe^{2+} + 4H^+ \quad (1)$$

Catalyst B

$$4Fe^{2+} + O_2 + 4H^+ \longrightarrow 4Fe^{3+} + 2H_2O \quad (2)$$

Section 2

Information required for the National Environmental Policy Act

Task 3.1

2.1 Objective

Environmental, health, and safety information for the demonstration must be documented and approved before demonstration testing can proceed. The objective of this task is to assimilate information regarding all applicable Federal, state, and local regulations and requirements affecting the planned demonstration of the DETOX^{SM} process, and to document how each category of applicable regulations and requirements have been addressed to ensure regulatory compliance.

2.2 Procedure

The procedure used to accomplish this task has been a three step process: (1.) A series of meetings and ongoing communication with WSRC NEPA coordinator and the DOE-FETC Environmental Project Manager to acquire the necessary information that must be addressed in the NEPA documentation, (2.) Review of all applicable standards and regulations, (3.) Preparation of tables, calculations, and supporting documentation to determine process characteristics, (4.) Preparation and submittal of an Environmental Evaluation Checklist, (5.) Preparation of draft NEPA documentation for review and comment, and (6.) Preparation of final NEPA documentation for review and approval. Subsequent changes in environmental requirements at the SRS facility have resulted in on-going modifications to the approved NEPA, which will likely continue into Phase IV activities. As of the end of Phase III, Revision 2 of the NEPA documentation is approved to begin Phase IV testing; however, modifications reflecting minor changes in composition and amounts of waste types to be tested are in preparation and anticipated to be approved in sufficient time to maintain the project schedule.

2.3 Results

Initially Delphi acquired copies of all federal, state, and local standards and regulations that were known to be applicable to the demonstration effort. As changes and/or additions in applicable regulations were made, the list of inventoried documentation was updated. A review of all standards and regulations was conducted and applicable sections of these standards and requirements were noted. Preliminary work on NEPA documentation provided some broad information regarding requirements at Savannah River Site and Weldon Spring Site Remedial Action Project (WSSRAP).

SRS

At the beginning of Phase III, March 1995, representatives of Delphi met with WSRC staff to further delineate site requirements for NEPA approval. Delphi was given an Environmental Evaluation Checklist to complete and submit. A draft of the EEC was submitted to SRS in June 1995. In early October 1995, a final version of the NEPA documentation was submitted to SRS for approval, and a formal Notice of NEPA Approval (NONA) was issued by SRS later that month. Concurrence by the FETC Environmental Project Manager was issued in December 1995.

WSSRAP

Concurrent with the preparation of the NEPA documentation for SRS, was the effort to verify that all environmental, health, and safety issues were being addressed at the selected host site for low-level mixed waste (LLMW) applications. In September of 1995, confirmation was received from WSSRAP that the proposed DETOX^{SM} project was covered under the CERCLA Record of Decision and Remedial Investigation/Feasibility Study documentation for WSSRAP. In October a determination was made by DOE-FETC that NEPA documentation for the transportation of the demonstration unit from SRS to WSSRAP would not be required. In January of 1996, WSSRAP issued notice to Delphi and DOE-FETC that they were withdrawing as a host site for the DETOX^{SM} demonstration, since they had received authorization from the state of Tennessee to ship DETOX^{SM} candidate LLMW's to the Oak Ridge Reservation for incineration.

RFETS

With the withdrawal of WSSRAP, the host site for LLMW applications in Phase IV was reevaluated and the secondary site chosen during the site selection process in Phase II, Rocky Flats Environmental Technology Site or RFETS, was approved in late April 1996 as the replacement to WSSRAP. Since no prior history of NEPA evaluation had been performed at RFETS, efforts were initiated to obtain information necessary to prepare an Environmental Evaluation Checklist and NEPA documentation. Efforts to prepare documentation continued until June of 1997 when RFETS issued its withdrawal to support the demonstration due to lack of funding and scheduling of waste treatment.

The resulting NEPA documentation approvals are shown in Appendix A to this document.

Section 3

Drafting of the Demonstration Test Plan

Task 3.2

3.1 Objective

A Demonstration Test Plan (DTP) was identified as a requirement to be added to the project work plan in Phase II of the effort. The development of the plan has continued throughout Phase III. The plan is designed to provide an overview of the project, including identification of stakeholders, experimental test program, site integration, environmental compliance and monitoring, and project schedules. The primary objective of the DTP is to provide defensible data for future application of the DETOX^{SM} process. The DTP will also provide each participant a comprehensive report of the demonstration project and methodologies to be used meeting all technical, regulatory, and commercial objectives of the project. Approval by primary stakeholders (DOE-SR, WSRC, DOE-FETC, Delphi Research, Inc., and Jacobs Applied Technology, Inc.) is required before the demonstration is allowed to proceed to Phase IV.

3.2 Procedure

The Demonstration Test Plan reflects the contributions and concerns of all primary stakeholders and the suggestions and recommendations of regulatory agencies, potential customers, and other interested parties. This document was established to be the responsibility of Delphi in terms of its preparation, but due to the required approval process invoked by primary stakeholders including the sites, MWFA, and DOE-FETC, the document is the collective input of all parties. The method used to revise and obtain approval of this document consisted of a three step process: (1.) Whenever modifications or improvements occurred in the project that were known to affect the technical objectives, a revision effort was initiated to modify the document to maintain its accuracy and reflect the current plan for demonstration. Delphi initiated the drafting of all changes of this type and forwarded a draft copy of the new revision of the plan to each of the primary stakeholders for review and comment. (2.) Comments received by the stakeholders were then addressed and incorporated in a revised version of the DTP. (3.) A final draft was submitted to stakeholders for approval and distribution.

3.3 Results

Revision 4 of the Demonstration Test Plan is the latest approved version of the plan. Modifications to the plan have been accomplished to accommodate the change in sites for LLMW applications and in recommended additions to the waste type to be tested in Phase IV. Revision 4 of the DTP provides the basis for initiating testing in Phase IV and in that respect, the objective of this task has been achieved.

Note: From this point is a description of work yet to be completed on this task as of the date of this report.

In December of 1997 a meeting held with representatives of the Mixed Waste Focus Area, DOE-FETC, DOE-SR, Delphi, and WSRC, where it was concluded that the test program needed to be reduced and/or consolidated to the extent that the most meaningful information would be obtained from the test program for commercial applications. However, the DTP will not have to be revised.

Section 4

Fabrication and Assembly of Prototype Unit

Task 3.3

4.1 Objective

This task defines the steps necessary to fabricate and assemble the DETOX^{SM} unit, to ensure that the unit is operated in a controlled and safe manner, and to obtain regulatory authorization to operate the unit for the planned period of demonstration.

4.2 Procedure

This task was organized into three major subtasks:

Task 3.3.1 Permit Process

Task 3.3.2 Fabrication and Assembly,

Task 3.3.3 Safety and Operations Analysis.

Task 3.3.2, Fabrication and Assembly and Task 3.3.3 Safety and Operations Analysis were further subdivided into sub-sub tasks. The tasks, subtasks, sub-subtasks, party performing the work, and the item signifying completion of the task are shown in Table 1 of this report.

4.3 Results

Subtask 3.3.1. Permit Process

SRS

Contact with regulatory authorities was initiated through the WSRC Environmental Protection Division, direct contact with South Carolina Department of Health and Environmental Control (SCDHEC), and with EPA, regionally and nationally. Meetings with WSRC staff resulted in a strategy to obtain authorization to operate under a wastewater treatment unit exclusion, as opposed to a RCRA Research, Development, & Demonstration (RD&D) permit. The facility at TNX was determined to be a non-RCRA facility due to its proximity to the Savannah River and subsequent releases of effluent water into the effluent water treatment system would require delisting, a prohibitively expensive process. A request to operate the DETOX^{SM} demonstration unit as a wastewater treatment unit was submitted to SCDHEC in January 1996. Since completion of fabrication and installation of the unit at TNX was still a few months away, Delphi requested at that time that SCDHEC not begin reviewing the application until June 1996. Authorization to operate was granted in June 1996. The approval period is 18 months from the date of "start-up". Similarly, an Air Emissions Exemption was granted in November 1995. Guidance documents were then prepared to ensure that all effluents from the process remained in compliance with the conditions of these approvals. It should be noted that even though the regulatory approvals are not technically full permits, they are considered by SCDHEC to be permits, and are enforced as such.

Modifications to the wastewater approval and air emissions exemption were made to account for changes in the waste types, Title V air quality regulations for SRS, and characteristics of the unit installation and operation. All modifications were approved and are in place as required to begin Phase IV testing. Due to DOE funding constraints, reduction in the scope of work for demonstration was required. These changes make it necessary to modify these approvals again. and the application and approval process may extend into Phase IV.

In addition to the authorization to operate, Delphi prepared a Best Management Practices and Spill Prevention, Control, and Countermeasures (BMP-SPCC) Plan to meet the site requirement for the handling and storage of liquid hazardous materials (e.g., oils, solvents) during operation. A waste profile form for DETOX^{SM} solution residue disposal was developed by Delphi and submitted to WSRC to facilitate documentation required for residue disposal at the culmination of testing in Phase IV.

Weldon Spring Site Remedial Action Project

Permitting activity at WSSRAP was relegated to preparation of the information necessary to meet CERCLA requirements using a RCRA Part B application format. This was mandated by the Missouri Department of Natural Resources. Work on the permit was suspended in January 1996 when WSSRAP withdrew from the demonstration.

Rocky Flats Environmental Technology Site

In June of 1996, it was determined that RFETS would require the DETOX^{SM} demonstration to operate under a RCRA RD&D permit. Activity was initiated to transfer the work performed at WSSRAP to the RFETS applications. A screening-level air modeling study was performed using an EPA-approved computer code for single stationary sources on simple terrain. Results of this testing were used to estimate maximum lifetime cancer risk and acute and chronic non-cancer health risks. Results showed those lifetime cancer risks to have a probability of occurrence of 1 in 100,000,000 and the non-cancer health adverse health effects to be rated at 1.0. Delphi made contact with the Colorado Department of Public Health and Environment (CDPHE) to verify emission limits and regulatory requirements to ensure that all aspects of applicable law were being addressed in the RD&D permit and air emissions exemption applications. Permit application efforts were suspended in June of 1997 when RFETS withdrew its support from the demonstration due to lack of funds and incompatible scheduling of the treatment of LLMW's.

Subtask 3.3.2 Fabrication and Assembly

Sub Task 3.3.2.1 Manufacturing Engineering

This task began with a review and update of the information gleaned from Phase II engineering design work and advanced studies regarding overhead condenser calculations. Initial work was begun to modify the system components to ensure that all HAZOP recommendations were adopted and/or addressed.

Additional work was performed to further reduce secondary emissions from stack gases in the form of incomplete products of oxidation. Based on studies conducted on thin film apparatus for RFETS, a second stage reactor was added to the design to ensure that emission levels were maintained well below lower limits of hydrocarbon emissions established by the states of South Carolina and Missouri.

Laboratory tests were conducted to determine filtration efficiencies and appropriate operating specification for the helical filter system to be installed on the unit. Simulations on the overhead condenser system were performed to verify that the reflux and overhead condensers had adequate capacity to remove the heat emitted from the unit.

A review of the Piping and Instrument Drawings (P&ID's) and HAZOP of the effected changes was conducted prior to initiating detailed drawings and procurement activities. Representatives from WSSRAP, WSRC, Jacobs Applied Technology, and Delphi participated in the initial HAZOP and approved the resulting HAZOP document. Procurement of the long-lead items was initiated using subcontract mechanisms pre-approved by DOE-FETC and in accordance with contract provisions.

Work continued to define layouts and material specifications for key components. Quotations for major components were received in December of 1995 and indicated a substantial escalation in price of the unit. An effort was immediately undertaken to mitigate the cost escalations by reviewing and removing system features that were not essential to process control or safety. Resulting changes were implemented and resulted in savings of approximately $800,000. System layout and the resulting isometric drawings were prepared. The task was completed in June of 1996 with the receipt of the Title III Design Package. Updates to P&ID's and ISO's continued to take place throughout installation of the unit to reflect the unit design "as installed".

Subtask 3.3.2.2 Equipment Procurement

Subsequent to the completion of the initial Title III design and HAZOP, quotations for equipment were obtained to verify engineering estimates. After review and modification to reduce costs, a revised design was approved and procurement activity was initiated to secure the long-lead items; e.g. reactor and filter assembly. Contact was made with suitable vendors and Delphi participated in the negotiation process to ensure that all applicable procurement regulations were met. Subcontracts with major suppliers were prepared and submitted to DOE-FETC for approval prior to the placement of the order.

Procurement of secondary items then proceeded to take place as specifications and JAT defined materials of construction in consultation with Delphi's Principal Investigator and Project Engineer. Monitoring of the procurement activity was conducted via JAT's own QC program, by Delphi's Quality Consultant, and by an ASME certified Owner's Independent Inspector to ensure that all materials purchased satisfied applicable specifications and in general met the ASME B-31.3 certification. Delphi generated an internal Quality Assurance Report on the overall engineering, fabrication, and assembly performance including procurement. An Owner's Independent Inspection Report was issued certifying that the unit meets ASME B-31.3 specifications (Appendix B).

Subtask 3.3.2.3 Assembly

Assembly of the unit was initiated in June of 1996 and culminated in Modules 1,2,3, the MCC, and the control trailer being ready for loading and shipment the first weeks of September and October 1996. The only notable exception to anticipated results in the assembly process was the fact that the gas compressor and vent hood blower were off-loaded from the unit due to congestion of equipment of level 3 of Module 1. The resulting changes impacted the installation of piping and electrical connections as well as overall system configuration previously submitted to SRS.

Subtask 3.3.2.4 Ship and Install

Module 3 was transported to SRS and off-loaded, but not placed at 673-T, on September 11, 1996. The control trailer was delivered during the latter part of September 1996 and placed adjacent to 673-T. Modules 1and 2 were transported to SRS, off-loaded, and placed in 673-T on October 10, 1996. The motor control center, the recycle compressor, the vent hood blower, and the ship loose materials were delivered to the site on October 3, 1996. Anthony Crane was used as a subcontractor service to install the unit by pick and place, since WSRC had made no provision for installation even though the cost of placement was clearly defined in the CRDA agreement. Authorization was received from DOE-FETC to proceed with the placement, since the cost of the tractor-trailers would continue to mount while the SRS procurement process was taking place.

This task also includes the final installation of the demonstration unit by the performance of the electrical terminations and piping installations. A site-approved subcontractor hired by Delphi performed this. It involved several facets of the final installation, including installation of the conduit cable tray, pulling of the electrical and instrument cable, termination and testing of the electrical and instrument cable, and verification of the reliability of the work for operations. The piping portion of the task included the installation of the piping footers for the heating and cooling piping for the unit, the installation of the piping for the heating and cooling system, the installation of the piping for the recycle compressor and vent hood blower, installation of the footers and piping for the nitrogen and oxygen feed systems, final flange to flange terminations between modules 1 and 2, and verification of the work performed in the tasks meets applicable site and construction requirements. The remaining tasks associated with final installation of the unit included the installation of a scaffold system for access of sections of module 1, movement of the control trailer to meet NFPA 101 requirements, and the electrical grounding of the unit.

The completion of this final subtask 3.2.4 fulfills the requirements for completion of Task 3, Fabrication and Assembly of the Prototype Unit.

Subtask 3.3.3 Safety and Operations Analysis

Subtask 3.3.3.1 Operations Sensitivity

Safe Operating Limits Study. One of the first tasks initiated at the beginning of Phase III was a study to determine safe operating limits for the DETOX^{SM} process. A contractual agreement was arranged with Sandia National Laboratories to enlist the services of the Explosive Projects and Diagnostics Department to define safe operating limits for oxygen, steam, and organic vapors. A test fixture was fabricated and initial tests were conducted to optimize mixing and ignition variables. A final series of tests was conducted in duplicate, which provided meaningful results with some noted aberrations. A considerable amount of effort was expended on the interpretation and verification of the data relative to its significance and implementation into safe operating limits of the demonstration unit. The final report (Appendix C) concludes that the unit may be safely operated when oxygen levels less than 3 vol% are maintained. Results of this study were implemented in the control system and safe operating limit setpoints for system sensors and monitors.

Subtask 3.3.3.2 Dynamic Systems Simulation

The Principal Investigator and Project Engineer met with University of New Mexico Chemical Engineering Department staff to define the parameters to be used in establishing a Dynamic System Model for the DETOX^{SM} demonstration unit. The scope of the model was defined to include system start-up, shutdown, and steady state conditions, as well as simulation of operating parameter excursions. Data from the model were utilized in finalizing the Title III design, component specifications, and instrument boundary conditions. The model was completed and delivered in September 1995.

Subtask 3.3.3.3 System Safety Analysis

Final Waste Form Studies. Evaluation of bench scale laboratory samples of DETOX^{SM} solution solids were conducted by Argonne National Laboratory for the purpose of determining the feasibility of using the chemically bonded phosphate ceramic as a means of stabilizing radionuclides and listed metals in a matrix suitable for land disposal. After review and approval by DOE-FETC, tests were initiated to determine formulations and loadings that would yield optimum mechanical and chemical stability meeting EPA Toxicity Characteristic Leaching Procedure (TCLP) requirements. Optimal loading of the DETOX^{SM} residues for formulations found to meet TCLP and mechanical test criteria was reported to be 36%. This loading figure was later informally reported to be improved to 70% by Argonne personnel.

Samples of actual residues obtained from testing of heavy metal contaminated surrogate wastes are planned to be forwarded to Argonne for advanced testing during the Phase IV activity. Results of such testing are strategically important to the commercial applications of the process, in that the process can be shown to produce a final waste form that meets RCRA Land Disposal Restriction (LDR) requirements.

Filtration Studies. Filtration studies were conducted on DETOX^{SM} residues anticipated to be produced from proposed waste types to test in Phase IV. Laboratory tests were conducted on various materials to determine filtration rates that were then used to determine physical characteristics required of the full-scale filtration unit. The use of filtering aides was also evaluated to improve the rate of filtration. Diatomaceous earth (10% loading) was found to yield significant improvement in filtration rates.

Advanced Physical Characteristics Studies. Metals solubility data established in Phase I of the effort was sufficient to obtain Title II design of the unit. However, as the final design and fabrication phases of the project neared, a need was identified to establish additional physical data to better estimate the properties of secondary waste streams. A determination of metals solubility, specific gravity, viscosity, and surface tension characteristics were made and test results are shown in the report entitled "Operations Sensitivity Data for DETOX^{SM} Solutions", February, 1996 Monthly Report (Appendix D).

Materials Oxidation Studies. A series of tests was conducted on candidate gasket materials and candidate materials of construction for critical components to verify manufacturers' claims for safe use. Results of these tests were used to select gasket materials and valve components critical to safe operation of the demonstration unit.

Subtask 3.3.3.4 Safe Work Plans

Preparation of test and work plans was initiated to meet site and regulatory requirements. A description of the some of the more significant documents is as follows:

- Administrative Control Procedure - Electrical
- Administrative Control Procedure - Instrument
- Best Management Practices Plan
- Delphi Environmental Health and Safety Plan
- Demonstration Safety Manual
- Incident Report Procedure
- Issue Management Plan
- Lockout Tagout Procedure
- Medical Surveillance Plan
- Emergency Operating Procedures
- Sampling and Analysis Plan
- Super Quality Plan
- Receiving, Handling, and Storage of Hazardous Materials

Documents prepared under this task were included in the Document Hierarchy (Appendix E) presented in the Operational Readiness Review in August of 1997. All safety documents have been prepared, approved and implemented, completing the requirements of this subtask.

A Process Hazards Review (Appendix F), and Fire Hazards Analysis (Appendix G), were prepared and approved by WSRC in preparation for system installation and start-up verifying that the equipment, personnel, and procedures were in place to meet all applicable regulations regarding the storage, handling, and use of hazardous materials. All aspects of site requirements imposed on the demonstration by SRS were satisfied.

Subtask 3.3.3.5 TaSSAs

Task Specific Safety Assessments were to be prepared for the specific tasks to be undertaken at WSSRAP. Preparation of a Safe Work Plan was initiated and attempts to coordinate the Process Hazards review at SRS were underway when the WSSRAP issued notice of their withdrawal from the demonstration project in January of 1996.

All subtasks 3.3.3.1 through 3.3.3.5 have been accomplished, thus completing the requirements of subtask 3.3.3. Additionally, all subtasks related to Task 3.3 Fabrication and Installation have been completed.

Section 5

Shakedown Testing

Task 3.4

5.1 Objective

Once the unit has been placed and the essential safety measures have been addressed, the last step in this phase of the project is to complete installation and hookup of the unit, complete all site requirements in preparation for startup and to ready the demonstration unit for Phase IV testing, which consists of performing shakedown tests to adequately test the proper functioning of system controls and procedures.

5.2 Procedure

This task was organized into 6 subtasks as follows:

Task 3.4.1	Site Integration
Task 3.4.2	Training
Task 3.4.3	Run-in
Task 3.4.4	Shakedown
Task 3.4.5	Solids Feed
Task 3.4.6	Inspection, maintenance, and modification

All work performed under this task has been conducted in accordance with approved Statement of Work, Demonstration Test Plan, Cooperative Research and Development Agreement with Westinghouse Savannah River Co., and the approved NEPA documentation on file with DOE-SR and DOE-FETC.

5.3 Results

Subtask 3.4.1 Site Integration

Determining the site requirements applicable to the DETOX^{SM} demonstration has been found to represent a major portion of the effort expended for this task. At the beginning of Phase III a kick-off meeting was held to define roles and responsibilities relative to the hookup and startup of the unit. Although roles and responsibilities immediately began to emerge in the form of a Cooperative Research and Development Agreement, issues regarding utility connections, waste water treatment requirements, safety features, and the authorization procedures to perform work were not addressed in sufficient detail as to determine their true cost and schedule impact on the project. Efforts primarily dealt with preparation of pads, floor coating evaluations for the containment of Module 1. The Process Hazards Review was initiated in September of 1995 but was not actively pursued until June of 1996.

In April 1996 a preliminary meeting was held at SRS to address installation issues, utilities requirements, and oxygen and nitrogen installation requirements. An inspection and walk down of the proposed site for the modules to determine information necessary to facilitate tie-ins to SRS utilities was made by a JAT engineer. In May 1996, a meeting with SRS was held to further define details of installation and to identify the information needed by SRS to prepare for installation later in the summer or early fall.

In anticipation of the completion of fabrication and subsequent installation of the unit, Delphi relocated its Project Engineer to Aiken, SC and established an Aiken office to oversee operations during the remainder of the demonstration. Numerous meetings were attended by the Project Engineer to discuss PHR, chemical inventories, safety requirements affecting installation, such as safety shower, chemical splash protection, steam venting, lightning suppression, equipment grounding, emergency lighting, and details of rigging, electrical classification, inspection and maintenance issues regarding erected structures. As time for delivery of modules approached, details regarding installation of the effluent water treatment system were raised by WSRC and the need for holding tanks (2) to collect process water was first made known to Delphi. Late in September, WSRC revealed that there was insufficient time to prepare the procurement of the crane services to place the unit upon delivery. After discussion with DOE-FETC, it was agreed that Delphi would arrange for the placement of the unit upon delivery, but that installation was clearly the responsibility of site. The unit was delivered to the site on October 3, 1996. Modules 1 and 2 were delivered to the site on October 10, 1996. Module 3 was delivered to the site on September 11, 1996.The motor control center, the recycle compressor, the vent hood blower, and ship loose materials were delivered to the site on October 3, 1996.

The ensuing six months were spent addressing costs and schedule impacts related to installation. The efforts by Delphi to resolve the issue of installation are well documented in the monthly reports covering this period. During this time, the system components were labeled, and preparation of the gas calibration systems, HCl addition systems, GC/MS installation, effluent water system, and the vent stack system were all defined by engineering calculations and/or drawings. Preparation of documentation was also initiated during this period.

In March of 1997, Delphi was given authorization to proceed with installation of the units. The services of a site-approved subcontractor were enlisted and after meeting all site requirements for training and certification, work on installation finally commenced in April of 1997. The installation of electrical and mechanical systems continued until

August of 1997. At that time an Operational Readiness Review (ORR) was conducted to verify that the equipment, personnel and documentation met the requirements of SRS. Subsequent to the ORR, a checklist of items was generated involving the modification of specific documents, verification that the unit met ASME 31.3 standards, and that training of personnel was completed. Work has continued to perform functionality and run-in tests on subsystems and components, to verify that the system is fully functional, and to verify that all alarms and controls are duly calibrated. The hot oil and chilled oil systems were charged to the unit in December 1997.

Remaining work to be performed on this subtask is the loading of DETOX^{SM} to the unit, which is scheduled to occur in March 1998. Once accomplished, work to be performed under Task 3.3 will be completed.

Subtask 3.4.2 Training

Training of personnel to meet all site requirements has been completed including site, electrical safety, hazardous energy control, fire hazard, and first aid. Key personnel have received training in the Camile® computer process control system to ensure that qualified personnel are available on each shift to maintain safe operating conditions using the control system.

In preparation for development of a training program for chemical process technicians, the Principal Investigator and Project Engineer attended a Process Safety Management training program in March 1996 and a seminar on preparing Operating Procedures.

In May, 1996 an effort was initiated to develop a two week, performance-based, integrated training program which includes compliance with OSHA's Hazardous Waste Operations and Emergency Response (HAZWOPER) standard, aspects of Process Safety Management, site specific safety and training, fundamentals of the DETOX^{SM} process, the demonstration unit, unit operation and control, process hazards and responses, and emergency response. Performance testing was based on the Self-Assessment Computer Analyzed Testing (SACAT) method, which reinforces the learning of correct information and helps to identify and correct misinformation. The training program has been administered three times to date and preliminary results indicate the training is highly effective and meets all of the objectives of this task.

Subtask 3.4.3 Run-In and Operational Checks

A series of tests are presently being performed to verify that the system is fully functional and ready for Phase IV testing. Electrical system checks have been performed to ensure that all electrical equipment is properly installed and fully operational as per manufacturer's specifications. Instrumentation has been calibrated and tested to verify proper functionality. Extensive work has been conducted on the Camile® Control system to ensure that all system controls are functioning as designed and as specified. Significant testing has been performed on the interlock system to ensure this meets the requirements of the HAZOP. Testing of the Solids feed system indicated that the transition shroud on the shredder was not watertight and the transition piece did not allow for the removal of the classifier screen. The assembly had to be removed and reworked to ensure that would be leak tight. The transition piece had to be retrofit with a flexible boot to allow for vertical movement of the classifier screen when removal was needed for cleaning. Pressure testing of the solution cooler revealed that a tell-tail connection installed by JAT was improperly routed and resulted in the collapse of a tantalum liner. The heads on the solution cooler were shipped back to the manufacturer for rework. The load cells on the primary reactor were found to be in a bind, which causes them to measure loads incorrectly or not allow for repeatable measurement. JAT was asked to provide flexible piping connections in several sections of the feed piping to the reactor to alleviate the bind on the load cells. Other than these major items, all other system corrections and/or repairs were minor and the run-in test are ready to proceed with water checks.

Subtask 3.4.4 Shakedown Testing

Development of a Validation Plan was performed with Arthur D. Little after they were selected in concert with approval from DOE-FETC. The Validation program was formulated in a meeting at ADL April 18, 1997 with representatives of DOE-FETC, DOE-MWFA, Energetics, and Delphi in attendance. Recommendations were made to further enhance the plan and it was agreed that the validation program would provide for a complete and defensible set of certifications and claims that would be admissible to a broad range of customers and regulatory authorities. A copy of the Validation Plan objective is shown in Appendix H.

Phase III Shakedown testing using oil and paper and plastic solids was combined with Phase IV performance testing.

Validation of the performance of the DETOX^{SM} process will be done in Phase IV by comparing the measured destruction efficiencies with destruction efficiencies required by the hazardous waste regulations.

Subtask 3.4.5 Solids Feed

The work defined under this task has been combined with Phase IV organics destruction performance testing (See Note below).

Subtask 3.4.6 Inspection, Maintenance, and Modification

Work defined under this task to inspect the unit, perform maintenance and modification has been combined with Phase IV performance testing (see Note below).

Note: On December 3, 1997 a meeting with DOE-FETC, DOE-SR, DOE-MWFA, WSRC, and Delphi Representatives was convened at SRS to reevaluate the tasks required to finish Phase III and surrogate waste types to be tested in Phase IV. It was agreed that to conserve the schedule at SRS, shakedown testing with organics would be consolidated with Phase IV organics destruction performance testing, obviating the need for task 3.4.6. Work is anticipated to progress to Phase IV testing immediately after the completion of task 3.4.3, Run-In and Operational Checks.

Appendix A – NEPA Documentation Approvals

Department of Energy
Morgantown Energy Technology Center
P.O. Box 880
3610 Collins Ferry Road
Morgantown, West Virginia 26507-0880

December 18, 1995

Dr. Donald T. Robertson
Delphi Research, Inc.
701 Haines Ave., N.W.
Albuquerque, NM 87102

Dear Dr. Robertson:

I have enclosed a copy of the Morgantown Energy Technology Center's determination under the National Envrionmental Policy Act for Phase IV of the wet oxidation process project. This determination covers the entire phase, so no further action would be necessary unless there are major changes to the project.

If you have any questions, please give me a call at (304) 285-4294.

Sincerely,

Wennona A. Brown

Wennona A. Brown
Environmental Project Manager

Enclosure

cc:
W. J. Huber (w/out enclosure)
NEPA file #226 (w/out enclosure)

Department of Energy
Morgantown Energy Technology Center
P.O. Box 880
3610 Collins Ferry Road
Morgantown, West Virginia 26507-0880

December 15, 1995

MEMORANDUM FOR NEPA FILE #226

CONTRACT NO: DE-AC21-92MC29107

SUBJECT: NEPA Determination

DOE proposes to continue the wet oxidation process contracted with Delphi Research, Inc., into Phase IV demonstration. The proposed demonstration would be conducted at two sites: DOE's Savannah River Site, South Carolina, and Weldon Springs site, Missouri.

The Savannah River Site has determined that the demonstration at its site falls within a B3.10 categorical exclusion. The Argonne National Lab, who is responsible for the Weldon Springs site, has determined that the demonstration at its site falls within its site-wide EIS and needs no further documentation. Transportation of the equipment between the two sites will be in accordance with all Department of Transportation regulations concerning transport of hazardous materials.

The Morgantown Energy Technology Center accepts the NEPA determinations of the host sites. Therefore, I have determined that the Phase IV demonstration of the wet oxidation process under contract with the Morgantown Energy Technology Center is adequately covered by NEPA documentation.

John R. Ganz
NEPA Compliance Officer

Date 12/15/95

WESTINGHOUSE SA .NNAH RIVER COMPANY

INTER-OFFICE MEMORANDUM

September 9, 1996

SRT-AEC-96-0886

TO: Frank E. Lustig, SWER, 705-3C

FROM: Nancy L. Turner NLT
773-A

NOTICE OF NEPA APPROVAL (NONA) (U)

EEC No.: TC - T - 95 - 034 Rev. No.: 1
Title: DETOX Wet Oxidation Waste Treatment Technology Demonstration

It has been determined that no further National Environmental Policy Act (NEPA) documentation is required for the scope of work identified in the subject Environmental Evaluation Checklist (EEC). The proposed action is documented as being:

- ☒ Covered under sitewide Categorical Exclusion (CX): B3.6
- ☐ Covered by DOE approved CX:
- ☐ Covered by previous NEPA documentation:
- ☐ NEPA is not required as all prescreens are "NO"

The requirements of NEPA for the proposed activity have been satisfied and you are no longer restricted by NEPA to proceed with this action. Please note, however, that this concurrence relates to NEPA only. All other applicable environmental, safety, and management activities must be met for activities related to this action.

File this determination and the original Environmental Evaluation Checklist with the proposed project or activity file.

If you determine that actions or modifications outside of the originally approved scope of work must be performed, these changes must be submitted for evaluation to insure continued compliance with the National Environmental Policy Act.

Attached: Signed EEC

cc: AEC Files, 773-43A

Environmental Evaluation Checklist (EEC)

NEPA/Environmental Permits

EEC No. TC-T-96-034

Rev No. 1

Instructions

- Please print or type both sides of the checklist.
- Submit one copy of the completed Checklist with supplemental information to the Department NEPA Coordinator (DNC) and the department's Environmental Coordinator (EC). The DNC will distribute to the Site NEPA Coordinator (if applicable) for NEPA level determination and the EC will distribute the completed checklist to the EPD Permits Coordinator for evaluation and approval and send a copy to SWMD Waste Forecast Coordinator.

Activity Title and Project No. (if any)
DETOX Wet Oxidation Waste Treatment Technology Demonstration

Date 8/27/96

Checklist Preparer (Name, Organization, Location)
Frank E. Lustig, SWER, 705-3C

Phone No. 7-6372

Approved By (Manager) (Name, Organization, Location)
Mark A. Fachada, SWER, 705-3C

Manager's Signature On File

Date 8/27/96

Proposed Activity Start Date	Proposed Activity End Date	Estimated Cost	Activity Location
5/1/96	9/30/97	$668,000	TNX

Activity Description This should be a brief but thorough description of the proposed activity. Be very specific in explaining the purpose and location (a developed/non-developed area, outside/inside adjacent to existing building no., etc.). Attach a copy of Functional Performance Requirement, conceptual scope of work, maps, charts, or other equivalent information.

Summary:
The DOE is sponsoring a technology demonstration of Delphi Research, Inc.'s patented DETOX wet oxidation waste treatment process. The process is being developed primarily to treat hazardous and mixed wastes within the DOE complex as an alternative to incineration. The results of this demonstration will be intensively studied and used to validate the technology. The demonstration will be located at 673-T.

Attachments (FPR, CDR, System Description, etc.)? ● Yes ○ No

Detailed Description:

The DETOX demonstration unit consists of two transportable modules (one vertical, one horizontal) that are field connected by process piping and structural connections. The vertical module contains the reactor tank, solids feed assembly, the stack, filters, and associated equipment. The horizontal module contains the liquid feed assembly, pumps, and storage tanks. The size of the reactor is approximately 264 gallons and the waste feed rate will be 25 kg/hr. The unit is designed to be operated 24 hours a day. Operations will be periodically interrupted to collect data, make adjustments, and remove solid residues.

The DETOX technology uses wet oxidation to transform complex organic compounds into carbon dioxide and water. Organics containing chorine produce hydrogen chloride in addition to the carbon dioxide and water. Organic compounds are destroyed and metallic elements are solubilized in the oxidation solution. Liquids, sludges, and solids may be introduced to the process via siphons or hoppers; dry solids must be shredded or other wise rendered suitable for introduction into the system. The wet oxidation reaction takes place in a pressurized vessel (the primary reactor with stirrer) at a temperature of approximately 392 F and a pressure of 110 psig. The oxidation of organics is performed by ferric iron with platinum and ruthenium acting as co-catalysts. As the ferric iron oxidizes organics, it is reduced to ferrous iron. Oxygen is used to oxidize the ferrous iron, thus regeneration ferric iron, to carry on the continuous oxidation cycle.

Please see the attached document for additional supporting information.

NATIONAL ENVIRONMENTAL POLICY ACT (NEPA)
CHECKLIST

EEC No. TC-T-95-034

Rev No. 1

Pre-Screen Evaluation: *

Will the proposed activity:

• Result in a change in emissions, generation rates, or new discharges of hazardous, radioactive, petroleum substance, or other pollutants from a facility or process to the environment (air, water, land, etc.)?	● yes ○ no
• Be located outside of a previously developed area and have the potential for environmental impact.?	○ yes ● no
• Involve siting, construction, modification, or D&D of facilities or processes which could potentially result in an environmental impact.?	○ yes ● no
• Potentially affect environmentally sensitive resources such as floodplain/wetlands, sites of archeological significance, threatened or endangered species and/or their habitat, special sources of water (i.e., aquifer)?	○ yes ● no
• Pose some change in the level of health and/or safety risks (e.g., result in an Unreviewed Safety Question)?	○ yes ● no
• Involve site characterization, environmental monitoring, or field research programs?	○ yes ● no

*Note: - If any unknown, call Department NEPA Coordinator (DNC) for consultation.
- If all are "No", no further NEPA action needed. Consult with DNC to verify; file with project & complete PERMITS CHECKLIST.
- If any are "Yes", complete rest of NEPA CHECKLIST & the PERMITS CHECKLIST.

Environmental Impacts Evaluation: (Note: If any are "Yes", provide specifics/supplemental information.)

Air	
• Will there be a new air emission or a change in the quantity or quality of an existing air emission?	● yes ○ no
Surface Water	
• Will there be a liquid release to streams, swamps, wetlands, seepage basins, storm drains, process sewers, ponds, or lakes?	○ yes ● no
• Will river or stream water be utilized?	○ yes ● no
Groundwater	
• Will there be a discharge to groundwater?	○ yes ● no
• Will groundwater be utilized?	○ yes ● no
Safety	
• Is there a potential exposure to hazardous substances (e.g., radiological/toxic/chemical materials)?	● yes ○ no
• Is there a potential for explosion or criticality?	● yes ○ no
• Does action involve transportation of hazardous materials?	● yes ○ no
Natural/Cultural Resources	
• Is there a potential for impacts on wetlands, swamps, streams, river beds, ponds, set aside areas?	○ yes ● no
• Is there a potential impact on fish/wildlife resources or habitats?	○ yes ● no
• Is there a potential impact on protected species (e.g., sensitive, rare, threatened, endangered)?	○ yes ● no
• Is there a potential for impacting archeological sites?	○ yes ● no
• Does this action require a site clearance/site use permit (see WSRC Manual 1D, Procedure 3.02)?	○ yes ● no
• If a site of five acres or more is required, contact Site Land Use Coordinator.	

For Department NEPA Coordinator Use Only

• Are there potential cumulative effects when combined with other actions?	○ yes ● no
• Is the proposed activity a component of a larger line item project? ____________ (document title/number)	○ yes ● no

Department NEPA Coordinator (Name, Organization, Location)			Phone
Nancy L. Tumer	SRTC & TNX	773-A	5-5641

Decision ☒ Approved by DNC CX Applied: B3.6
☐ No further NEPA is required as all prescreens are "NO"
☐ Forwarded to Site NEPA Coordinator

DNC Signature

Date 9/9/96

For Site NEPA Coordinator Use Only

☐ DOE Approved CX CX Applied: ____________ 10 CFR 1021 dtd.: 1996

☐ Covered by previous NEPA Documentation? (CX, EA, EIS) ____________

☐ Additional NEPA Documentation Required ☐ EA ☐ EIS ☐ SA (document title/number)

Site NEPA Coordinator

SNC Signature

Date

ENVIRONMENTAL PERMITS CHECKLIST

EEC No. IC-T-95-034

Rev No. 1

General:

Does this activity involve any land disturbance which may potentially result in erosion or sedimentation? ○ yes ● no
(If "yes", what is the approximate disturbance?)
☐ Less than 1/2 acre ☐ 1/2 acre to 2 acres ☐ 2 to 5 acres ☐ Greater than 5 acres

Will the proposed activity install, modify, or remove an (including tie-in to) Underground Storage Tank: ○ yes ● no

Will the proposed activity consist of a Renovation or Demolition to an existing building/structure? ○ yes ● no
(Please specify): ☐ Renovation ☐ Demolition
Is asbestos containing material present? ○ yes ○ no
"If "no", inspector signature and license number required
Inspector Signature: ____________ License Number: ____________

Will you import or manufacture a new chemical substance? ○ yes ● no

Will the proposed activity impact a Site Evaluation Area or RCRA CERCLA Area or an associated 200 ft Buffer Zone? ○ yes ● no

Will the proposed activity involve construction or modification, or to a facility or process where the potential exists for a radioactive emission? ○ yes ● no

Air:

Will the proposed activity impact a non-radionuclide air emission source? (answer "yes" if any of the following are "yes") ● yes ○ no

- Will the project install or modify a piece of equipment which will emit, or have the potential to emit, an air emission?
- Will the project modify (including demolition) an existing permitted facility or process, which emits an air emission?
- Will the project modify (including demolition) an existing facility or process, not already permitted by SCDHEC, which emits, or has the potential to emit an air emission?
- Will the project be a demonstration (short term or long term) of a new technology which will emit an air emission?
- Will the project install or modify a piece of equipment that is used to sample or monitor air emissions?

Air emissions include regulated criteria pollutants (i.e., particulate matter, lead, nitrogen oxides, carbon monoxide, sulfur dioxide, volatile organic compounds (VOC's), etc.) and hazardous and toxic pollutants identified in SCDHEC R.61-62.5 Standard 8 and Section 112(b) of the Clean Air Act.

Examples of typical permitted equipment or process air emission sources include, but are not limited to the following:

- coal or fuel oil fired boilers
- diesel generators
- diesel powered equipment
- process feed chemical storage tanks
- fuel oil storage tanks
- waste combustion incinerators
- paint booths
- lead melters
- air strippers, etc.
- degreasing operations
- HVAC and chiller equipment

Groundwater:

Will the proposed activity: (See "HOW" Manual WSRC-IM-91-69 for permitting guidance)
Install a monitoring well or piezometer(s)? ○ yes ● no
Involve subsurface penetration for a hydrogeological investigation, or characterization? ○ yes ● no
Involve the injection of a fluid, gas, or air mixture into the subsurface? ○ yes ● no
Involve the extraction of a fluid or air mixture from the subsurface? ○ yes ● no

Wastewater:

Will the proposed activity install, construct, modify, or demolish: (See "HOW" Manual WSRC-IM-91-69 for permitting guidance)
A sanitary/industrial process wastewater treatment system? ● yes ○ no
A sanitary/industrial process wastewater collection system? ● yes ○ no
A pump station(s) to transfer sanitary/industrial waste? ○ yes ● no
A septic tank/tile field system? ○ yes ● no
A stormwater management system? ○ yes ● no

Domestic Water:

Will the proposed activity install, construct, modify, or demolish:
A domestic water distribution/treatment system? ○ yes ● no
A domestic or process water well? ○ yes ● no

ENVIRONMENTAL PERMITS CHECKLIST (cont.)

EEC No. TC-T-95-034
Rev No. 1

Wastes:

Will the proposed activity install, construct, modify, demolish, or otherwise impact a RCRA permitted facility? ○ yes ● no

Will the proposed activity generate a mixed waste? ○ yes ● no

If yes: Does a wastestream with similar characteristics currently exist at SRS? (Consult with Facility Environmental Coordinator if assistance is needed) ☐ yes ☐ no

Will the proposed activity generate a hazardous waste? ● yes ○ no

Will you be sending hazardous/mixed waste to other on-site Treatment/Storage/Disposal (TSD) facilities? ○ yes ● no

Is the TSD permitted to accept this waste? ○ yes ○ no

(If "yes", provide the following)

- name of receiving facility ______
- source used to confirm facility can accept waste ______

Is this activity to take place at an existing TSD (including groundwater unit, vadose zone, process sewer, Carolina Bay, secondary containment system, etc.)? ○ yes ● no

Would this activity impact an existing TSD (including changing or improving stormwater runoff/runon drainage, security, communications, electrical, etc.)? ○ yes ● no

Does this activity involve Research and Development (R&D)? ● yes ○ no

(If "yes", answer the following)

- Does it involve hazardous/mixed waste? ☒ yes ☐ no
- Does it treat more than 1,000 kg of hazardous waste? ☒ yes ☐ no
- Does it involve polychlorinated biphenyls (PCBs)? ☐ yes ☒ no
- Will this activity continue for more than 30 days? ☒ yes ☐ no
- Will more than 250kg of hazardous waste be introduced into treatment in a single day? ☒ yes ☐ no
- Does it treat more than 500kg of soil, water, or debris contaminated with acute hazardous or 1kg of acute hazardous waste? ☐ yes ☒ no
- Does it involve the placement of hazardous waste on the land or open burning of hazardous waste? ☐ yes ☒ no

Waste Identification, Generation and Management:

Will the proposed activity include the purchase of lead or lead components? (If "yes", complete OSR 29-6 for each item and submit with Checklist) ● yes ○ no

Will the proposed activity disturb soil, sludge or water at or near a RCRA/CERCLA Unit or Site Evaluation Area? ○ yes ● no

If "yes", were any listed wastes disposed of at this facility? (Consult with Facility EC if assistance is needed) ○ yes ○ no

If "yes", please contact EPD for guidance regarding the Investigation-Derived Waste Management Plan.

Does this activity result in a new liquid and/or solid waste generation (one-time or continuous), or a change in the quantity or the characteristic of an existing waste stream? If "yes" check all that apply: ● yes ○ no

- ☐ TRU
- ☐ Mixed - Covered by LDR FFCA
- ☐ Low-Level
- ☐ High-Level
- ☐ Hazardous
- ☒ Suspect Hazardous
- ☐ Sanitary/Industrial
- ☐ Used/Waste Oil
- ☐ TSCA (PCB)
- ☒ Wastewater
- ☐ Acute Hazardous
- ☐ Other...(specify) ______

Where will waste be stored/disposed/treated? See attached documentation

Is the facility permitted to manage this waste? ○ yes ● no

If "yes", complete the following items and submit with the Checklist See attached documentation - EPD

- ☒ Source utilized to confirm facility is permitted to accept the waste.
- ☒ Description of generated waste.
- ☒ Dates generation is to begin/end.
- ☒ Estimate of waste generation rate for each category.
- ☒ Description of activity/process generating waste.
- ☒ Description of waste reduction principles (reducing the volume, mass, or toxicity) for this activity.

Has the proposed activity been evaluated for waste minimization/pollution prevention? ○ yes ● no

Additional Comments:

This is a temporary demonstration and is not a permanent facility and will be permitted as such. All hazardous materials will be removed by Delphi after completion of testing. The anticipated test period is 10 months.

Department EC Nancy L. Turner **Phone** 5-5641 **Address** 773-A	**Signature** Nancy L Turner	**Date** 9/6/96
EPD Permit Coordinator	**Signature**	**Date**

Specific and Supplemental Information for the Environmental Evaluation Checklist, Westinghouse Savannah River Company, Savannah River Site

1.0 EEC Checklist

1.1 Activity Description

1.1.1 Activity Title, Dates, and Location

Title: DETOX^{SM} Wet Oxidation Waste Treatment Technology Demonstration

Prepared By:
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Revision Number: **2**

Date This Document Prepared: October 29, 1996

Proposed Activity Start Date: September, 1996

Proposed Treatment Start date: December, 1996

Proposed Activity End Date: September , 1997

Proposed Activity Location: Building 673-T, TNX Operations Area, Savannah River Site

1.1.2 Summary Activity Description

The US Department of Energy - Morgantown Energy Technology Center is sponsoring a technology demonstration of Delphi Research, Inc.'s patented DETOX^{SM} wet oxidation waste treatment process. The process is being developed primarily to treat hazardous and mixed wastes within the DOE complex as an alternative to incineration, but it has significant potential to treat wastes in the commercial sector. The results of the demonstration will be intensively studied and used to validate the technology. Based on the results of the validation efforts, the technology will be commercialized and used widely for waste management, pollution prevention, and other environmentally beneficial applications.

Components of the DETOX^{SM} demonstration unit are being fabricated by Jacobs Applied Technology in Orangeburg, South Carolina. Delphi has had discussions with WSRC/Environmental Protection Department representatives regarding the strategy for obtaining operating approval for the demonstration unit. Delphi and WSRC have obtained a letter of temporary approval from the South Carolina Department of Health and Environmental Control (SCDHEC) to operate the demonstration unit as a wastewater treatment unit. The demonstration unit components will be shipped by truck beginning in September 1996 to the Savannah River Site (SRS) where they will be assembled into a full-scale demonstration unit in Building 673-T in the TNX Operations Area. The location of Building 673-T within TNX is shown in Exhibit 1. The demonstration unit will be entirely contained within the building and will include numerous spill prevention measures and operational safety features. The system will be hydrotested after fabrication and then initially operated (shakedown testing) using an influent stream of pure chemical feedstocks (simulants). The DETOX^{SM} unit will then be operated using several introduced substances either already available at SRS or procured as simulants. These include liquid and solid organic hazardous materials, including wood and other oxidizable solids. Delphi anticipates that only a minimal amount of engineering and construction activity will be needed to interface the DETOX^{SM} demonstration unit with existing utilities in Building 673-T.

The duration of the demonstration will be approximately thirteen months and is set to begin in September 1996. Waste simulant treatment, however, will not begin until December 1996. The thirteen month schedule is broken into two phases. In the first phase, installation and shakedown testing will occur in which pure product simulants will be introduced to establish the ability of the unit to operate safely and reliably. The first phase is expected to last 7 months. In the second phase, operational testing will be performed using characteristic hazardous wastes provided by SRS. (If suitable hazardous wastes are not available, testing will continue with simulants.) The second phase is expected to last 6 months. The demonstration unit will operate 24 hours a day, 5 days per week and will process materials at a maximum rate of 25. kg organic/hr. Once the demonstration is complete, the unit will be dismantled and cleaned as necessary to allow transportation to the Rocky Flats Environmental Technology Site near Golden, Colorado where it will be reassembled and used for destruction and volume reduction of radioactively-contaminated organic wastes.

1.1.3 Detailed Activity Description

The DETOX^{SM} demonstration unit consists of two transportable modules (one vertical, one horizontal) that are field-connected by process piping and structural connections (see Exhibit 2). The vertical module contains the reactor tanks, solids feed assembly, the stack, filters, and associated equipment. The horizontal module contains the liquids feed assembly, pumps, and storage tanks. After the modules are connected, the size of the assembled unit will be 24. ft. long by 16. ft. wide by 32. ft. tall. The unit is designed to be operated 24 hours a day, 5 days a week. Operations will be periodically interrupted to collect data, make adjustments, and remove solid residues.

Exhibit 1. Map of TNX Operations Area

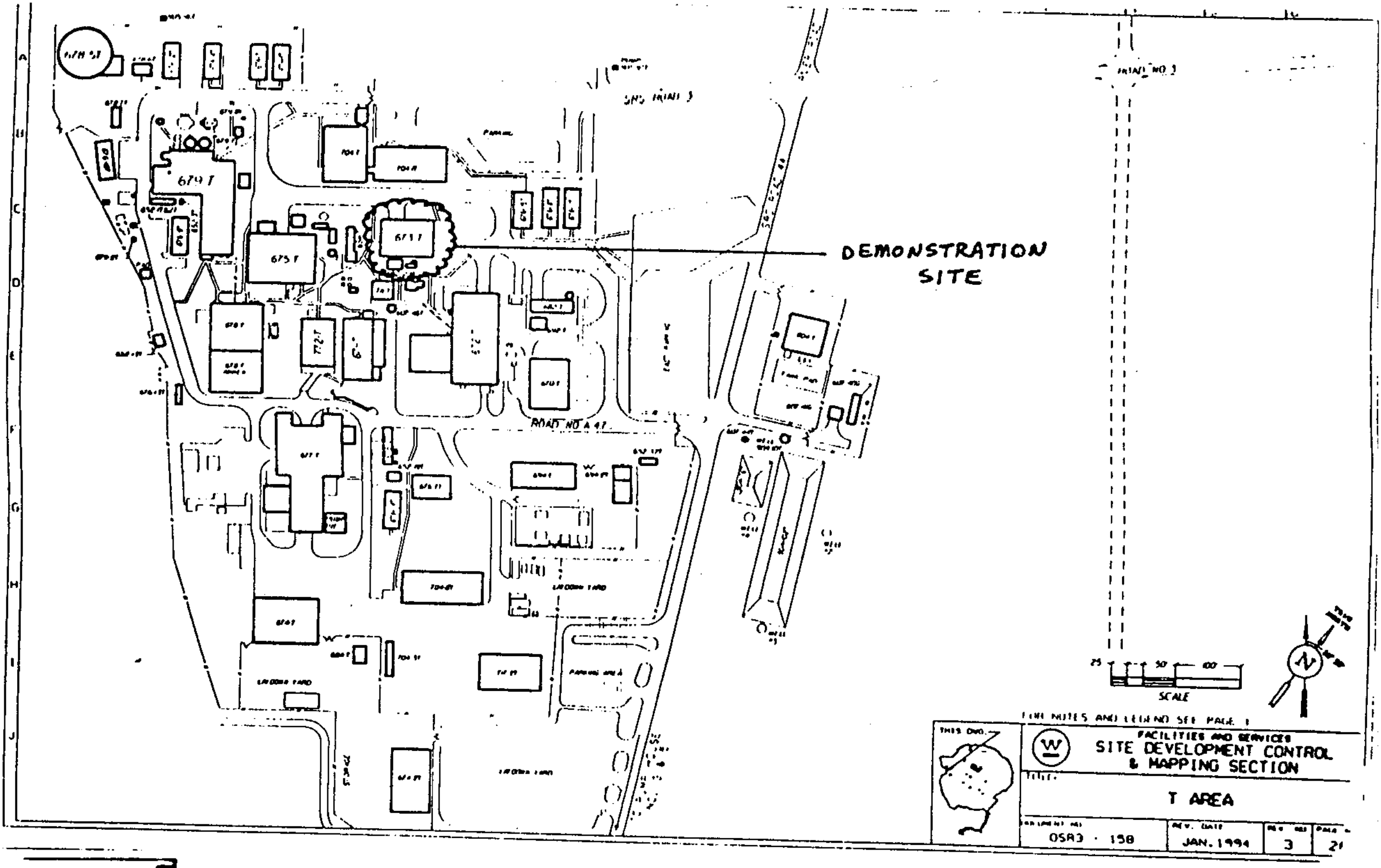


Exhibit 2. DETOX Demonstration Unit.

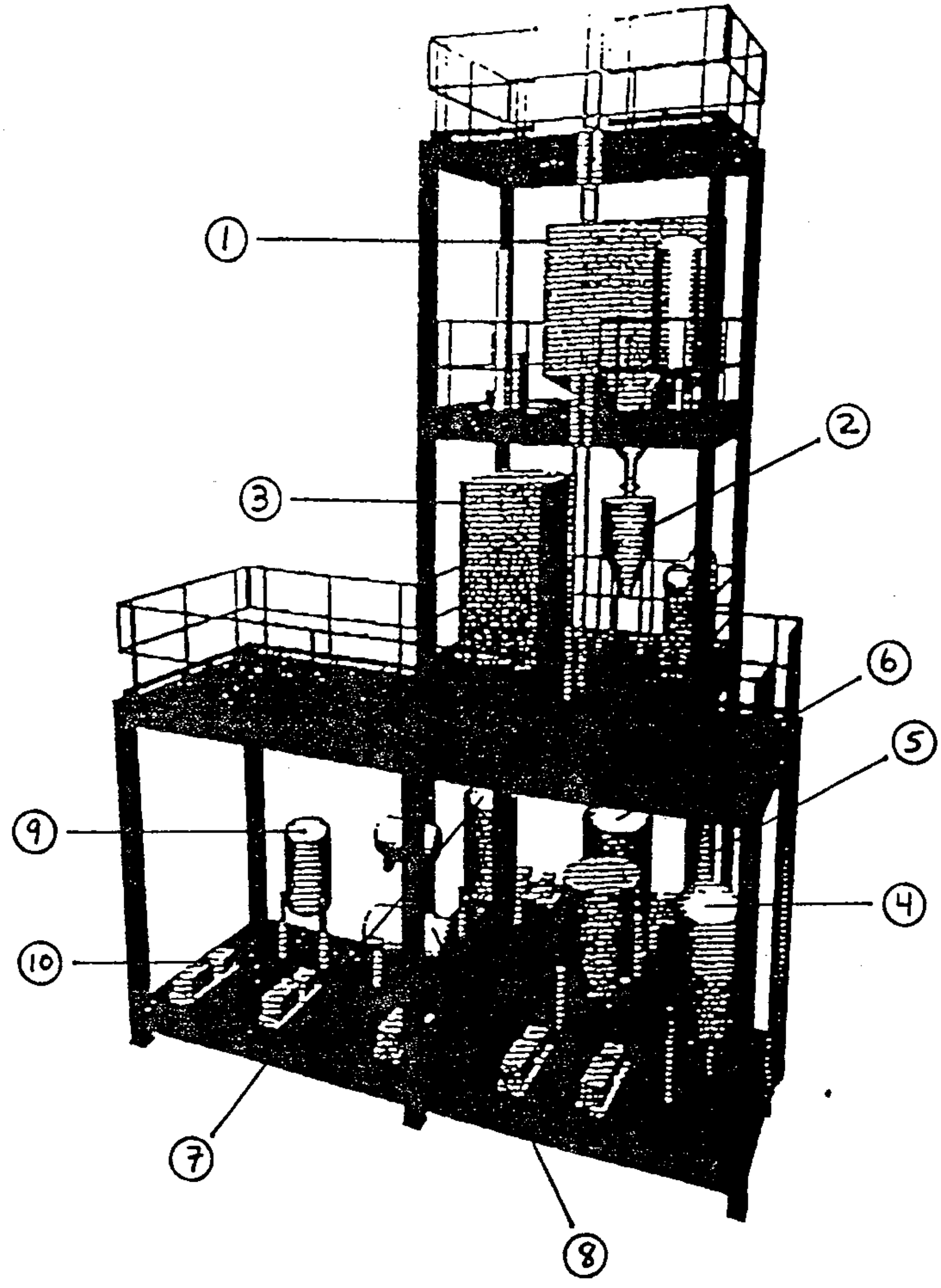


EXHIBIT 2. DETOX Demonstration Unit (cont'd)

Item Number	Description
1	Solids Feed Shredder
2	Solids Feed Hopper
3	Helical Filter to remove Precipitates/Solid Residues
4	Relief Vent Knockout Tank
5	Granular Activated Carbon Bed
6	Primary Oxidation Reactor
7	Secondary Oxidation Reactor
8	Overhead Distillate Receiver Tank
9	Wastewater Collection and Neutralization Tank
10	Pumps

There are two reactor vessels in the demonstration unit, a primary tank and a secondary tank. The size of the primary reactor is approximately 228. gallons and the maximum feed rate to this vessel will be 25. kg organic/hr. The size of the secondary reactor is approximately 162. gallons and is fed by offgas from the primary reactor. Liquid feeds are completely contained in process lines until their introduction into the primary reactor. The solids feed assembly consists of a shredder, a feed hopper, and a pump. The hopper is completely sealed to the atmosphere and is connected directly to the shredder. The potential for gaseous and particulate fugitive emissions is low, since the shredder is equipped with a vent hood. The exhaust from this hood is passed through a carbon bed to remove volatile organic compounds (VOCs), then through a HEPA filter to remove any particulates before being released to the atmosphere. All vents to atmosphere will pass through a single stack directly to the outdoors. Operations will be interrupted should the emissions control devices fail.

The DETOX^{SM} technology uses catalyzed wet oxidation to transform complex organic compounds into carbon dioxide (CO_2) and water. Chlorinated organics produce hydrogen chloride (HCl) in addition to CO_2 and water. Organic compounds are destroyed and metallic elements are solubilized in the oxidation solution. Liquids, sludges, and solids may be introduced to the process via siphons or hoppers; dry solids must be shredded and slurried or otherwise rendered suitable for introduction into the system. The wet oxidation reaction takes place in a stirred pressurized vessel (the primary reactor) at a temperature of approximately 473. K (392. F) and a pressure of 760. kPa (110. psig). The oxidation of organics is performed by ferric iron with platinum and ruthenium acting as co-catalysts. As the ferric iron oxidizes organics, it is reduced to ferrous iron. Pure gaseous oxygen is used to oxidize the ferrous iron, thus regenerating ferric iron, to carry on the continuous oxidation cycle. Exhibit 3 illustrates the process chemistry.

Offgases from the primary reactor are routed through a second oxidation reactor. The overhead from this secondary reactor is condensed and captured for storage, neutralization, and batch release to the wastewater treatment system. The secondary reactor will operate at very similar conditions of temperature and pressure and provides treatment in a manner similar to the primary reactor, except it will be sparged rather than stirred. Prior to discharge, the wastewater will be neutralized with aqueous caustic (sodium hydroxide) and the pH tested to ensure that neutralization is complete. Tailgas is vented to atmosphere after passage through a carbon bed and a HEPA filter. Precipitated solid residues will be removed from the primary reactor by filtration and stored in drums in a sheltered environment prior to testing and disposal. The residues will be stored properly if they are hazardous.

The demonstration unit has been designed to minimize the likelihood and consequence of spills. Spill prevention features include:

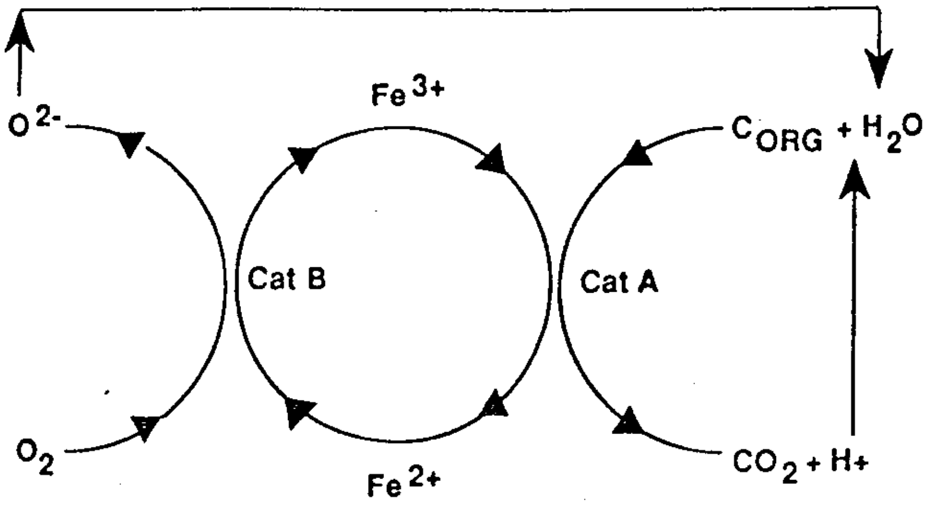
- Unflanged vessel bottoms to reduce the likelihood of gravitational leakage;
- Tantalum-lined vessels to resist corrosion and prevent leakage;
- Tantalum- and TEFLON®-lined pipes to resist corrosion and prevent leakage;
- Placement of the entire unit in an impermeable containment basin sized to accommodate the total volume of all process vessels;
- Elevated pipes and vessels to facilitate leak detection;

Exhibit 3.

DETOX Process Description

$$C^{0(AVG)}_{ORG} + 4Fe^{3+} + 2H_20 \xrightarrow{\text{Catalyst A}} CO_2 + 4Fe^{2+} + 4H^+ \quad (1)$$

$$4Fe^{2+} + O_2 + 4H^+ \xrightarrow{\text{Catalyst B}} 4Fe^{3+} + 2H_2O \quad (2)$$



- Placement of liquids to be pumped into the primary vessel in the containment basin;
- Placement of the entire demonstration unit and containment basin in a building to prevent rainfall from contacting and exacerbating a spill; and
- Placement of spill response kits, equipped specifically for potential DETOX^{SM} releases, at strategic locations around the equipment.

Exhibit 4 is a simplified schematic of the DETOX^{SM} demonstration unit that highlights some of the measures to prevent and control spills. As a precaution, Delphi has the capability of inerting the reactor headspaces with nitrogen to prevent the formation of a flammable mixture in the event of an upset. This is also shown in Exhibit 4.

The DETOX^{SM} process will be initially tested using pure feedstocks as simulants of the hazardous substances mentioned previously. After the unit has been proven safe and reliable, actual wastes will be introduced. All hazardous materials introduced during the demonstration at SRS, with the possible exception of the pure feedstock simulants, will come from SRS.

However, the hazardous waste inventory at SRS is not predictable and Delphi cannot be sure that a chosen waste stream will be available when needed for introduction into the DETOX^{SM} demonstration unit. Therefore, Delphi has prepared a list of anticipated feed materials based on seven waste types to be introduced to the DETOX^{SM} unit as part of the demonstration. These seven feed types are: hydrocarbon oils, chlorinated solvents, non-chlorinated solvents, organic (oxidizable) solids, wastewaters, scintillation fluids, and chlorinated oils. The feeds list, including estimated quantities, is presented in Exhibit 5. The anticipated feed materials are all simulants except the railroad ties and organic-contaminated wastewater, which constitute actual SRS waste streams. The oxidizable solids feed is a mixture of approximately 30. wt% poly(vinyl chloride) sheeting, 40. wt% polyethylene sheeting, 5. wt% latex rubber, and 25. wt% paper. Even though Delphi is preparing to process only simulants during the demonstration (except railroad ties and wastewater), their desire is to demonstrate treatment of actual characteristic hazardous wastes (listed hazardous wastes will not be used). All hazardous wastes provided by SRS for demonstration testing will be transported to the unit and staged prior to introduction in accordance with the requirements of the South Carolina Hazardous Waste Management Regulations (SCHWMR).

There will be three classes of waste coming out of the DETOX^{SM} demonstration unit: wastewater discharges, air emissions, and solid residues. Delphi estimates that approximately 3200. - 3250. kg of solid residues will be produced during the thirteen months of demonstration operations at SRS. These solid residues will consist of approximately 3000. kg of ferric phosphate and 200. - 250. kg of hydrated ferric oxide containing small amounts of various metals, primarily lead and mercury added for test purposes. The ferric phosphate is a by-product of oxidizing tributyl phosphate during the shakedown phase of the demonstration. Delphi anticipates that this residue will be non-hazardous. The heavy metals-bearing hydrated ferric oxide will be the result of precipitating the solid portion of the DETOX^{SM} oxidation solution after operations involving waste feeds are complete. The metals will have originated in the waste feeds to the reactor unit. As much as 3.7 wt% each of lead and mercury (as their chloride salts) will be present in the ferric oxide residue. The chloride salts of cerium and neodymium (as much as 1.8 wt% each) may also

Exhibit 4. DETOX^{SM} System Schematic

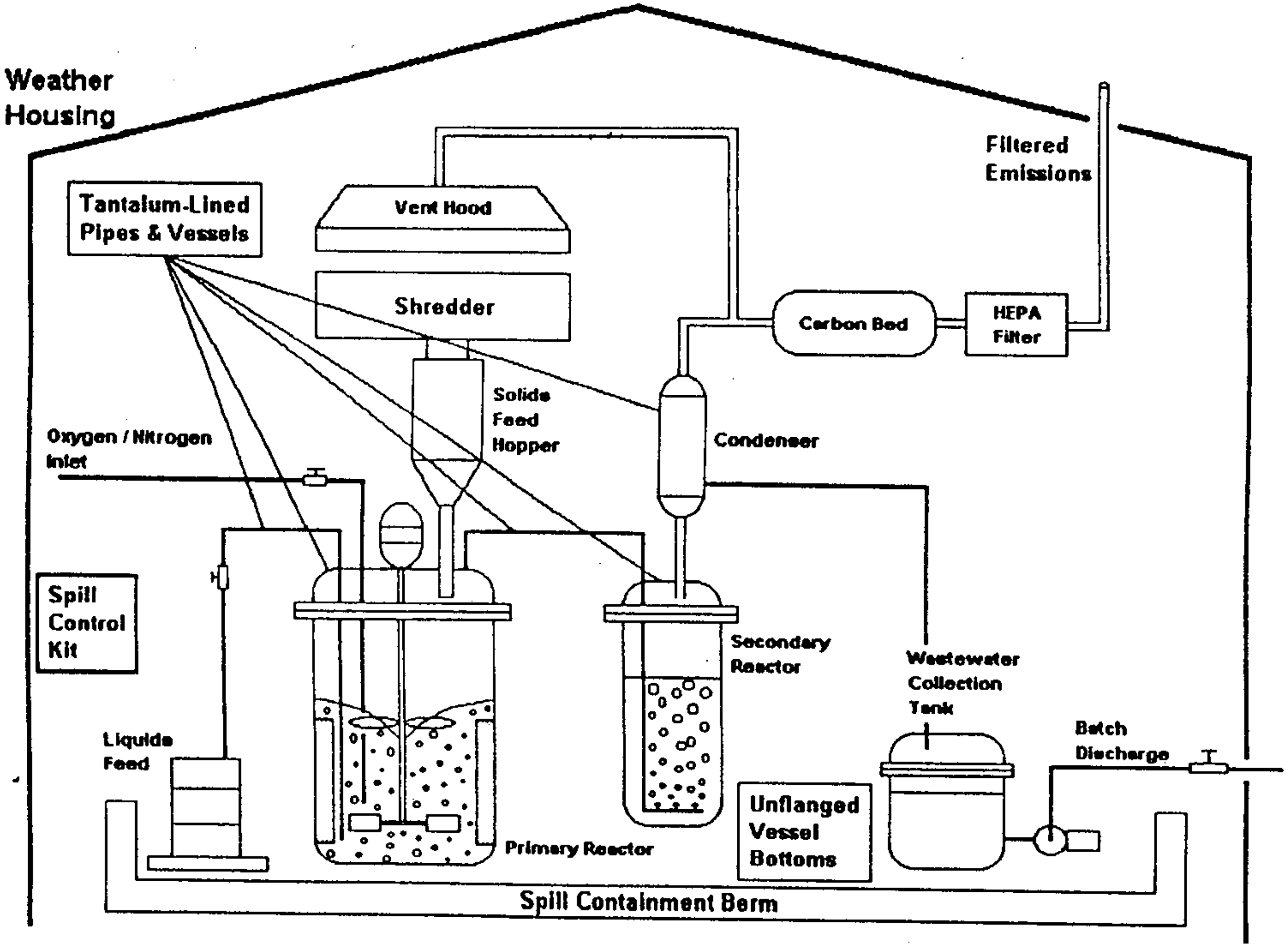


Exhibit 5. List of Anticipated DETOX Demonstration Unit Simulant Feeds and Solid Wastes

Demonstration Phase	Anticipated Feeds to Unit	Anticipated Contaminents	Anticipated Total Feed Quantity (kg)	Solid Waste Generated During Demonstration Phase	Solid Waste Disposition
Shakedown Testing	Mineral Oil	none	3903	Non-hazardous ferric phosphate residue (approx. 3200 -3250 kg).	Transferred to SRS for off-site disposal.
	Tributyl Phosphate	none	5103		
Operational Testing	Mineral Oil	1000 ppmw each Pb and Hg	7300	Suspected hazardous solid waste made up of hydrated ferric oxide containing 3.7 wt% each of lead and mercury as the chlorides (approx. 200 - 250 kg). May also contain 1.8 wt% each cerium and neodymium as the chlorides.	WSRC Solid Waste Management will handle; WSRC will manifest and Delphi will pay for analysis, transportation, and disposal.
	Wastewater (1) Blended with Mineral Oil	165 ug/L nitrate 16400 ug/L silica 5413 ug/L sulfate 63 ug/L aluminum 13 ug/L barium 19 ug/L zinc	7300 (4867 kg wastewater + 2433 kg mineral oil)		
	1:1 Toluene:Acetone	none	7300		
	1:1:1 Trichloroethylene: 1,4-Dichlorobenzene: Methylene Chloride	none	7300		
	Railroad Ties (1)	soaked with creosote	3650		
	Oxidizable Solids (mix of paper, PVC, latex, and polyethylene)	1000 ppmw each Ce and Nd 4000 ppmw naphthalene (2) 3000 ppmw 1,2-dichlorobenzene (2) 3000 ppmw chlorobenzene (2)	3650		
	TRIM SOL (chlorinated machining lubricant)	none	416		
	HYDROCOUNT (scintillation fluid)	none	416		

1) Wastewater and railroad ties are actual SRS waste streams.
2) Organic spikes added to determine destruction efficiency.

be present. The solid ferric oxide residue will be tested using EPA Method 1311 Toxicity Characteristic Leaching Procedure and disposed of accordingly. Delphi intends to remove a small amount (5 - 10 kg) of this solid waste and transport it to their laboratory facility in Albuquerque, NM for the purpose of conducting a treatability study. The treatability study will investigate the suitability of a chemically-bonded phosphate ceramic technique developed by Argonne National Laboratory for permanently stabilizing the DETOX^{SM} solid residues for ultimate disposal.

Delphi plans to discharge wastewater to one of two temporary storage tanks about every 1 - 2 hours. The amount of wastewater discharged in each batch will range from about 32. kg to about 200. kg, depending on the material being processed and the discharge interval. Mass balance calculations have shown that when mineral oil is being processed, about 32. kg/hr of wastewater should be produced, and when tributyl phosphate is being processed, about 100. kg/hr of wastewater should be produced. Since the demonstration unit will operate 24 hours per day, the amount of wastewater discharged in a 24 hour period will range from about 200. gallons to about 630. gallons. Prior to discharge to the temporary holding tanks, the wastewater will be neutralized with aqueous caustic (sodium hydroxide) and the pH tested to ensure that neutralization is complete. As currently planned, wastewater will be discharged from the DETOX^{SM} unit in batch fashion through the existing organics removal facility (ORF) to the TNX effluent treatment plant (ETP). Discharge to the ORF from the temporary holding tanks will take place every 2 - 3 days.

Delphi estimates that no metals will be released in the wastewater from the demonstration unit because of the low temperature nature of the process and the solubility of many heavy metals in the oxidation solution. Bench-scale tests have confirmed this estimate for cerium and neodymium. However, there is the very small probability that lead and mercury could make their way into the wastewater. Therefore, as a precaution, the wastewater produced during processing of feeds with added metals will be tested for those metals. If lead and mercury are present, the wastewater will be passed through the TNX ion exchange resin facility to remove these metals prior to being sent to the ORF for removal of organics.

During waste processing, CO_2, small amounts of oxygen and water vapor, HCl, and VOCs are vented to atmosphere. Most of the HCl and water vapor are condensed out of the overhead gas stream leaving the reaction section of the unit. Tailgas from the reaction section, including the remaining HCl and moisture, is passed through a carbon bed to remove VOCs. The control efficiency of the carbon bed was estimated to be 95% for each compound. The estimated emission rate of HCl is low, about 154. lb/month, hence Delphi does not plan to operate with a lime bed or caustic scrubber to remove the HCl produced.

Delphi has identified four primary chemical compounds (other than water vapor, carbon dioxide, and oxygen) that will be released in the air emissions from the DETOX^{SM} demonstration unit. These four are: HCl, methyl chloride, methylene chloride, and chloroform. Their estimated emissions after the control equipment are listed in Exhibit 6.

The emissions listed in Exhibit 6 are a worst case estimation based on a specific configuration using mineral oil feed with azeotropic composition of HCl/water leaving the reaction vessel.

Exhibit 6. Estimated Air Emissions (After Control Equipment) from the DETOX^{SM} Demonstration Unit

Parameter	Estimated Value	Units
HCl Emission Rate	0.027	g / s
	154.	lb / month
HCl Emission Concentration	1.4	g / m^3
Methyl Chloride Emission Rate	2.5×10^{-5}	g / s
	0.14	lb / month
Methyl Chloride Emission Concentration	1.3×10^{-3}	g / m^3
Methylene Chloride Emission Rate	1.2×10^{-5}	g / s
	0.069	lb / month
Methylene Chloride Emission Concentration	6.4×10^{-4}	g / m^3
Chloroform Emission Rate	5.7×10^{-5}	g / s
	0.33	lb / month
Chloroform Emission Concentration	3.1×10^{-3}	g / m^3

Mineral oil was one basecase used in designing the demonstration unit. The identity of the VOCs exiting the unit is only partially dependent on the type of waste feed. Emissions from other feeds will be very similar in both identity of the constituent and the emission rates.

Oxygen, water vapor, HCl, and CO_2 will not be removed from the vent gas stream, although these gases will pass through the control equipment prior to atmospheric outfall. Delphi estimates the emissions of oxygen, water vapor, and CO_2 at 4.3 kg/hr (6811. lb/month; 63. g/m^3), 0.2 kg/hr (317. lb/month; 2.9 g/m^3), and 77.6 kg/hr (123,066. lb/month; 1141. g/m^3), respectively. Acetone, though not a VOC, was estimated to be emitted at a rate of 0.21 g/hr (0.33 lb/month; 0.0031 g/m^3) after the control equipment. The exiting gas mixture is below the flammability limits for the estimated organics concentrations (see Exhibit 6).

A Level II modeling analysis was performed using the estimated emissions listed in Exhibit 6. A summary of this analysis is given in Exhibit 7. In all cases, the estimated maximum adjusted off-site 24 hour average concentrations are below the maximum allowed by SCDHEC Air Pollution Control Regulation 62.5 Standard No. 8. The results of the Level II analysis indicate that the DETOX^{SM} demonstration unit will be a negligible contributor to Site emissions.

Delphi is using commercial items specific for this demonstration application (such as valves, seals, and flanges) and they do not anticipate any emissions. Therefore, Delphi has made no estimate of fugitive emissions from these items.

Once demonstration operations are complete, the unit will be dismantled and cleaned as necessary to allow transportation to the Rocky Flats Environmental Technology Site near Golden, Colorado where it will be reassembled and used for destruction and volume reduction of radioactively-contaminated organic materials. Dismantlement of the DETOX^{SM} unit will be conducted with precautions to prevent spills. Pipes and vessels will be air-purged using instrument air already available in Building 673-T. The unit will be disassembled and cleaned entirely within the spill containment berm in Building 673-T. Cleaning water, if necessary with a small amount of

EXHIBIT 7. Level II Modeling Analysis Summary of Air Emissions From the DETOX Demonstration Unit

Toxic Pollutant	CAS No.	SCDHEC Reg. 62.5, Stand. No. 8 MAAC (ug/m^3)	Estimated Maximum Hourly Emission Rate (lb/hr)	Estimated Maximum Daily Emission rate (lb/day)	Maximum 24-hour Average Concentration factor (ug/m^3)	Estimated Maximum Adjusted Off-Site 24-hour Average Concentration (ug/m^3)
Hydrogen Chloride	7647-01-0	175.00	0.21	5.0	10.8	54
Methyl Chloride	74-87-3	515.00	1.9×10^{-4}	4.6×10^{-3}	10.8	0.050
Methylene Chloride	75-09-2	8750.00	9.9×10^{-5}	2.4×10^{-3}	10.8	0.026
Chloroform	67-66-3	250.00	4.5×10^{-4}	1.1×10^{-2}	10.8	0.12

MAAC = Maximum Allowable Ambient Concentration

Stack Parameters:

Stack Height = 4.9 m (16 ft)
Stack Internal Diameter = 0.05 m (2 in.)
Exit Velocity = 9.6 m/s
Exhaust Temperature = 298 K (77 °F)

Other Parameters:

Emission rates based on 24 hours per day operation.
Source type is a point source located 4.9 m (16 ft.) from ground level.
Used normalized maximum 24 hour average concentration factor of 10.8 for emission height of 16 ft. and 656 ft. to property boundary.
Note: Proposed demonstration site is 1500 ft. from nearest property boundary.
Used 24 hours of operation adjustment factor of 1.00.

commercial detergent, will be used sparingly to minimize waste generation. Wastewater generated during cleaning will be tested prior to introduction into the TNX wastewater treatment facility.

2.0 NEPA Checklist

2.1 Pre-Screen Evaluation

2.1.1 New Discharges to the Environment

There will be two types of discharges coming out of the DETOX^{SM} demonstration unit, wastewater discharges and air emissions.

The wastewater will contain several organic pollutants, some of which have aquatic and human health water quality limits and safe drinking water criteria specified by South Carolina DHEC. However, Delphi has estimated that the in-stream concentrations in the Savannah River (the receiving stream) of the listed pollutants will be well below the criteria specified by SCDHEC.

Delphi estimates that the wastewater will never exceed 0.04% of the stated water quality criteria value for the compounds for which these criteria are listed by SCDHEC. Delphi has also estimated Total Organic Carbon (TOC) values of the wastewater just prior to discharge to the Savannah River. The worst case estimated TOC value is 0.50 mg/L for 1:1:1 trichloroethylene:1,4-dichlorobenzene:methylene chloride (see Exhibit 5 in section 1.1.3 for a list of anticipated feeds). The NPDES discharge permit for discharges from the TNX effluent treatment plant specifies a monthly average TOC value of 20 mg/L with a daily maximum value of 100 mg/L. A detailed discussion can be found in section 3.3.

The principal constituents of the air emissions from the DETOX^{SM} unit will be hydrogen chloride (HCl) and volatile organic compounds (VOCs). The VOCs are methyl chloride, methylene chloride, and chloroform. Some acetone will also be vented (although acetone is not listed as a VOC). Delphi has estimated the emission rates for HCl and VOCs to be 0.027 g/s (154. lb/month) and 9.4×10^{-5} g/s (0.54 lb/month), respectively. The results of a Level II modeling analysis using these emission rates (see Exhibit 7 in section 1.1.3) indicate that the DETOX^{SM} demonstration will be a negligible contributor to Site emissions. More detailed discussions are given in sections 1.1.3 and 2.2.1.

2.2 Environmental Impacts Evaluation

2.2.1 Air

Installation and operation of the DETOX^{SM} demonstration unit will constitute a new air emission source. Tailgas coming from the reaction section of the unit will include hydrogen chloride (HCl), carbon dioxide (CO_2), small amounts of oxygen and water vapor, and volatile organic compounds (VOCs). The VOCs the unit will emit are methyl chloride, methylene chloride, and chloroform. The three VOCs and HCl are all identified as toxic air pollutants in SCDHEC Air Pollution Control Regulation 62.5 Standard No. 8. Their estimated emission rates after the control equipment are listed in Exhibit 6 of section 1.1.3.

During waste processing, most of the HCl and water vapor are condensed out of the overhead gas stream leaving the reaction section of the unit. Tailgas from the reaction section, including the remaining HCl and moisture, is passed through a carbon bed to remove VOCs. The estimated emission rate of HCl is low, about 154. lb/month, hence Delphi does not plan to operate with a lime bed or caustic scrubber to remove the HCl produced. The VOC control efficiency was estimated at 95% for each compound. The identities of the VOCs exiting the unit are partially dependent on the type of waste feed. Emissions from other feeds will be very similar in both identity of the constituent and the emission rates. The emissions listed in Exhibit 6 are a worst case estimation based on a specific configuration using mineral oil feed with azeotropic composition of HCl/water leaving the reactor. Mineral oil was one basecase used in designing the demonstration unit.

Acetone, though not a VOC, was estimated to be emitted at a rate of 0.21 g/hr (0.33 lb/month; 0.0031 g/m^3) after the control equipment. Oxygen, water vapor, HCl, and CO_2 will not be removed from the vent gas stream, although these gases will pass through the control equipment prior to atmospheric outfall. Delphi estimates the emissions of oxygen, water vapor, and CO_2 at 4.3 kg/hr (6811. lb/month; 63. g/m^3), 0.2 kg/hr (317. lb/month; 2.9 g/m^3), and 77.6 kg/hr (123,066. lb/month; 1141. g/m^3), respectively. The exiting gas mixture is below the flammability limits for the estimated organics concentrations (see Exhibit 6).

A Level II modeling analysis was performed using the estimated emissions listed in Exhibit 6. A summary of this analysis is given in Exhibit 7. In all cases, the estimated maximum adjusted off-site 24 hour average concentrations are below the maximum allowed by SCDHEC Air Pollution Control Regulation 62.5 Standard No. 8. The results of the Level II analysis indicate that the DETOX^{SM} demonstration unit will be a negligible contributor to Site emissions.

The potential for gaseous and particulate fugitive emissions from the solids feed assembly of the unit is low, since the shredder is equipped with a vent hood. The exhaust from this hood is passed through a carbon bed to remove volatile organic compounds (VOCs), then through a HEPA filter to remove any particulates before being released to the atmosphere. Delphi is using commercial items specific for this demonstration application (such as valves, seals, and flanges) and they do not anticipate any emissions. Therefore, Delphi has made no estimate of fugitive emissions from these items.

2.2.2 Safety

2.2.2.A Potential Exposure to Hazardous Substances

During the demonstration, there is a potential for accidental operator exposure to hazardous substances. Hazardous substances are present in the DETOX^{SM} oxidation solution, solid residues from the process, and in the waste feeds to the unit. In addition, the storage of liquid oxygen near the process presents unique safety concerns.

The DETOX^{SM} oxidation solution has a pH of 0 and is highly corrosive. It is comprised of two chemicals that themselves either are acidic (hydrochloric acid) or produce an acidic solution (ferric chloride) when introduced into water. Hydrochloric acid is corrosive; exposure to skin can cause severe irritation, inflammation, and chemical burns. Ferric chloride is also corrosive with skin exposures resulting in the same effects as with hydrochloric acid. Accidental exposure to the oxidation solution and its components could occur by leaks from the unit (including seals, valves, and flanges) and during solution preparation and handling.

At the conclusion of the demonstration at SRS, the DETOX^{SM} process will produce a hydrated ferric oxide solid residue that will be contaminated with lead and mercury chlorides, and possibly also cerium and neodymium chlorides. The lead and mercury salts are toxic, and lead chloride is a suspected human carcinogen. The potential for exposure comes in handling the hydrated ferric oxide residue as it is deposited into its lined-drum receptacles, and in taking samples for a planned treatability study, including preparing them for transportation.

In the shakedown phase of the demonstration, the DETOX^{SM} unit will be operated with pure chemical feedstocks as simulants of the hazardous substances Delphi wants to introduce later in the operational phase of the demonstration. This approach is being taken to ensure the unit can be operated safely and reliably. Once this is proven, actual wastes will be introduced. However, since the waste management system at SRS rapidly moves wastes to an appropriate off-site disposal facility, it is not possible to choose a particular waste stream for processing until shortly before the beginning of the demonstration. Therefore, Delphi cannot make a determination of the exact nature of the hazards posed by the waste feeds until the waste streams are identified. It is possible that no hazardous wastes (other than organic-contaminated wastewater) will be processed at all. In this case, Delphi will continue testing with simulants. The simulants will come from six classes of materials: hydrocarbon oils, chlorinated solvents, non-chlorinated solvents, organic (oxidizable) solids, scintillation fluids, and chlorinated oils. Delphi will add naphthalene, 1,2-dichlorobenzene, and chlorobenzene to the oxidizable solids feed as spikes to help determine destruction efficiency (see Exhibit 5 of section 1.1.3). 1,2-Dichlorobenzene is toxic and naphthalene is a suspected human carcinogen. Also, Delphi will blend mineral oil with hazardous wastewater (provided by SRS) in one test to demonstrate the applicability of the process in treating organic-contaminated wastewaters. The feed composition can be found in Exhibit 5 of section 1.1.3. The wastewater contains trichloroethylene and tetrachloroethylene, both of which are mutagens and suspected human carcinogens.

Delphi will mitigate accidental operator exposure to the oxidation solution using both administrative and engineering controls. Administrative controls include rigorous training of operator personnel in the hazards of the oxidation solution and its components, institution of safe operating procedures for the handling of the solution and its components, implementation of standard practices to detect leaks, and use of appropriate personal protective equipment. Engineering controls include proper ventilation of the area the unit is located in, use of area HCl monitors to warn of a leak or spill, unflanged vessel bottoms to minimize the potential for gravitational leakage, tantalum-lined vessels, and tantalum- and TEFLON®-lined pipes, to resist corrosion, and elevated pipes and vessels to facilitate leak detection.

Mitigation of exposure to the reactor residue, including treatability study samples, will be accomplished by wearing appropriate personal protective equipment and keeping the residue stream contained during its entire journey from the filtration section of the unit to its lined-drum receptacles. The drums will be managed according to SCHWMR provisions; the drums will be properly sealed and labeled, and stored in a sheltered environment. The drum contents will be tested using EPA Method 1311 Toxicity Characteristic Leaching Procedure and then disposed of through WSRC Solid Waste Management; Delphi will pay for analysis, transportation, and disposal. The treatability study samples will only be handled when proper personal protective equipment is worn and the area is well ventilated. The samples will be handled in accordance with SCHWMR and packaged and transported to New Mexico in accordance with provisions of the Hazardous Materials Transportation Act (HMTA) and all other applicable USDOT, South Carolina, and New Mexico regulations. Safe work practices will be instituted, including proper ventilation, and appropriate personal protective equipment will be worn when handling both simulant and actual hazardous waste feeds.

Gaseous oxygen is needed by the DETOX^{SM} process to regenerate the ferric iron oxidant. Oxygen will be supplied to the process from a 6000. gallon liquid oxygen storage tank which will be provided by an off-site vendor. Vaporizers will be used to vaporize the liquid oxygen to gaseous oxygen, which will then be supplied to the process.

The storage tank and vaporizers will remain the property of the vendor and will be serviced and maintained by vendor personnel. Security for the tank installation will be provided in the form of a chain-link fence to protect against physical damage; the gate will be kept locked when the tank is not being serviced. The installation, including the concrete pad upon which the tank will be placed, will conform to all applicable US Occupational Safety and Health Administration (OSHA), National Fire Protection Association (NFPA), and Site requirements.

Hazards associated with handling liquid oxygen include the potential for tissue freezing and accelerated combustion of flammable and combustible substances. Typically, there should be no reason for operating personnel to access the liquid oxygen tank. However, should the need arise, personnel will be required to wear insulated, impermeable gloves that are approved for use with liquid oxygen (such as CRYO-GLOVES® from Lab Safety Supply, Inc. or thick welding gloves with a high gauntlet), goggles or safety glasses, and face shields to protect against tissue freezing due to contact with the cryogenic gas. Because of the potential for tissue freezing, gloves will be large enough that they easily come off when the wearer throws his hands downward. Impermeable aprons will also be worn to protect against splashes, if the potential for splashing is deemed high. Trouser bottoms will be kept outside of footwear so that no liquid can accumulate in them.

Oxygen is itself a non-flammable gas, but it is a powerful oxidizer. Oxygen will vigorously accelerate a fire, significantly increasing its severity. Flammable and combustible materials will not be stored near the storage tank. Smoking and open flames will be prohibited in the vicinity of the tank. The installation will be placarded with the legend "OXYGEN - NO SMOKING - NO OPEN FLAMES", or an equivalent warning. These precautions will guard against a fire starting near - and subsequently involving - the storage tank installation.

2.2.2.B. Potential for Fire or Explosion

The DETOX^{SM} demonstration unit is equipped with a continuous emission monitor for volatile organic compounds (VOCs). The VOC monitor is a stand-alone flame ionization detector (FID) that operates by burning VOCs sampled from the tailgas in a hydrogen-air flame. The potential for fire lies in the use of gaseous hydrogen for the flame.

Delphi will use a hydrogen generator to supply hydrogen gas to the VOC monitor. This eliminates the need for pressurized, bottled hydrogen. The use of the hydrogen generator virtually eliminates the possibility of forming a flammable or explosive hydrogen-air mixture in the vicinity of the monitor. The generator is designed to deliver hydrogen at low pressure (maximum pressure approximately 65. psig) and automatically shut off the flow of hydrogen should a leak occur in the system or in downstream tubing. Also, the maximum quantity that is ever contained in the generator is approximately 150 mL, so a catastrophic leak would release no more than this quantity of hydrogen to the atmosphere. This quantity should quickly disperse through diffusion, virtually eliminating the chance to form a flammable or explosive mixture with air (the lower and upper flammability limits of hydrogen in air at 298. K (77. °F) and 1 atm. are 4.0 vol% and 75. vol%, respectively).

During demonstration operations, process control samples will be taken and analyzed inside the unit control trailer by gas chromatography-mass spectrometry (GC-MS). The GC-MS instrument requires hydrogen for proper operation. The hydrogen will be supplied by a second hydrogen generator similar to the one used by the VOC monitor in the unit.

2.2.2.C Transportation of Hazardous Materials

It will be necessary to transport either pure chemicals or actual wastes both to and from the demonstration unit (located in Building 673-T in TNX). In addition, liquid oxygen will be brought into the TNX area about once per month by the vendor to refill the 6000. gallon storage tank. Transportation of liquid oxygen will be in USDOT-approved equipment only.

Transportation of pure chemical feedstock simulants and components of the DETOX^{SM} oxidation solution to the unit will be done in the original containers, whenever possible. If this is not possible, properly labeled containers appropriate for the quantity being transported, and compatible with the material, will be used. Hazardous pure chemicals to be transported to the unit include hydrochloric acid and ferric chloride (corrosivity), toluene and acetone (flammability), methylene chloride, methyl chloroform, trichloroethylene, naphthalene, and 1,2-dichlorobenzene (flammability and toxicity), and lead and mercury oxides (toxicity).

If Delphi is successful in securing actual hazardous wastes for processing in the demonstration unit, then these will have to be transported on a daily basis (pending regulatory approval) from a permitted storage area to the unit. Only the amount of waste that can be processed within a 24 hour period will be transported at any one time. All hazardous wastes provided by SRS will be transported to the unit and staged prior to introduction in accordance with the requirements of the South Carolina Hazardous Waste Management Regulations (SCHWMR).

The solid ferric oxide residue produced during the DETOX^{SM} demonstration is a suspect hazardous waste due to the presence of lead and mercury chloride salts. This residue will be managed according to SCHWMR provisions; the residue will be placed in lined drums which will be properly sealed and labeled, and stored in a sheltered environment. The contents will be tested using EPA Method 1311 Toxicity Characteristic Leaching Procedure and then disposed of through WSRC Solid Waste Management; Delphi will pay for analysis, transportation, and disposal.

Treatability study samples, taken from the solid ferric oxide residue, will be handled in accordance with SCHWMR and packaged and transported to New Mexico in accordance with provisions of the Hazardous Materials Transportation Act (HMTA) and all other applicable US DOT, South Carolina, and New Mexico regulations.

3.0 Environmental Permits Checklist

3.1 General

3.1.1 Renovation or Demolition of an Existing Building/Structure

There will be minor additions to Building 673-T (TNX area) to interface the DETOX^{SM} demonstration unit with existing utilities (e.g., electricity, instrument air, etc.), to connect the unit to the organics removal facility adjacent to 673-T, to connect the unit to the TNX ion exchange resin facility to allow discharge of wastewater, to pipe in oxygen and nitrogen gases, and to accommodate the vent stack so that tailgas from the unit can be vented to atmosphere. In addition, a 6000. gallon liquid oxygen storage tank will be installed near Building 673-T to provide oxygen to the DETOX^{SM} process. This installation will include a concrete pad for the storage tank.

3.2 Air

This project will install a short-term (approximately thirteen months) demonstration unit which will release process emissions to the atmosphere. The compounds that will be emitted from the unit are hydrogen chloride (HCl), methyl chloride, methylene chloride, and chloroform. All of these compounds are identified as toxic air pollutants in SCDHEC Air Pollution Control Regulation 62.5 Standard No. 8. Delphi estimates that the emission rates of these four pollutants will be very low: the estimated emission rate being 154. lb/month for HCl, and the estimated combined emission rate for the VOCs being 0.54 lb/month. For more detailed discussions, see sections 1.1.3 and 2.2.1.

The potential for gaseous and particulate fugitive emissions from the solids feed assembly of the unit is low, since the shredder is equipped with a vent hood. The exhaust from this hood is passed through a carbon bed to remove volatile organic compounds (VOCs), then through a HEPA filter to remove any particulates before being released to the atmosphere. Delphi is using commercial items specific for this demonstration application (such as valves, seals, and flanges) and they do not anticipate any emissions. Therefore, Delphi has made no estimate of fugitive emissions from these items.

3.3 Wastewater

Installation of the DETOX^{SM} demonstration unit in Building 673-T will temporarily modify the TNX process wastewater treatment system. Modification will entail running pipe to connect the DETOX^{SM} unit with the feed tank of the organics removal facility (ORF). This connection will allow discharge of wastewater to the ORF feed tank. Wastewater produced in the DETOX^{SM} demonstration unit will be neutralized, then transferred to one of two temporary storage tanks. Each tank will have a capacity of between 3000. gallons and 5000. gallons. The tanks will be placed inside the berm that provides secondary containment for the DETOX^{SM} unit. Alternatively, if the tanks cannot be placed inside the berm, each tank will have a spill containment pool (made, for example, from polyethylene or HYPALON®) placed under it.

Wastewater that has accumulated in one of the tanks will be sampled and analyzed for volatile and semi-volatile organic compounds (VOCs and SVOCs, respectively) prior to discharge to the ORF. Under normal circumstances, wastewater can be discharged to the ORF after analytical results are received and evaluated. Under off-normal conditions, the treatability of the wastewater by the TNX ETP will need to be assessed before the wastewater can be discharged from the temporary holding tanks. Normal and off-normal discharge procedures are defined and discussed in the Appendix. Two tanks will be used so that one can receive wastewater from the DETOX^{SM} unit while the other is being tested for discharge. Wastewater will be discharged from the temporary holding tanks to the ORF every 2 - 3 days.

In cases where metals are added to the feeds, the wastewater will be tested for the presence of those metals. If metals, especially lead and mercury, are present, the wastewater will be discharged to the TNX ion exchange resin facility to remove them prior to discharge to the ORF for removal of organics. No other modification to the existing TNX wastewater facility is anticipated. The facility will simply continue to operate normally with an additional effluent stream that will come from the DETOX^{SM} unit.

3.4 Wastes

3.4.1 Generation of Hazardous Waste

The DETOX^{SM} waste treatment technology demonstration will generate potentially hazardous solid waste. A solid residue of about 200. - 250. kg of hydrated ferric oxide containing various heavy metals, including lead and mercury, will be produced at the end of the demonstration by evaporating hydrochloric acid from the DETOX^{SM} oxidation solution. As much as 3.7 wt% each of lead and mercury (as their chloride salts) will be present in the ferric oxide residue. The chloride salts of cerium and neodymium (as much as 1.8 wt% each) may also be present. This solid residue will be managed according to SCHWMR provisions; the residue will be placed in lined drums which will be properly sealed and labeled, and stored in a sheltered environment. The drum contents will be tested using EPA Method 1311 Toxicity Characteristic Leaching Procedure and then disposed of through WSRC Solid Waste Management; Delphi will pay for analysis, transportation, and disposal.

Delphi intends to remove a small amount (5 - 10 kg) of the ferric oxide residue solid waste and transport it to their laboratory facility in Albuquerque, NM for the purpose of conducting a treatability study. The treatability study will investigate the suitability of a chemically-bonded phosphate ceramic technique developed by Argonne National Laboratory for permanently stabilizing the DETOX^{SM} solid residues for ultimate disposal. The treatability study samples will be handled in accordance with SCHWMR and packaged and transported to New Mexico in accordance with provisions of the Hazardous Materials Transportation Act (HMTA) and all other applicable US DOT, South Carolina, and New Mexico regulations.

3.4.2 Research and Development Activity

The DETOX^{SM} technology demonstration is a research and development (R&D) activity. This activity is intended to validate the suitability of the technology in treating hazardous and mixed wastes that are found throughout the US Department of Energy's nationwide complex of facilities. Therefore, it is Delphi's intention to introduce characteristic hazardous waste, provided by SRS, to the unit for processing.

The hazardous waste inventory at SRS is not predictable and Delphi cannot be sure that a chosen waste stream will be available when needed for introduction into the DETOX^{SM} demonstration unit. Therefore, Delphi has prepared a list of anticipated feed materials based on seven waste types to be introduced to the DETOX^{SM} unit as part of the demonstration. These seven feed types are: hydrocarbon oils, chlorinated solvents, non-chlorinated solvents, organic (oxidizable) solids, wastewaters, scintillation fluids, and chlorinated oils. The feeds list, including estimated quantities, is presented in Exhibit 5 in section 1.1.3. The anticipated feed materials are all simulants except the railroad ties and organic-contaminated wastewater, which constitute actual SRS waste streams. The oxidizable solids feed is a mixture of approximately 30. wt% poly(vinyl chloride) sheeting, 40. wt% polyethylene sheeting, 5. wt% latex rubber, and 25. wt% paper. These simulants will be used in the demonstration if no suitable hazardous wastes can be found on-site. In most cases, more than 1000. kg of material will be processed for each waste type. This will also be the case if suitable hazardous wastes are found; approximately 7300 kg each of hazardous wastes from the hydrocarbon oils, chlorinated solvents, non-chlorinated solvents, and oxidizable solids categories will be processed.

The demonstration is scheduled to begin in September 1996 and continue to the end of September 1997, a duration of approximately thirteen months. Waste simulant treatment is expected to begin in December 1996. The unit will operate 24 hours a day, 5 days per week and treat wastes at a maximum rate of 25. kg organic/hr (600. kg organic/day).

3.5 Waste Identification, Generation, and Management

3.5.1 Purchase of Lead or Lead Components

A quantity of lead oxide may be purchased to add to simulant mineral oil feed for the demonstration (see Exhibit 5 in section 1.1.3). Delphi requires enough lead oxide to reach a concentration of 1000 mg/kg lead in the feed.

3.5.2 New Liquid and/or Solid Waste Generation

3.5.2.A Suspect Hazardous Solid Waste

The DETOX^{SM} waste treatment technology demonstration will generate potentially hazardous solid waste. A solid residue of about 200. - 250. kg of hydrated ferric oxide containing various heavy metals, primarily lead and mercury, will be produced at the end of the demonstration by evaporating hydrochloric acid from the DETOX^{SM} oxidation solution. As much as 3.7 wt% each of lead and mercury (as their chloride salts) will be present in the ferric oxide residue. The chloride salts of cerium and neodymium (as much as 1.8 wt% each) may also be present. This solid residue will be managed according to SCHWMR provisions; the residue will be placed in lined drums which will be properly sealed and labeled, and stored in a sheltered environment. The drum contents will be tested using EPA Method 1311 Toxicity Characteristic Leaching Procedure and then disposed of through WSRC Solid Waste Management; Delphi will pay for analysis, transportation, and disposal.

Delphi intends to remove a small amount (5 - 10 kg) of the ferric oxide residue solid waste and transport it to their laboratory facility in Albuquerque, NM for the purpose of conducting a treatability study. The treatability study will investigate the suitability of a chemically-bonded phosphate ceramic technique developed by Argonne National Laboratory for permanently stabilizing the DETOX^{SM} solid residues for ultimate disposal. The treatability study samples will be handled in accordance with SCHWMR and packaged and transported to New Mexico in accordance with provisions of the Hazardous Materials Transportation Act (HMTA) and all other applicable US DOT, South Carolina, and New Mexico regulations.

3.5.2.B Wastewater

Delphi plans to discharge wastewater to two temporary storage tanks about every 1 - 2 hours. The amount of wastewater discharged in each batch will range from about 32. kg to about 200. kg, depending on the material being processed and the discharge interval. Mass balance calculations have shown that when mineral oil is being processed, about 32. kg/hr of wastewater should be produced, and when tributyl phosphate is being processed, about 100. kg/hr of wastewater should be produced. Since the demonstration unit will operate around the clock, the amount of wastewater discharged in a 24 hour period will range from about 200. gallons to about 630. gallons. Prior to discharge to the temporary holding tanks, the wastewater will be neutralized with aqueous caustic (sodium hydroxide) and the pH tested to ensure that neutralization is complete.

As currently planned, wastewater will be discharged from the DETOX^{SM} unit in batch fashion through the existing organics removal facility (ORF) to the TNX effluent treatment plant (ETP). Discharge to the ORF from the temporary holding tanks will take place every 2 - 3 days.

The wastewater will contain several organic pollutants, some of which have aquatic and human health water quality limits and safe drinking water criteria specified by South Carolina DHEC.

However, Delphi has estimated that the in-stream concentrations in the Savannah River (the receiving stream) of the listed pollutants will be well below the criteria specified by SCDHEC. Delphi estimates that the wastewater will never exceed 0.04% of the stated water quality criteria value for the compounds for which these criteria are listed by SCDHEC. Delphi has also estimated Total Organic Carbon (TOC) values of the wastewater just prior to discharge to the Savannah River. The worst case estimated TOC value is 0.50 mg/L for 1:1:1 trichloroethylene:1,4-dichloroethylene:methylene chloride (see Exhibit 5 in section 1.1.3 for a list of anticipated feeds). The NPDES discharge permit for discharges from the TNX effluent treatment plant specifies a monthly average TOC value of 20 mg/L with a daily maximum value of 100 mg/L. Therefore, Delphi concludes that wastewater discharged from the DETOX^{SM} demonstration unit will not exceed either the SCDHEC water quality criteria for Savannah River in-stream concentrations of organics, nor the TOC limitation.

Delphi estimates that no metals will be released in the wastewater from the demonstration unit because of the low temperature nature of the process and the solubility of many heavy metals in the oxidation solution. Bench-scale tests have confirmed this estimate for cerium and neodymium. However, there is the very small probability that lead and mercury could make their way into the wastewater. Therefore, as a precaution, the wastewater produced during processing of feeds with added metals will be tested for those metals. If lead and mercury are present, the metals-containing wastewater will be passed through the ion exchange resin facility to remove lead and mercury prior to discharge to the ORF for removal of organics.

3.5.3 Treatment/Storage/Disposal of Newly Generated Waste

The suspected hazardous solid residue removed from the DETOX^{SM} demonstration unit will be managed according to SCHWMR provisions; the residue will be placed in lined drums which will be properly sealed and labeled, and stored in a sheltered environment. The drum contents will be tested using EPA Method 1311 Toxicity Characteristic Leaching Procedure and then disposed of through WSRC Solid Waste Management; Delphi will pay for analysis, transportation, and disposal.

Delphi intends to remove a small amount (5 - 10 kg) of the ferric oxide residue solid waste and transport it to their laboratory facility in Albuquerque, NM for the purpose of conducting a treatability study. The treatability study will investigate the suitability of a chemically-bonded phosphate ceramic technique developed by Argonne National Laboratory for permanently stabilizing the DETOX^{SM} solid residues for ultimate disposal. The treatability study samples will be handled in accordance with SCHWMR and packaged and transported to New Mexico in accordance with provisions of the Hazardous Materials Transportation Act (HMTA) and all other applicable US DOT, South Carolina, and New Mexico regulations.

Organic pollutants in the wastewater discharged from the unit will receive treatment by granular activated carbon (GAC) in the TNX organics removal facility (ORF) prior to release to the TNX effluent treatment plant (ETP). The wastewater will then be discharged to the Savannah River. Delphi estimates that no metals will be released in the wastewater from the demonstration unit because of the low temperature nature of the process and the solubility of many heavy metals in the oxidation solution. Limited bench-scale tests have confirmed this estimate for cerium and neodymium. However, there is the very small probability that lead and mercury could make their way into the wastewater. Therefore, as a precaution, the wastewater produced during processing of feeds with added metals will be passed through the TNX ion exchange resin facility prior to being sent to the ORF and ETP.

3.5.4 Facility Permits to Manage Newly Generated Waste

3.5.4.A Suspect Hazardous Solid Waste

Delphi has consulted with Lynn C. Martin of WSRC/EPD (Bldg. 742-A, 803-725-1793) concerning the acceptance criteria for wastes bearing lead and mercury chloride salts. A description of the solid residue can be found in section 3.5.2.A. WSRC Solid Waste Management will dispose of the hazardous solid wastes generated by the DETOX^{SM} demonstration through a third party under contract to SRS; Delphi will pay for analysis, transportation, and disposal.

As currently planned, generation of solid waste will occur near the end of the DETOX^{SM} technology demonstration. The generation will be a one-time event, occurring sometime between May 1997 and September 1997.

3.5.4.B Wastewater

Delphi has consulted with William L. Payne of WSRC/EPD (Bldg. 742-A, 803-725-3465) to determine if the TNX wastewater treatment facility is permitted to accept DETOX^{SM} wastewater. A brief description of the wastewater can be found in section 3.5.2.B. It appears that the TNX wastewater facility is permitted to accept DETOX^{SM} wastewater, but regulatory approval to discharge from the DETOX^{SM} unit must be obtained.

Appendix

Compliance Guidance: DETOX^{SM} Demonstration Operations at SRS

COMPLIANCE GUIDANCE: DETOX^{SM} DEMONSTRATION OPERATIONS at SRS

Date This Document Prepared: September 12, 1996
Revision Number: 0
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1.0 Introduction

1.1 Definitions

Acronym or Abbreviation	Definition
DRI	Delphi Research, Inc.
EPD	Environmental Protection Department (WSRC)
ETP	Effluent Treatment Plant (TNX)
IX	Ion Exchange (resin) Facility (TNX)
ORF	Organics Removal Facility (TNX)
SCDHEC	South Carolina Department of Health and Environmental Control
SVOA	Semi-Volatile Organics Analysis
USDOE	US Department of Energy
USEPA	US Environmental Protection Agency
TCLP	Toxicity Characteristic Leaching Procedure
VOA	Volatile Organics Analysis
WSRC	Westinghouse Savannah River Company

1.2 Purpose of this Document

The Bureau of Water Pollution Control of the South Carolina Department of Health and Environmental Control (SCDHEC) has granted permission to conduct the DETOX^{SM} waste treatment technology demonstration at the TNX Operations Area of the Savannah River Site. This document provides guidance for complying with the conditions of this approval, and with the environmental laws of the State of South Carolina. Four areas of environmental regulatory compliance are discussed:

- Anticipated feeds to the DETOX^{SM} unit;
- Wastewater discharge from the DETOX^{SM} unit;
- DETOX^{SM} solution solid residue characterization and disposal; and
- Recordkeeping and reporting.

1.3 Background

Regulatory permission to conduct the demonstration was obtained from SCDHEC in June 1996 in the form of a letter of temporary approval.[1] This approval was granted following review of an application[2] that provided the following information:

- A brief description of the planned demonstration;
- A brief description of the technology, including best management practices and spill prevention measures;
- A list of materials anticipated to be introduced into the DETOX^{SM} unit, including estimated quantities; and
- A detailed projection of chemical species and their concentrations in the DETOX^{SM} wastewater.

This approval allows the DETOX^{SM} facility to be operated as a wastewater treatment unit, and allows wastewater from the unit to be discharged to the TNX wastewater treatment facilities. Thus, the demonstration will be conducted under the joint regulatory framework provided by the South Carolina Pollution Control Act[3] and the National Pollutant Discharge Elimination System.[4]

2.0 Regulatory Requirements: Demonstration Approval Conditions

The approval period is for 18 months from the date of facility startup. The approval conditions are listed in Reference 1.

3.0 Compliance Guidance

Note: Please call Don Robertson at 505-243-3111 if you have any questions regarding this guidance.

3.1 Anticipated Feeds to the DETOX^{SM} Unit

Only materials listed in Exhibit 5 in the application[2] can be processed. The demonstration is limited to the quantities listed in Exhibit 5 (both simulant and contaminant) unless written permission to increase them is obtained from SCDHEC. Listed and characteristic hazardous wastes are not approved for treatment in the DETOX^{SM} demonstration unit. Treatment of mixed wastes is specifically prohibited.

3.2 DETOX^{SM} Demonstration Facility Startup

Facility startup commences when the first simulant (Exhibit 5 of Reference 2) is introduced into the DETOX^{SM} unit. Installation and functionality testing do not constitute startup. For example, running machinery (e.g., pumps) or boiling water to test sensors and confirm that no blockages exist in the process lines are not considered to be part of facility startup. The demonstration, including facility closure, must be completed within 18 months of facility startup.

3.3 Wastewater Discharge from the DETOX^{SM} Unit

3.3.a. Pollutants Approved for Discharge

A detailed projection of the identities and concentrations of organics and metals to be found in the wastewater of the DETOX^{SM} demonstration unit is presented in Exhibit 6 of the application.[2] Only those pollutants listed in Column 2 of Exhibit 6 (Constituent in Unit Effluent) are approved for discharge. The maximum quantities of these pollutants approved for discharge are listed in Column 4 of Exhibit 6 (Projected Concentration Exiting Unit).

3.3.b. Approved Method Of Wastewater Discharge

At present, the only approved method of discharging wastewater from the DETOX^{SM} facility is through the TNX wastewater treatment facilities. If the wastewater contains no metals (e.g., lead and/or mercury), it must be discharged to the ORF. The ORF provides treatment by removing organics before the stream enters the ETP. Wastewater that is found to contain metals must first be discharged to the IX for removal. The wastewater will automatically be transferred to the ORF from the IX.

Wastewater can neither be discharged to another wastewater treatment facility, nor shipped off-site, without prior approval from SCDHEC. WSRC/EPD will coordinate the approval.

3.3.c. Wastewater Discharge: Normal Procedure

The following procedure should be employed when discharging wastewater from the DETOX^{SM} demonstration unit in order to ensure compliance with the demonstration approval.

1. Transfer neutralized wastewater from the DETOX^{SM} demonstration unit to a temporary storage tank.

2. Collect VOA and SVOA samples as directed by the Project Engineer or his designee. If materials containing metals (e.g., lead and/or mercury) have been processed, collect a metals sample as well.

3. After receiving the VOA and SVOA (and metals, where appropriate) analytical results, the Project Engineer or the Principal Investigator should review the results.

4. If there are no organics present that are different from the list of pollutants in Column 2 of Exhibit 6 of the application,[2] and their concentrations are below the levels listed in Column 4 of Exhibit 6 of the application, the wastewater may be discharged to the ORF.

5. If there are no organics present that are different from the list of pollutants in Column 2 of Exhibit 6 of the application,[2] and their concentrations are below the levels listed in Column 4 of Exhibit 6 of the application, and if metals are present in any concentration, the

wastewater must first be discharged to the IX to remove them before the wastewater can be released to the ORF.

3.3.d. Wastewater Discharge: Off-Normal Procedure

If pollutant concentrations are above the maximums, the blended concentrations in the Savannah River will need to be estimated and compared to SCDHEC's Toxic Control Strategy document[5] before wastewater can be discharged to either the ORF or IX. In the event that the concentrations of pollutants are above levels that would compare favorably with the Toxic Control Strategy, WSRC/EPD will have to obtain permission to discharge the wastewater in an alternative manner. This alternative discharge method may entail transporting the wastewater to another wastewater treatment facility on-site, or arranging to have the wastewater disposed of off-site. WSRC/EPD will also have to be notified and must concur with any decision to release the wastewater, even if calculations indicate it meets SCDHEC's Toxic Control Strategy.

If new pollutants are found in the wastewater, their treatability in the ORF (and the IX, if appropriate) must be determined, then their blended concentrations in the Savannah River will need to be estimated and compared to the Toxic Control Strategy. The results of these evaluations will be presented to SCDHEC by WSRC/EPD as part of a request for permission to discharge new pollutants from the DETOX^{SM} demonstration unit. New pollutants cannot be discharged without prior approval from SCDHEC.

3.4 DETOX^{SM} Solution Solid Residue Characterization and Disposal

The DETOX^{SM} demonstration will generate potentially hazardous solid waste. A solid residue of about 200. - 250. kg of hydrated ferric oxide containing various heavy metals, including lead and mercury, will be produced at the end of the demonstration by evaporating hydrochloric acid from the DETOX^{SM} oxidation solution. As much as 3.7 wt% each of lead and mercury (as their chloride salts) will be present in the ferric oxide residue. The chloride salts of cerium and neodymium (as much as 1.8 wt% each) may also be present. This residue must be handled as hazardous waste until physical and chemical characterization demonstrates that it is non-hazardous.

3.4.a. Solid Residue Characterization

The DETOX^{SM} ferric oxide solid residue will be placed in lined drums as it exits the DETOX^{SM} unit filter. Hazardous waste management begins with the first drop of solid residue being generated (i.e., transferred from the filter to accumulation drums). The drums must be kept closed unless they are being filled or samples of the solid residue are being collected.[6] Contact Marie Berry, the TNX Environmental Coordinator, for establishing a satellite accumulation area 1 - 2 days before generating the solid residue.

The residue will need to be tested using USEPA Method 1311 Toxicity Characteristic Leaching Procedure for leachable organics, mercury, and lead to determine if it is hazardous by toxicity characteristic.[7,8] If the residue is semi-solid (i.e., the consistency of wet sludge), USEPA Method

9095 Paint Filter Liquids Test, must also be performed. If the residue fails the Paint Filter Test, its pH must be measured. The residue will be considered hazardous by corrosivity characteristic if its pH is less than or equal to 2, or greater than or equal to 12.5. The residue will be managed as hazardous waste, according to the provisions of the South Carolina Hazardous Waste Management Regulations,[9] until analytical data prove that it is non-hazardous.

3.4.b. Solid Residue Disposal

If the DETOX^{SM} solution solid residue is determined to be non-hazardous, it can be transferred to WSRC for disposal as non-hazardous solid waste.

If the DETOX^{SM} solid residue is found to be hazardous (e.g., leachable organics, lead, and/or mercury above TCLP limits[8]), WSRC will arrange for off-site disposal as hazardous waste (Delphi will be responsible for cost of disposal and transportation). A letter from the disposal company (or other entity) will be obtained in advance stating that it agrees to accept and dispose of the DETOX^{SM} solution solid residue. Also, any or all of this residue can be shipped to Delphi's Albuquerque laboratory facility to be used in a treatability study.

3.5 Changes to Approved Operation

Any changes to the operation of the DETOX^{SM} demonstration facility, as discussed above and in the wastewater treatment unit application,[2] must be approved by SCDHEC before they can be implemented. If any operational changes need to be made during the demonstration, notify Don Robertson in Albuquerque so that the necessary documentation can be prepared for submission to WSRC and SCDHEC. Note that a reasonable lead time must be allowed for both the preparation of the documentation, and review and approval by WSRC and SCDHEC. Where possible, this lead time should be 1 - 2 months.

3.6 Reporting and Recordkeeping

3.6.a. Reporting Procedure

The person responsible for resolving day-to-day environmental questions and problems is Marie Berry, the TNX Environmental Coordinator. Marie can be reached by phone at 557-7681, or by pager at 557-PAGE, enter # 6414. If Marie is not available, contact her alternate, Nancy Turner, by phone at 725-5641, or by pager at 925-PAGE, enter 6417. Marie is the first person to be contacted at WSRC when an environmental question or problem arises. Marie, in turn, will be responsible for contacting WSRC/EPD (when needed).

If samples of the wastewater from the DETOX^{SM} unit show that pollutant concentrations are above the maximums listed in Column 4 (Projected Concentration Exiting Unit) of Exhibit 6 of the application,[2] or if the samples contain new pollutants that are not listed in Column 2 (Constituent in Unit Effluent) of Exhibit 6, Marie Berry or her designated alternate should be notified as soon as possible. Also, Don Robertson in Albuquerque should be contacted so that treatability calculations can be performed and estimates of pollutant blended concentrations in the Savannah River can be made. If Don can't be reached, contact Pat Dhooge in Albuquerque.

3.6.b. Records to be Kept in the Demonstration Unit

The following records must be maintained in the DETOX^{SM} facility as part of the operating record and be made available for review by SCDHEC and USDOE.

1. Copies of the application[2] and the associated letter of temporary approval.[1]

2. Copies of Material Safety Data Sheets (MSDSs) for all chemicals present in the DETOX^{SM} demonstration unit. Also, forward a copy of each MSDS to W. G. Wilson at TNX.

3. Records of all materials introduced into the DETOX^{SM} unit.

4. Records of the analytical methods used to analyze the wastewater from the unit prior to discharge, and all analytical results.

5. Records of the analytical methods used to characterize the DETOX^{SM} solution solid residue (USEPA Method 1311 and any other methods of waste characterization) and all analytical results.

6. Records of the method of DETOX^{SM} solution solid residue disposal (e.g., through a commercial disposal company).

7. A letter from the entity that agrees to accept DETOX^{SM} solid residue for disposal.

8. Shipping manifests and records of disposal if wastewater is disposed of off-site.

3.6.c. Records to be Sent to the TNX Environmental Coordinator

Copies of the following records must be sent to Marie Berry. Marie will be responsible for sending any copies to WSRC/EPD.

1. Environmental training records (e.g., training for satellite accumulation areas).

2. Identities and quantities of all materials introduced into the DETOX^{SM} demonstration unit.

3. Wastewater analyses performed prior to discharge, including analytical methods and all analytical results.

4. Shipping manifests and records of disposal if wastewater is disposed of off-site. Marie Berry will provide copies of these records to WSRC/EPD.

Finally, when the DETOX^{SM} ferric oxide solid residue is generated, Marie Berry must be notified:

a. 1 - 2 days prior to generating (i.e., drumming out) the DETOX^{SM} solid residue;
b. Of the date(s) of solid residue generation;
c. Of the amount(s) of solid residue generated;
d. Of the analytical methods used to characterize the solid residue as hazardous or non-hazardous;
e. Of the analytical results of solid residue characterization; and
f. Of the method of solid residue disposal.

4.0 References

1. Letter from M. G. Vickers, SCDHEC, to W. L. Payne, WSRC/EPD, dated June 28, 1996.

2. *DETOX^{SM} Wet Oxidation Waste Treatment Technology Demonstration in the TNX Operations Area, Savannah River Site* and cover letter from D. T. Robertson, DRI, to W. L. Payne, WSRC/EPD, dated January 8, 1996.

3. 1976 South Carolina Code of Laws, Title 48, Chapter 1, Section 48-1-90(a).

4. a. 40 CFR 122; b. SCDHEC Regulation 61-9, *NPDES Permits*; c. See also Section 307(b) of the Clean Water Act (33 USC 1317).

5. a. SCDHEC *Toxic Control Strategy for Wastewater Discharges*, Oct. 1990; b. See also SCDHEC Regulation 61-68, *Water Quality Classifications and Standards.*

6. 40 CFR 264.173(a)

7. 40 CFR 262.11.

8. 40 CFR 261 Subpart C.

9. 1976 South Carolina Code of Laws, Title 48, Section 48-1-100; *ibid.*, Title 44, Section 44-56-30.

WESTINGHOUSE SAVANNAH RIVER COMPANY

INTER-OFFICE MEMORANDUM

10/11/95 SRT-AEC-95-1180

TO: Mark Fachada, SWER, 705-3C

FROM: Nancy L. Turner NLT
773-A

NOTICE OF NEPA APPROVAL (NONA) (U)

EEC No.: TC - T - 95 - 034 Rev. No.: 0
Title: DETOX Wet Oxidation Waste Treatment Technology Demonstration

It has been determined that no further National Environmental Policy Act (NEPA) documentation is required for the scope of work identified in the subject Environmental Evaluation Checklist (EEC). The proposed action is documented as being:

- ☒ Covered under sitewide Categorical Exclusion (CX): B3.10
- ☐ Covered by DOE approved CX:
- ☐ Covered by previous NEPA documentation:
- ☐ NEPA is not required as all prescreens are "NO"

The requirements of NEPA for the proposed activity have been satisfied and you are no longer restricted by NEPA to proceed with this action. Please note, however, that this concurrence relates to NEPA only.
All other applicable environmental, safety, and management activities must be met for activities related to this action.

File this determination and the original Environmental Evaluation Checklist with the proposed project or activity file.

If you determine that actions or modifications outside of the originally approved scope of work must be performed, these changes must be submitted for evaluation to insure continued compliance with the National Environmental Policy Act.

Attached: Signed EEC

cc: AEC File, 773-43A

Fax Transmittal

No. of Pages 5

To D. ROBERTSON	From FACHADA
Dept/Agency DELPHI	Phone No. WSRC
505 Fax No. 293-3183	Fax No.

Warning: Many Fax machines produce copies on thermal paper. The image produced is highly unstable and will deteriorate significantly in a few years. It should be copied on a plain paper copier prior to filing as a record.

Environmental Evaluation Checklist (EEC)

NEPA/Environmental Permits

EEC No. TC-T-95-034
Rev No. 0

Instructions (Please print or type both sides of the checklist)

- Submit one copy of the completed Checklist with supplemental information to the Department NEPA Coordinator (DNC) and the department's Environmental Coordinator (EC). The DNC will distribute to the Site NEPA Coordinator (if applicable) for NEPA level determination and the EC will distribute the completed checklist to the EPD Permits Coordinator for evaluation and approval and send a copy to SWMD Waste Forecast Coordinator.

Activity Title and Project No. (If any) DETOX Wet Oxidation Waste Treatment Technology Demonstration	Date 9/6/95
Checklist Preparer (Name, Organization, Location) Mark Fachada, SWER, 705-3C	Phone No. 7-6323
Approved By (Manager) (Name, Organization, Location) G.L. Hohmann, SWER, 705-3C	
Manager's Signature For CLH	Date 10/12/95

Proposed Activity Start Date	Proposed Activity End Date	Estimated Cost	Activity Location
5/1/96	3/1/97	$968,000	TNX

Activity Description This should be a brief but thorough description of the proposed activity. Be very specific in explaining the purpose and location (a developed/non-developed area, outside/inside/adjacent to existing building no., etc.). Attach a copy of Functional Performance Requirement, conceptual scope of work, maps, charts, or other equivalent information.

Summary:

The DOE is sponsoring a technology demonstration of Delphi Research, Inc.'s patented DETOX wet oxidation waste treatment process. The process is being developed primarily to treat hazardous and mixed wastes within the DOE complex as an alternative to incineration. The results of this demonstration will be intensively studied and used to validate the technology. The demonstration will be located at 673-T.

Detailed Description:

The DETOX demonstration unit consists of two transportable modules (one vertical, one horizontal) that are field connected by process piping and structural connections. The vertical module contains the reactor tank, solids feed assembly, the stack, filters, and associated equipment. The horizontal module contains the liquid feed assembly, pumps, and storage tanks. The size of the reactor is approximately 264 gallons and the waste feed rate will be 25 kg/hr. The unit is designed to be operated 24 hours a day. Operations will be periodically interrupted to collect data, make adjustments, and remove solid residues.

The DETOX technology uses wet oxidation to transform complex organic compounds into carbon dioxide and water. Organics containing chorine produce hydrogen chloride in addition to the carbon dioxide and water. Organic compounds are destroyed and metallic elements are solubilized in the oxidation solution. Liquids, sludges, and solids may be introduced to the process via siphons or hoppers; dry solids must be shredded or other wise rendered suitable for introduction into the system. The wet oxidation reaction takes place in a pressurized vessel (the primary reactor with stirrer) at a temperature of approximately 392 F and a pressure of 110 psig. The oxidation of organics is performed by ferric iron with platinum and ruthenium acting as co-catalysts. As the ferric iron oxidizes organics, it is reduced to ferrous iron. Oxygen is used to oxidize the ferrous iron, thus regeneration ferric iron, to carry on the continuous oxidation cycle.

Please see the attached document for additional supporting information.

NATIONAL ENVIRONMENTAL POLICY ACT (NEPA)
CHECKLIST

EEC No. TC-T-96-034

Rev No. 0

Pre-Screen Evaluation: *

Will the proposed activity:

- Result in a change in emissions, generation rates, or new discharges of hazardous, radioactive, petroleum substance, or other pollutants from a facility or process to the environment (air, water, land, etc.)? Attachment 2.1.1 ● yes ○ no
- Be located outside of a previously developed area and have the potential for environmental impact.? ○ yes ● no
- Involve siting, construction, modification, or D&D of facilities or processes which could potentially result in an environmental impact.? ○ yes ● no
- Potentially affect environmentally sensitive resources such as floodplain/wetlands, sites of archeological significance, threatened or endangered species and/or their habitat, special sources of water (i.e., aquifer)? ○ yes ● no
- Pose some change in the level of health and/or safety risks (e.g., result in an Unreviewed Safety Question)? ○ yes ● no
- Involve site characterization, environmental monitoring, or field research programs? ○ yes ● no

*Note: - If any unknown, call Department NEPA Coordinator (DNC) for consultation.
- If all are "No", no further NEPA action needed. Consult with DNC to verify; file with project & complete PERMITS CHECKLIST.
- If any are "Yes", complete rest of NEPA CHECKLIST & the PERMITS CHECKLIST.

Environmental Impacts Evaluation: (Note: If any are "Yes", provide specifics/supplemental information.)

Air

- Will there be a new air emission or a change in the quantity or quality of an existing air emission? Attachment 2.2.1 ● yes ○ no

Surface Water

- Will there be a liquid release to streams, swamps, wetlands, seepage basins, storm drains, process sewers, ponds, or lakes? ○ yes ● no
- Will river or stream water be utilized? ○ yes ● no

Groundwater

- Will there be a discharge to groundwater? ○ yes ● no
- Will groundwater be utilized? ○ yes ● no

Safety

- Is there a potential exposure to hazardous substances (e.g., radiological/toxic/chemical materials)? Att. 2.2.2.A ● yes ○ no
- Is there a potential for explosion or criticality? ○ yes ● no
- Does action involve transportation of hazardous materials? Attachment 2.2.2.B ● yes ○ no

Natural/Cultural Resources

- Is there a potential for impacts on wetlands, swamps, streams, river beds, ponds, set aside areas? ○ yes ● no
- Is there a potential impact on fish/wildlife resources or habitats? ○ yes ● no
- Is there a potential impact on protected species (e.g., sensitive, rare, threatened, endangered)? ○ yes ● no
- Is there a potential for impacting archeological sites? ○ yes ● no

- Does this action require a site clearance/site use permit (see WSRC Manual 1D, Procedure 3.02)? ○ yes ● no
- If a site of five acres or more is required, contact Site Land Use Coordinator.

For Department NEPA Coordinator Use Only

- Are there potential cumulative effects when combined with other actions? ○ yes ● no
- Is the proposed activity a component of a larger line item project? ○ yes ● no

(document title/number)

Department NEPA Coordinator (Name, Organization, Location)			Phone
Nancy L. Turner	SRTC & TNX	773-A	5-5641

Decision	DNC Signature	Date
■ Approved by DNC CX Applied: B3.10 ☐ NEPA is not required as all prescreens are "NO" ☐ Forwarded to Site NEPA Coordinator		9/21/95

For Site NEPA Coordinator Use Only

☐ DOE Approved CX CX Applied: ______

☐ Covered by previous NEPA Documentation? (CX, EA, EIS) ______

☐ Additional NEPA Documentation Required ☐ EA ☐ EIS (document title/number)

Site NEPA Coordinator	SNC Signature	Date
John R. Sessions		9/21/95

ENVIRONMENTAL PERMITS
CHECKLIST

EEC No. TC-T-95-034
Rev No. 0

General:

Does this activity involve any land disturbance which may potentially result in erosion or sedimentation?
(If "yes", what is the approximate disturbance?) ○ yes ● no

☐ Less than 1/2 acre ☐ 1/2 acre to 2 acres ☐ 2 to 5 acres ☐ Greater than 5 acres

Will the proposed activity install, modify, or remove an (including tie-in to) Underground Storage Tank: ○ yes ● no

Will the proposed activity consist of a Renovation or Demolition to an existing building/structure?
(Please specify): ☐ Renovation ☐ Demolition ○ yes ● no

Is asbestos containing material present? ○ yes ○ no

*If "no", inspector signature and license number required

Inspector Signature: __________ License Number: __________

Will you import or manufacture a new chemical substance? ○ yes ● no

Will the proposed activity impact a Site Evaluation Area or RCRA/CERCLA Area or an associated 200 ft Buffer Zone? ○ yes ● no

Will the proposed activity involve construction or modification, or to a facility or process where the potential exists for a radioactive emission? ○ yes ● no

Air: Attachment 3.2

Will the proposed activity impact a non-radionuclide air emission source? (answer "yes" if any of the following are "yes") ● yes ○ no

- Will the project install or modify a piece of equipment which will emit, or have the potential to emit, an air emission?
- Will the project modify (including demolition) an existing permitted facility or process, which emits an air emission?
- Will the project modify (including demolition) an existing facility or process, not already permitted by SCDHEC, which emits, or has the potential to emit an air emission?
- Will the project be a demonstration (short term or long term) of a new technology which will emit an air emission?
- Will the project install or modify a piece of equipment that is used to sample or monitor air emissions?

Air emissions include regulated criteria pollutants (i.e., particulate matter, lead, nitrogen oxides, carbon monoxide, sulfur dioxide, volatile organic compounds (VOC's), etc.) and hazardous and toxic pollutants identified in SCDHEC R.61-62.5 Standard 8 and Section 112(b) of the Clean Air Act.

Examples of typical permitted equipment or process air emission sources include, but are not limited to the following:

- coal or fuel oil fired boilers
- diesel generators
- diesel powered equipment
- process feed chemical storage tanks
- fuel oil storage tanks
- waste combustion incinerators
- paint booths
- lead melters
- air strippers, etc.
- degreasing operations
- HVAC and chiller equipment

Groundwater:

Will the proposed activity: (See "HOW" Manual WSRC-IM-91-69 for permitting guidance)

Install a monitoring well or piezometer(s)? ○ yes ● no

Involve subsurface penetration for a hydrogeological investigation, or characterization? ○ yes ● no

Involve the injection of a fluid, gas, or air mixture into the subsurface? ○ yes ● no

Involve the extraction of a fluid or air mixture from the subsurface? ○ yes ● no

Wastewater:

Will the proposed activity install, construct, modify, or demolish: (See "HOW" Manual WSRC-IM-91-69 for permitting guidance)

A sanitary/industrial process wastewater treatment system? Attachment 3.3 ● yes ○ no

A sanitary/industrial process wastewater collection system? ● yes ○ no

A pump station(s) to transfer sanitary/industrial waste? ○ yes ● no

A septic tank/tile field system? ○ yes ● no

A stormwater management system? ○ yes ● no

Domestic Water:

Will the proposed activity install, construct, modify, or demolish:

A domestic water distribution/treatment system? ○ yes ● no

A domestic or process water well? ○ yes ● no

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Rev No. 0

Wastes:

Will the proposed activity install, construct, modify, demolish, or otherwise impact a RCRA permitted facility? ○ yes ● no

Will the proposed activity generate a mixed waste? ○ yes ● no

If yes: Does a wastestream with similar characteristics currently exist at SRS? (Consult with Facility Environmental Coordinator if assistance is needed) ☐ yes ☐ no

Will the proposed activity generate a hazardous waste? Attachment 3.4.1 ● yes ○ no

Will you be sending hazardous/mixed waste to other on-site Treatment/Storage/Disposal (TSD) facilities? ○ yes ● no

Is the TSD permitted to accept this waste? ○ yes ○ no

(If "yes", provide the following)

- name of receiving facility ________________

- source used to confirm facility can accept waste ________________

Is this activity to take place at an existing TSD (including groundwater unit, vadose zone, process sewer, Carolina Bay, secondary containment system, etc.)? ○ yes ● no

Would this activity impact an existing TSD (including changing or improving stormwater runoff/runon drainage, security, communications, electrical, etc.)? ○ yes ● no

Does this activity involve Research and Development (R&D)? Attachment 3.4.2 ● yes ○ no

(If "yes", answer the following)

- Does it involve hazardous/mixed waste? ☒ yes ☐ no
- Does it treat more than 1,000 kg of hazardous waste? ☒ yes ☐ no
- Does it involve polychlorinated biphenyls (PCBs)? ☐ yes ☒ no
- Will this activity continue for more than 30 days? ☒ yes ☐ no
- Will more than 250kg of hazardous waste be introduced into treatment in a single day? ☒ yes ☐ no
- Does it treat more than 500kg of soil, water, or debris contaminated with acute hazardous or 1kg of acute hazardous waste? ☐ yes ☒ no
- Does it involve the placement of hazardous waste on the land or open burning of hazardous waste? ☐ yes ☒ no

Waste Identification, Generation and Management:

Will the proposed activity include the purchase of lead or lead components? (If "yes", complete OSR 29-6 for each item and submit with Checklist) Attachment 3.5.1 ● yes ○ no

Will the proposed activity disturb soil, sludge or water at or near a RCRA/CERCLA Unit or Site Evaluation Area? ○ yes ● no

If "yes", were any listed wastes disposed of at this facility? (Consult with Facility Environmental Coordinator if assistance is needed) ○ yes ○ no

If "yes", please contact EPD for guidance regarding the Investigation-Derived Waste Management Plan.

Does this activity result in a new liquid and/or solid waste generation (one-time or continuous), or a change in the quantity or the characteristic of an existing waste stream? If "yes" check all that apply: ● yes ○ no

- ☐ TRU
- ☐ Mixed - Covered by LDR FFCA
- ☐ Low-Level
- ☐ High-Level
- ☐ Hazardous
- ☒ Suspect Hazardous
- ☐ Sanitary/Industrial
- ☐ Used/Waste Oil
- ☐ TSCA (PCB)
- ☒ Wastewater
- ☐ Acute Hazardous
- ☒ Other...(specify) Attachment 3.5.2 A & B

Where will waste be stored/disposed/treated? See attached documentation Attachment 3.5.3 ○ yes ○ no

Is the facility permitted to manage this waste?

If "yes", complete the following items and submit with the Checklist

- ☒ Source utilized to confirm facility is permitted to accept the waste.
- ☒ Description of generated waste.
- ☒ Dates generation is to begin/end.
- ☒ Estimate of waste generation rate for each category.
- ☒ Description of activity/process generating waste.
- ☒ Description of waste reduction principles (reducing the volume, mass, or toxicity) for this activity.

See attached documentation - EPD

Attachment 3.5.4 A & B

Has the proposed activity been evaluated for waste minimization/pollution prevention? ○ yes ● no

Additional Comments:

This is a temporary demonstration and is not a permanent facility and will be permitted as such. All hazardous materials will be removed by Delphi after completion of testing. The anticipated test perior is 10 months.

Department Environmental Coordinator N.L. Turner	**Signature** NL Turner	**Phone** 5-5641	**Address** 773-A
EPD Permit Coordinator	**Signature**	**Date**	

Appendix B – Owner's Independent Inspection Report

Industrial Inspectors Inc.
523 Loreuville Road
New Iberia, La. 70560
(318)365-3179
Report # 6
Page 1 of 2

Date 12/1/97

PO No; 2905

Inspector; Robert Eppinett
CWI# 92070311

Vendor ID; IND 409

Company; Delphi Research Inc.
701 Halnes Ave. NW
Aluquerque, NM 87102

Fabricator; Jacobs Applied
Technology
Orangebeburg,SC

Conduct audit of Jacobs Applied Technology documents for inspection of Detox unit 80.0 for Delphi Research, Inc. profect to verify that piping was fabricated to comply to applicable codes. Also review documents for section 8 vessels and D1.1 code for structure steel.

1) a. Review Delphi subcontract award DRI-95-02-0010 dated 4/12/95.
   b. Review J.A.T. Quality Assurance Manual.
2) Review Jacobs Applied Technology Welding procedures and procedure qualification records for following;
   a. WP's 3.4.1.1 Rev. 7 Par 2502a and Par1a rev. 3 Material P1 to P1 GTAW.
   b. WP's 3.4.8-1 Rev. 3 and Par 1007 2710 Material P8 to P8 GTAW.
   c. WP's 3.4.52-1 and Par 2715 Material P52 to P52 GTAW.
   The above welding procedures and procedure qualification records are approved as to section nine of ASME code.
3) Review AWS prequalified welding procedure for welding requirements for D1.1 code for structure steel welding of unit. This procedure is approved for welding the structure of unit.
4) Review the following welders qualification records and continuity records. GH-3, Lu-40, QW-53, BB-33, QX-20, IN-23, Ng-27, Kl-17, 40-16 and El-14.
   The above welders were approved for welding on piping as to code.
   a. Review welders qualification records for field work. SC-17, SC-18, MU and DB.
5) REview Delphi piping service index dated May 28, 1996 Rev. 6.
6) Review total numbers of welds made in Jacobs shop on piping.
   a. 249 made, 26 RT., 1 rejected.
   b. Total inches made 1,787.14", 157.22" RT.
7) Review other NTD reports on structural steel.
   a. VT report #13-0301 dated 7/17/97.-acceptable.
   b. VT repore #13-0304-001 dated 1/10/96.-acceptable.
8) Review other NTD reports on vessels.
   a. VT reports 13-D300-001, 17-D308-001, 13-D303-001, 17-D303-001, 17-D311-001, 17-D304-001, 17-D308-001, 17-D307-001, !8-d304-001, 17-D305-001, 17-D309-001, 17-D310-001 and 17-D311-001. All acceptable.
9) Review design calculations, drawing, MT's and x-ray readersheets for the following ASME stamped vessels.
   a. T1-2 by Astro Metallurgical Inc.

Report #6
Page 2 of 2

b. T-402 by Jacobs. NB #3001
c. V-203 by Jacobs. NB #3297
d. V-501 by Javobs. NB #3303
e. V-002 by Jacobs. NB #3300
f. V-303 by Jacobs. NB #3299
g. V-301 by Jacobs. NB #3295
h. T-401 by Jacobs. NB #3296
i, V-601 by Jacobs. NB #3298.
These vessels are approved as to ASME code.

10) Review MTR's for P1 material piping.-acceptable.
Review MTR's for P8 material piping.-acceptable.
Review MTR's and CMTR's for material piping and welding material for P51 material.-acceptable.

11) Review five nonconformance reports.
#3149, 3150, 3151, 3152 and 3153. These were corrected and accepted.

12) Review Pressure Test Log.
Pressure Test Log acceptable.

13) Audit revealed the following.
a. Carbon steel piping manufactured to categoly "D" and "Normal" service of B31.3 code. Manufactured by Jacobs.
b. Teflon Line Pipe manufactured by PSI. to ASTM-1545-95 specification. Certificate of compliance supplied by PSI.
c. Tantalum Line Pipe manufactured by Cosmos Minerals Corporation and received the following NTD. Dye penetrant test, leak test and pressure test, according to documents supplied by Cosmos.
d.Titanium manufactured by Jacobs to B31.3 code.
e.CPVC pipe fabricated by Jacobs and receive a service test.

14) Field inspection.
Unit looked good with good workmanship. lines were level and plumbing insulation very good. Pipe flanges and fitting that were visual meet requirments as to code stamping. No tractability as to pipe.

If we can answer any questions about this audit please call.

I, the undersigned, holding a valid CWI issued by AWS has conducted the audit for Delphi Research, and to the best of my knowledge and belief, the manufacture and/or the assemble has constructed and assemble this unit to the applicable sections of codes that apply to the unit. By signing this report neither the inspector nor his employer makes any warranty, expressed or implied, concerning the unit. Furthermore, neither the inspector nor his employer shall be liable in any manner for personal or property damage or loss of any kind arising from or connected with this audit.

End of Report #6
Robert L Eppinett
Robert Eppinett
CWI 92070311
Industrial Inspectors, Inc.

ROBERT L. EPPINETT
92070311

Appendix C – Explosivity Test Report

Delphi Research, Inc.
Our best ideas are going to waste℠

Experimental Determination of Safe Operating Conditions When Mixing Oxygen and Organics in the DETOX^{SM} Catalytic Chemical Oxidation Process

Donald T. Robertson and James E. Moslander
Delphi Research, Inc.
Albuquerque, NM 87102

Kevin J. Fleming, Richard V. Saxton, and William W. Tarbell
Explosive Projects and Diagnostics Department
Sandia National Laboratories
Albuquerque, NM 87185

March 26, 1997

LIMITED RIGHTS NOTICE

These data are submitted with limited rights under
Government Contract No. DE-AC21-92MC29107.

Corporate Headquarters
701 Haines Ave., N.W.
Albuquerque, New Mexico 87102
Voice:505.243.3111 Fax:505.243.3188
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1359 Silver Bluff Rd., Suite B5
Aiken, South Carolina 29803
Voice: 803.502.0025
Fax: 803.502.0015

Delphi Research, Inc.
Chemical Research & Development

97-DTR-LO103

April 3, 1997

Mr. William J. Huber
U.S. Department of Energy
Federal Energy Technology Center
P. O. Box 880
3610 Collins Ferry Road
Morgantown, WV 26507-0880

Dear Mr. Huber:

We are pleased to submit to FETC the accompanying report, *Experimental Determination of Safe Operating Conditions When Mixing Oxygen and Organics in the DETOX^{SM} Catalytic Chemical Oxidation Process*. The report summarizes an experimental study conducted jointly by the Explosive Projects and Diagnostics Department of Sandia National Laboratories and Delphi Research, Inc. as part of the DETOX^{SM} catalytic chemical oxidation waste treatment process development program. Sandia was responsible for specifying and supplying the test equipment and instrumentation (except the test chamber itself, which Delphi supplied), conducting the tests, and gathering the data. Delphi was responsible for preparing the test mixtures and analyzing the data. The conclusions drawn as a result of data analysis are Delphi's alone.

In Delphi's DETOX^{SM} process, organics are in contact with small amounts of oxygen inside the oxidation reaction vessel. The potential exists to form a flammable organic-oxygen mixture in the reaction vessel headspace, particularly if high concentrations of oxygen are attained. Delphi recognizes that it is critical to have experimental data to validate that the oxygen levels anticipated to be used under nominal operating conditions (typically 1.-2. vol% O_2) are themselves safe. Therefore, the objective of this brief study was to measure the minimum concentration of oxygen that would sustain combustion under conditions similar to those expected during waste treatment. The maximum tolerable oxygen concentration, then, lies below the minimum needed to sustain combustion.

Three organic–oxygen–steam systems were studied. Within each system, several different concentrations of organic and oxygen were tested for flammability at 200 °C and 90 psia steam pressure. The three organics tested were acetone, toluene, and hexanes.

Delphi's Conclusions:
We found that among the three ternary systems, the minimum oxygen concentration that would support combustion was 5. vol%. Therefore, we believe that oxygen concentrations in the headspace of the DETOX^{SM} waste treatment reactor of 3. vol% or less will be continuously safe for waste treatment processing. There are aspects of the data we have collected that we do not understand, but we do not believe at this time that this detracts from our conclusion that we can tolerate a maximum headspace oxygen concentration of 3. vol%.

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Experimental Determination of Safe Operating Conditions When Mixing Oxygen and Organics in the DETOX^{SM} Catalytic Chemical Oxidation Process

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March 26, 1997

Abstract

Delphi Research, Inc., Albuquerque, NM, has patented and developed the DETOX^{SM} catalytic chemical oxidation process as an alternative to incineration for the treatment of wastes containing hazardous organics. Oxygen is used to regenerate ferric iron, the primary oxidant, so that the process can be operated continuously. The maximum concentration of oxygen in the treatment reactor headspace gas must be known in order to avoid forming a flammable mixture during processing. This brief study was conducted jointly by Delphi and the Explosive Projects and Diagnostics Department of Sandia National Laboratories. Sandia was responsible for specifying and supplying the test equipment and instrumentation (except the test chamber itself, which Delphi supplied), conducting the tests, and gathering the data. Delphi was responsible for preparing the test mixtures and analyzing the data. The conclusions drawn as a result of data analysis are Delphi's alone. The objective was to measure the minimum concentration of oxygen that would sustain combustion under conditions similar to those expected during waste treatment. Three organic–oxygen–steam systems were studied. Within each system, several different concentrations of organic and oxygen were tested for flammability at 200 °C and 90 psia steam pressure. The three organics tested were acetone, toluene, and hexanes. Our results indicate that of the mixtures tested in the three ternary systems, the minimum oxygen concentration that would support combustion was 5 vol%. Therefore, we believe that oxygen concentrations in the headspace of the DETOX^{SM} waste treatment reactor of 3 vol% or less will be continuously safe for waste treatment processing. There are aspects of the data we have collected that we do not understand, but we do not believe at this time that this detracts from our conclusion that we can tolerate a maximum headspace oxygen concentration of 3 vol%.

Acknowledgments

Our thanks to Frank M. Horine for designing and building the Model FH-GMI-1 custom fireset; and to Michael J. Navarro for his expertise in assembling the vacuum lines and all the valves and tubing. Support of this research by the US Department of Energy Morgantown Federal Energy Technology Center under contract number DE-AC21-92MC27109 is gratefully acknowledged.

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INTRODUCTION

This report summarizes the results of an experimental study conducted jointly by the Explosive Projects and Diagnostics Department of Sandia National Laboratories and Delphi Research, Inc. as part of the DETOX^{SM} catalytic chemical oxidation waste treatment process development program at Delphi. Sandia was responsible for specifying and supplying the test equipment and instrumentation (except the test chamber itself, which Delphi supplied), conducting the tests, and gathering the data. Delphi was responsible for preparing the test mixtures and analyzing the data. The conclusions drawn as a result of data analysis are Delphi's alone.

DETOX^{SM} is a patented process using ferric iron to oxidize the organic constituents of hazardous and mixed wastes to carbon dioxide and water at about 200 °C and about 115 psig total reaction vessel pressure. In this process, organics are in contact with small amounts of oxygen inside the oxidation vessel. The potential exists to form a flammable organic-oxygen mixture in the reaction vessel headspace, particularly if high concentrations of oxygen are attained. The engineering design for a full-scale continuous processing demonstration unit includes operating conditions, controls, many safety features (such as flow limiters and redundant valves), and safety interlocks to prevent the attainment of high concentrations of oxygen and organic in the reaction vessel. However, Delphi recognizes that it is critical to have experimental data to validate that the oxygen levels anticipated to be used under nominal operating conditions (typically 1–2 vol% O_2) are themselves safe.

In the worst case, a detonation would occur in the reaction vessel. In order to avoid the risk of such an event and to assure continuous safety during operations, three types of information must be determined:

- Estimates of the maximum pressures which would be attained for different organic-oxygen loadings if a detonation were to occur in the reaction vessel,
- An estimate of the response of the reaction vessel to the detonation pressure pulse, and
- Measurements of the flammability limits to be expected under operating conditions. These measurements would define the safe operating limits for oxygen in the reaction vessel.

The first two types of information potentially affect the design of the DETOX^{SM} unit. The third type of information directly affects the operation of the process.

A calculational study of detonability at operating conditions,[1] initiated in July 1994, was conducted jointly by the Facilities Safety and Risk Analysis Department of Sandia National Laboratories and Delphi Research, Inc. The three primary objectives of the study were to:

- Obtain information regarding maximum expected detonation pressures as functions of organic and oxygen concentrations,
- Estimate the response of the reaction vessel to a detonation pressure pulse, and
- Obtain an estimate of the maximum safe oxygen concentration in the reaction vessel headspace.

The results of the study made clear that a detonation could occur in the reaction vessel if the oxygen concentration was high enough, and it would be prohibitively expensive to try to design a reaction vessel that could contain the detonation overpressure. Also, the results strongly indicated that safe oxygen levels should be attainable under operating conditions, and that actual measurements of flammability limits under operating conditions were necessary to validate the calculations and define as precisely as possible safe oxygen levels for the DETOX^{SM} process.

Delphi is preparing to conduct a demonstration of the DETOX^{SM} catalytic chemical oxidation waste treatment process for the US Department of Energy–Morgantown Energy Technology Center. The demonstration unit is currently being fabricated. It is imperative that the extent of the safe operating region for mixtures of organic, oxygen, and steam be defined before demonstration operations begin. The safe operating region lies outside the flammability envelope for a given organic-oxygen-steam composition. In order to ensure that the headspace composition of organic-oxygen-steam will always be outside of the flammability envelope, the minimum oxygen concentration that will sustain combustion must be determined. Ideally, this means experimentally determining the "nose" region for each organic-oxygen-steam system that might be encountered during waste treatment operations.

The "nose" region of a flammability curve is the area at low organic, low oxygen, and high steam concentrations where the curve doubles back on itself forming the impression of a "nose". The tip of the nose is the mixture with the minimum concentration of oxygen and organic that sustains combustion. Organic-oxygen-steam compositions with oxygen concentrations less than that at the nose tip are continuously safe (i.e., non-flammable).

Funding for this study was limited, hence we did not explicitly measure upper and lower flammability limits for mixtures of organic, oxygen, and steam. Consequently, the entire flammability envelope for each mixture system was not determined. Instead, we focused on attempting to model the conditions that might be encountered in the vapor space of the DETOX^{SM} demonstration reaction vessel by trying to ignite various mixtures of organic and oxygen at constant steam pressure. The objective was to define the safe operating region for the process by determining the minimum oxygen concentration that would sustain combustion for three different organic compounds. The maximum safe oxygen concentration would lie just below this minimum. The three organics were chosen based on knowledge of the process chemistry and the waste types most likely to be treated by the DETOX^{SM} process.

This report presents the results of our efforts to determine the nose regions for mixtures of acetone-oxygen-steam, toluene-oxygen-steam, and hexanes-oxygen-steam at 200 °C and constant steam pressure. Our results indicate that of the mixtures tested in the three ternary systems, the minimum oxygen concentration that would support combustion was 5 vol%. Therefore, we believe that oxygen concentrations in the headspace of the DETOX^{SM} demonstration reaction vessel of 3 vol% or less will be continuously safe for waste treatment processing. There are aspects of the data we have collected that we do not understand, but we do not believe at this time that this detracts from our conclusion that we can tolerate a maximum headspace oxygen concentration of 3 vol%.

METHODOLOGY

Summary Experimental Description

The purpose of this series of experiments was to determine the minimum oxygen concentration necessary to sustain the combustion of various mixtures of organic compounds, steam, and oxygen at a temperature of 200 °C with static pressures greater than one atmosphere. In a typical experiment, a steel test chamber (Figure 1) was preheated to 200 °C, then evacuated using a vacuum pump to a pressure of less than one Torr. Evacuation of the chamber was done to ensure that volatile contaminants and unwanted gases (e.g., air) were removed. The chamber was pressurized with pure oxygen and the pressure measured by a static pressure transducer. Then, a mixture of organics and steam contained in a heated sample cylinder (heat tape not shown in Figure 1) was injected into the chamber. Since the sample cylinder was also heated to at least 200 °C, the organic-steam mixture was in a gaseous state[2] and was at a pressure which was greater than that of the chamber. The total chamber pressure was measured (by the static pressure transducer), then the chamber was sealed to make it ready for gas mixture ignition.

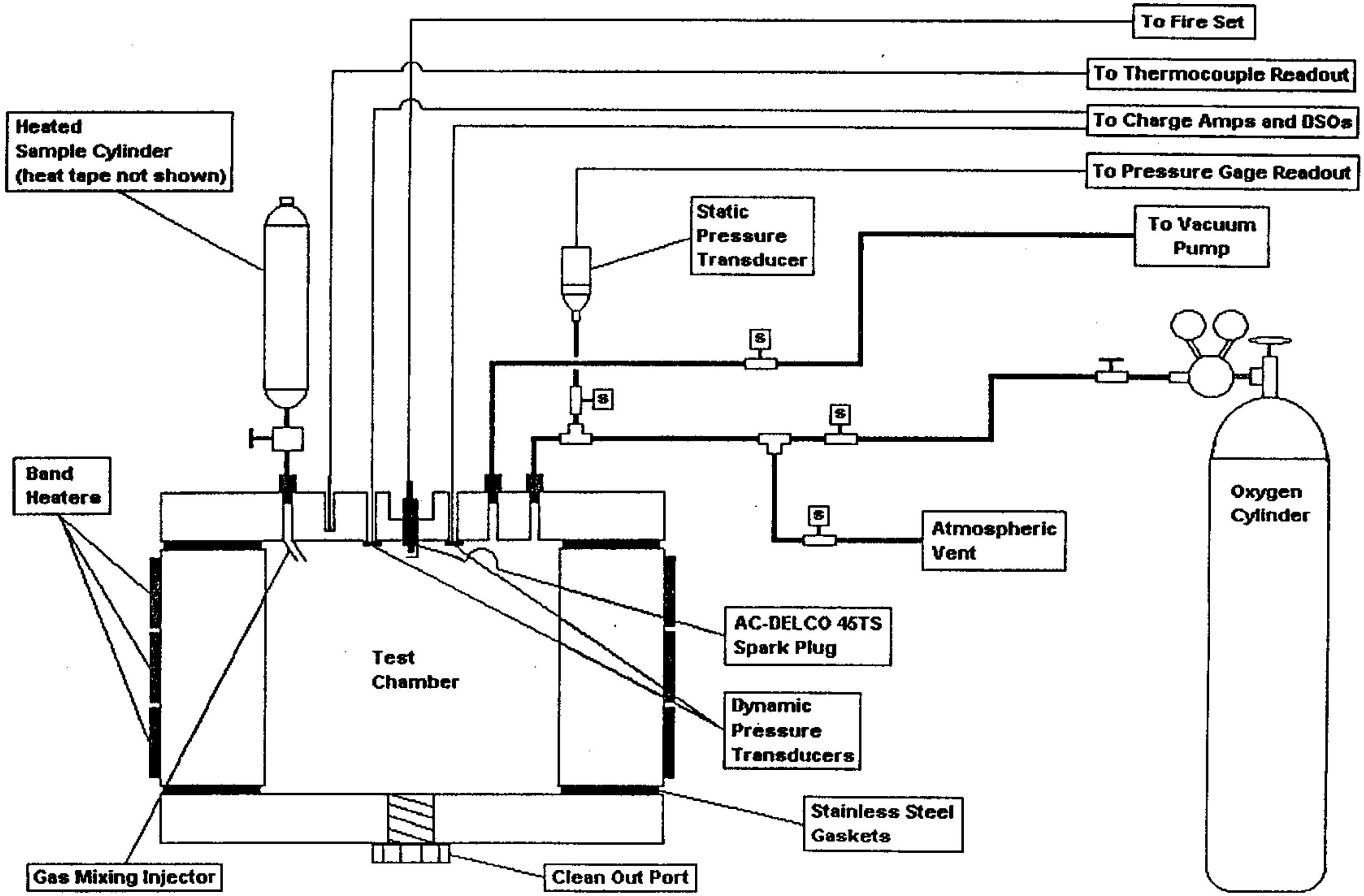
The sample cylinder inlet port in the top of the test chamber was designed to generate turbulent flow which would rapidly mix the organic, oxygen, and steam. As seen in Figure 1, the inlet for the organic-steam mixture is angled. This gas mixing injector forces the organic-steam mixture to flow tangentially to the test chamber walls as it is injected, thus facilitating gas mixing.

A spark plug connected to a 50,000 volt power supply provided the ignition source for each gas mixture. Dynamic pressure transducers were used to measure very rapid (transient) increases in pressure due to ignition. Both high and medium pressure transducers measured the pressure profile of the event. After ignition, the equilibrium chamber pressure was again measured using the static transducer. The use of both types of transducers allowed us to measure initial and final equilibrium chamber pressures (slow pressurization), as well as the much higher pressure pulses associated with propagation of the combustion front (rapid pressurization). In succeeding tests, the organic and oxygen concentrations were varied, at constant steam pressure, to determine flammable and non-flammable compositions. After each test, the clean out bolt was removed from the bottom of the chamber and the inside of the chamber was swept with pressurized air to remove any loose soot deposits on the chamber walls. The detailed test procedure is given in the Appendix.

Description of Organic-Oxygen-Steam Mixtures

In a typical sample cylinder charging procedure, a clean, dry sample cylinder with the pipe plug removed and the valve open, was first thoroughly flushed with nitrogen. After flushing, the valve was closed and the cylinder was immediately charged with liquid organic and liquid water. Then, it was sealed by replacing the pipe plug. The liquids were introduced volumetrically using two different, clean graduated pipets. This gave a mixture of water and organic that was essentially blanketed with nitrogen at 1 atm pressure. No correction was made for the presumed small amount of air that may have replaced some nitrogen during the charging procedure. Nitrogen blanketing was performed for safety reasons and to prevent any autoxidation of the organic prior

Figure 1. Gas Mixture Ignition Test System Schematic



Drawing not to scale

to injection into the test chamber. At this point, the sealed cylinder was ready to be wrapped with heating tape and mounted on the chamber.

Three organic compounds were used in testing: acetone, toluene, and hexanes (a mixture of structural isomers). Acetone was chosen because it has high volatility and flammability, and is a by-product of the DETOX^{SM} process (particularly when treating hydrocarbon oils). Thus, acetone is almost always found in the reaction vessel vapor space during operation. Toluene was chosen because it was intermediate in composition between benzene and xylenes. The US Department of Energy (USDOE) complex has many waste streams that contain all three of these aromatic hydrocarbons; toluene and xylene in particular are found in spent scintillation fluids. A mixture of hexane isomers was chosen because hexane has a high heat of combustion, high volatility, and is flammable at relatively low concentrations (1.2–7.4 vol% in air). As such, it was considered to be a worst-case compound. Also, it is representative of many non-chlorinated spent solvent waste streams in the USDOE complex, and DETOX^{SM} processing of hydrocarbon oils liberates smaller alkanes, including hexane, which then undergo destruction themselves.

The amount of oxygen was measured by its static pressure in the test chamber. Since oxygen was introduced at near ambient temperature, it was allowed to remain in the heated test chamber for a few minutes to ensure that it was heated to the run temperature. Next, the heated mixture of organic and steam was injected as quickly as possible to maximize the turbulent mixing of the three gases in the chamber. The pressure of gases in the sample cylinder prior to injection was never measured, but the cylinder temperature was constantly monitored. There was always a period near 200 °C when the rate of sample cylinder heating increased. This behavior was particularly pronounced for toluene. We interpreted this behavior as signaling that the liquid contents had vaporized to the maximum extent possible and the demand for heat to accomplish the phase change from liquid to gas had decreased.

We allowed the cylinder to remain idle at slightly above 200 °C (about 210 °C–215 °C) for a few minutes before injecting its contents into the test chamber. We felt that since all of the organics had boiling points below 200 °C, this would ensure that the organics were vaporized to the maximum extent possible. The adapter that connected the sample cylinder to the test chamber (i.e., the organic-steam injection fitting) was heated to above 100 °C to prevent condensation of organic and steam during injection. The valve attached to the cylinder was heated to about 200°C for the same reason. The concentrations of organic, oxygen, and steam for the acetone, toluene, and hexanes systems can be found in Tables 1, 2, and 3, respectively.

Calculation of the Concentrations of Organic, Oxygen, and Steam

In formulating the strategy for adding gases to the test chamber, we felt that introducing oxygen to the evacuated chamber before adding the organic-steam mixture was the best approach. This would give the most accurate measure of oxygen concentration. We also felt that since the organic-steam mixture would be injected rapidly into the chamber through a relatively small opening, backmixing of oxygen into the sample cylinder would be negligible. Hence, oxygen would be added to the evacuated chamber, then the organic-steam mixture would be injected until the pressure in the chamber leveled off (as measured by the static pressure transducer connected to the chamber). At this point, the valve to the sample cylinder would be immediately closed. This approach necessitated accounting for the residual organic-steam mixture that would be left in the sample cylinder.

We wished to scale model the conditions that are expected in the headspace of the full-scale DETOX^{SM} waste treatment reactor. In this environment, the steam pressure is constant at 90 psia. So, the calculation of the initial conditions of volume fraction of organic, oxygen, and steam begins with forcing the steam pressure in the test chamber to be constant at 90 psia for each test. This also forces the number of moles of steam in the chamber (and, hence, liquid water in the cylinder) to be constant. Next, a composition was chosen, for example, 5 vol% organic, 10 vol% oxygen, and 85 vol% steam. This fixes the ratio of organic to steam; in this case the ratio is 0.05:0.85 or 1:17. Multiplying the ratio of organic to steam by the number of moles of steam (which is fixed) gives the number of moles of organic in the test chamber.

The calculation above gives the number of moles of organic and steam in the test chamber. The extra amount of organic and steam (water) needed to account for the residual amounts that would be left in the sample cylinder after injection were calculated and added to the amounts calculated to be in the test chamber. For example, the total volume was found by adding the cylinder volume and the chamber volume (i.e., 0.5 L + 2.78 L, respectively). The fractional volume due to the cylinder is just (0.5 L/3.28 L). The amount of organic in the chamber was then divided by (1-(0.5/3.28)) to give the total number of moles of organic that would have to be charged to the sample cylinder to account for the amount needed in the chamber plus the residual amount that would be left in the cylinder after injection of the organic-steam mixture into the test chamber. Knowing the molecular weight and density of the organic gives the amount of liquid organic that would have to be charged to the sample cylinder. The extra amount of water necessary was also calculated.

The amount of oxygen needed in the chamber was found by taking the ratio of oxygen to organic, 0.10/0.05 in the example, and multiplying it by the number of moles of organic in the test chamber. Then, using the Ideal Gas Law the number of moles of oxygen gave the pressure that had to be introduced to the test chamber. The oxygen pressure inside the chamber was measured using the static pressure transducer connected to the chamber.

Since we assumed ideality and the chamber volume remained constant, the mole fraction of each component is also its volume fraction.

Example Calculation

An example calculation is given below for an example composition of 5 vol% organic, 10 vol% oxygen, and 85 vol% steam. Constants used throughout are the Gas Constant, R (0.08205 L atm/mol K), the test chamber volume (2.78 L), the sample cylinder volume (0.5 L), and the run temperature (200 °C).

The number of moles of steam in the test chamber only is fixed at 0.4385 mols (i.e., 90 psia). The number of moles of organic is given by:

(5/85)(0.4385 mol steam) = 0.0258 mol organic in test chamber only.

The number of moles of organic necessary to account for the residual amount that would be left in the sample cylinder after injection is given by:

(0.0258 mol organic) / (1 - (0.5/3.28)) = 0.0304 mol organic total.

If we assume that this organic is toluene with a molecular weight of 92.14 g/mol and a density of 0.865 g/mL, the amount needed to be charged to the sample cylinder is:

(0.0304 mol toluene)(92.14 g/mol)(1/0.865 g/mL) = <u>3.2 mL of toluene</u>.

In order to account for the amount of steam left in the sample cylinder after injection, the number of moles of steam in the test chamber only must be increased:

(0.4385 mol steam) / (1-(0.5/3.28)) = 0.5174 mol steam.

Therefore, the total amount of liquid water (density = 1 g/mL) that must be charged to the sample cylinder is:

(0.5174 mol steam)(18.02 g/mol water)(1/1 g/mL) = <u>9.3 mL water</u>.

The number of moles of oxygen in the test chamber is given by:

(10/5)(0.0258 mol organic in chamber) = 0.0516 mol oxygen in test chamber.

The pressure expected in the chamber at run temperature is given by the Ideal Gas Equation (PV = nRT):

[(0.0516 mol oxygen)(R)(473.2 K)] / 2.78 L = 0.72 atm, and

(0.72 atm)(14.7 psia/atm) = <u>11 psia oxygen in the test chamber</u>.

The total number of moles of all components in the test chamber is their sum: 0.5159 mol. The mole fractions are then calculated as for organic:

(0.0258 mol organic) / (0.5159 mol total) = 0.05 mol fraction organic.

Since we have assumed gas mixture ideality (i.e., 1 mol of gas = 22.4 L of gas) and the chamber volume remained constant, the mole fraction is also the volume fraction, from which the volume percentage is found:

(0.05 mol fraction organic) = (0.05 volume fraction of organic) = 5 vol% organic.

Instrumentation and Test System Description

The test chamber was designed for a maximum allowable working pressure of 900 psig (i.e., 900 psig flange configuration[3]) and fabricated from ASTM Grade A-36 carbon steel. The test chamber, shown in Figure 1, was a right-regular cylinder with a 6. in. internal diameter, a 15 in. outer diameter, and a 6 in. internal height, giving a wall thickness of 4.5 in. The flanges were 2.25 in. thick A-36 carbon steel and were sealed with Lamons 6 900 API 601 stainless steel high pressure gaskets. These gaskets have a chevron pattern that bows outward in response to an increase in pressure. Thus, the overall sealing surface area increases as pressure inside the test chamber increases. The volume of the test chamber was 2.78 L. All inside surfaces of the chamber were polished to a smooth finish to make cleaning easier and to remove surface irregularities that could potentially capture soot deposits from testing. The geometry of the test chamber was intended as a scale model of the headspace geometry of the DETOX^{SM} demonstration reaction vessel. Hence, the floor of the test chamber represents the surface of the DETOX^{SM} oxidation solution.

Each sample cylinder was a Whitey Company Type 304L stainless steel DOT-3E 1800 double-ended sample cylinder having an internal volume of 0.5 L and a pressure rating of 1800. psi. Each cylinder was sealed at one end with a pipe plug and at the other with a High Pressure Equipment Company Type 316 stainless steel two-way straight high pressure valve rated to 30,000 psi. Each valve stem was packed with GRAFOIL® graphite packing for this high temperature application. Prior to beginning the testing program, each sample cylinder, with valve and plug installed, was checked for leak-free integrity by pressurizing to 500 psig with nitrogen. Each cylinder was found to be leak-free under these conditions. This was the only time that the cylinders and valves were tested to determine if they would hold pressure.

The test chamber was heated with three Fast Heat Model BM3036 electric band style heaters controlled by two Omega Model 4001-KC controllers. The top two heaters were connected in parallel to one controller and the remaining heater to the other controller, providing a total heat capacity of about 1500 Watts at 115 VAC. This was sufficient to raise the insulated test chamber to the 200 °C operating temperature in about 5 hours. The chamber was maintained at the operating temperature during each experimental sequence to minimize both the heating cycle time per sample and any leakage due to repeated expansion and contraction of the vessel. The vessel was evacuated using a Welch Model 1402 vacuum pump.

Data instrumentation consisted of two dynamic pressure transducers, two charge amplifiers, and two digitizing oscilloscopes. The transducers were used to record the dynamic pressure rise within the test chamber. One transducer was a Kistler Model 7005 coupled to a Kistler model 5004 charge amplifier. This transducer had a maximum rating of 8,700 psi. The second transducer was a Kistler Model 607L rated at 30,000 psi, which was also connected to a Kistler Model 5004 charge amplifier, providing a duplicate measurement of pressure. Both data channels were fed to a Tektronix Model 544A Digital Storage Oscilloscope (DSO). Each signal was sampled at 20.0 μs (50 kHz) per sample for one second. A separate digitizer recorded the firing set voltage to ensure that it was operating correctly. After several reactions were noted with no trace data, the vertical sensitivity of the DSOs was increased, which resulted in pressure profiles in the range of 150–300 psi. It must be noted that these pressures were well below the 10% minimum rating of the transducers and should not be used as precision pressure profiles. However, they did show dynamic pressure changes within the test chamber.

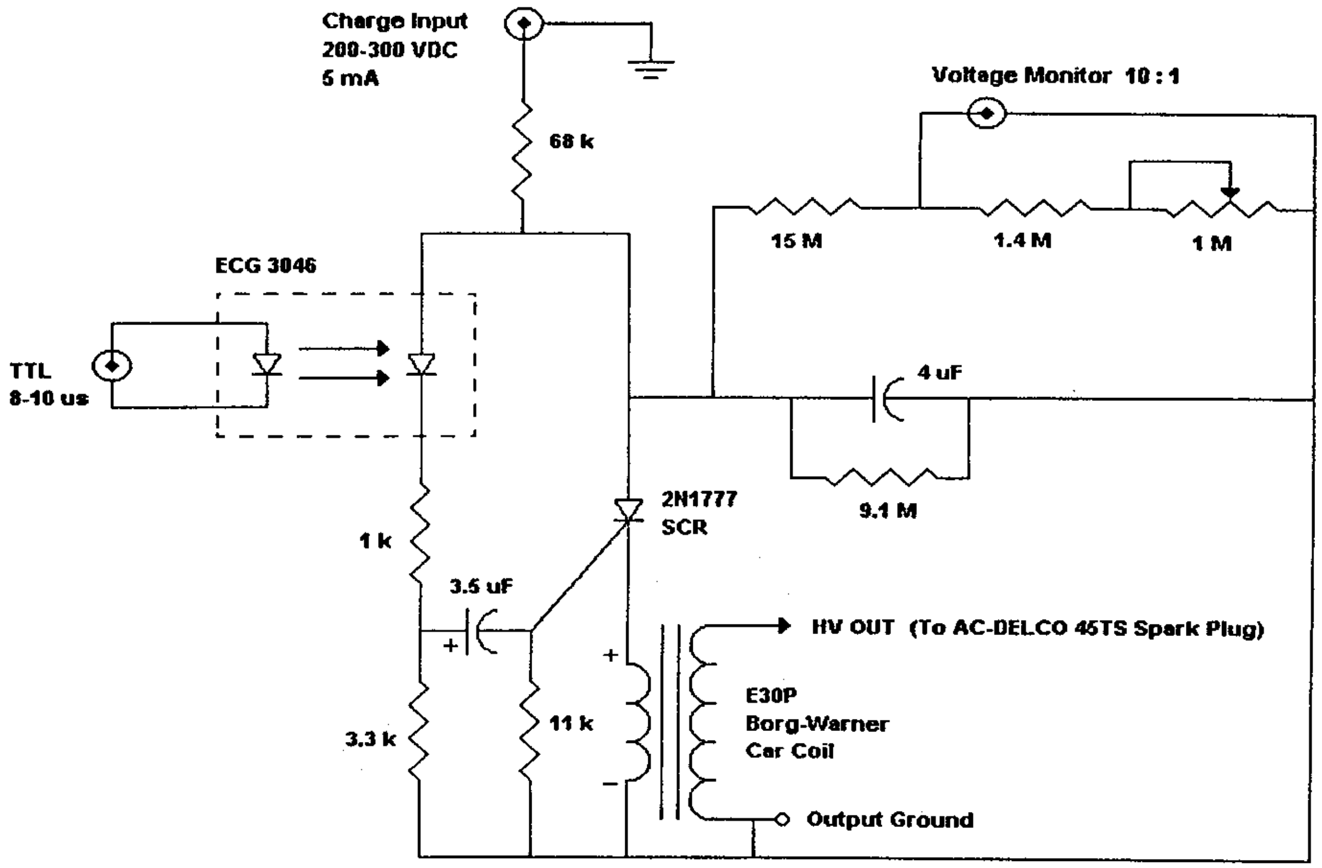
The static pressure in the test chamber was measured with an Omega Model PX605 diaphragm type transducer with a scale of 0-500 psi and an accuracy of 0.5%. This gage was used to measure both pre-test and post-test equilibrium chamber pressures. It was protected during ignition by closing a solenoid valve which isolated the gage from the test chamber. This gage was also used to measure the oxygen pressure in the test chamber prior to injection of the organic-steam mixture.

Type K thermocouples were used to measure the temperature of the test chamber, sample cylinder, and organic-steam injection fitting. The organic-steam injection fitting was maintained at greater than 100 °C to avoid any condensation of the sample during its injection into the chamber. Each sample cylinder was wrapped with a Barnstead Thermolyne Brisk Heat model BIH101060 120 V, 620 W electric heat tape prior to being installed on the chamber. Once the test was started, power was provided to the heat tape via a rheostat. The rheostat was used to control the sample cylinder temperature. After mounting to the injection fitting, the sample cylinder was covered with a phenolic tube for insulation. The cylinder temperature was monitored using a data acquisition program (WorkBenchPC™ by Strawberry Tree, Inc.) which displayed the temperature in chart format on a computer screen.

In order to enhance safety during each experiment, the test chamber was isolated and all operations were performed remotely after sample cylinder heating started. Except for the sample cylinder valve, which was opened and closed manually using a long rod and clamp that extended through a protective wall, all pressurization and evacuation operations were controlled by remote control electric valves (solenoid valves). Solenoid valves need to be kept cool in order to operate effectively. We learned early that the valve on the sample cylinder needed to be heated to about 200°C, so use of a solenoid valve at this location was impossible.

As an ignition source, a 50 kV firing set was custom built (Model FH-GMI-1; see diagram in Figure 2) to feed an AC-DELCO 45TS non-resistive spark plug that had a spark gap of 0.030 in. A differential voltage signal was generated by wrapping a one inch wide piece of copper tape around the output spark plug wire (Moroso BLUE MAX™ stainless steel) and feeding the signal to a 10X voltage probe on a DSO. This provided a means of monitoring the performance of the firing circuit as well as recording the spark signal.

Figure 2. Model FH-GMI-1 50 kV Fireset Diagram



EXPERIMENTAL RESULTS

Sixteen compositions containing acetone, oxygen, and steam; fifteen compositions containing toluene, oxygen, and steam; and thirteen compositions containing hexanes (a mixture of structural isomers), oxygen, and steam were investigated in this study. In all three ternary systems, each composition was tested in duplicate. The ultimate goal of this study was to define the maximum safe concentration of oxygen in the headspace of the DETOX^{SM} demonstration reaction vessel while conducting continuous waste treatment operations. We did this by defining experimentally the "nose" region of each ternary system. The "nose" of a flammability curve is the region at low organic, low oxygen, and high steam concentrations where the curve doubles back on itself forming the impression of a "nose". The tip of the nose is the mixture with the minimum concentration of oxygen and organic that sustains combustion. Organic-oxygen-steam compositions with oxygen concentrations less than that at the nose tip are continuously safe (i.e., non-flammable).

Extrapolating the curve back to the organic axis delineates the flammability envelope, or curve, for the system. Flammable compositions lie within the curve and non-flammable compositions lie outside the curve. A conceptual flammability curve that is typical of those obtained for mixtures of organic, air or oxygen, and inert gas (e.g., carbon dioxide, nitrogen, or steam) is presented in Figure 3. An excellent discussion of flammability curves is given by Zabetakis.[4]

Acetone-Oxygen-Steam

Figure 4 is a plot of acetone concentration as a function of steam concentration for the acetone-oxygen-steam gas mixture system. The concentration of oxygen for each composition can be found by subtracting both the acetone and steam concentrations from 100 %. This style of data presentation is common in the literature and we follow this style here. Flammable compositions are shown with squares, and non-flammable points are shown with dots. The flammable points are connected to form the impression of a "nose".

Table 1 lists the results of all of the gas mixture ignition experiments performed with acetone as the organic constituent. It can be seen that four compositions were flammable. The composition with the least amount of oxygen that was still flammable was 5 vol% acetone-5 vol% oxygen-90 vol% steam. Unfortunately, three of the four compositions were not found to be reproducible. The only reproducible composition was 10 vol% acetone-15 vol% oxygen-75 vol% steam.

We found several reproducible non-flammable compositions within the region of the flammability curve where we expected to find flammable mixtures. This is illustrated in Figure 4. This behavior is puzzling and common to all three systems studied.

Table 1 shows that for each non-flammable mixture, the final pressure was less than the initial pressure by an average of 4 %. One of the flammable compositions (experiment number 895235) also had a final pressure less than the initial pressure (decrease of 0.8 %).

Figure 3. Conceptual Flammability Curve

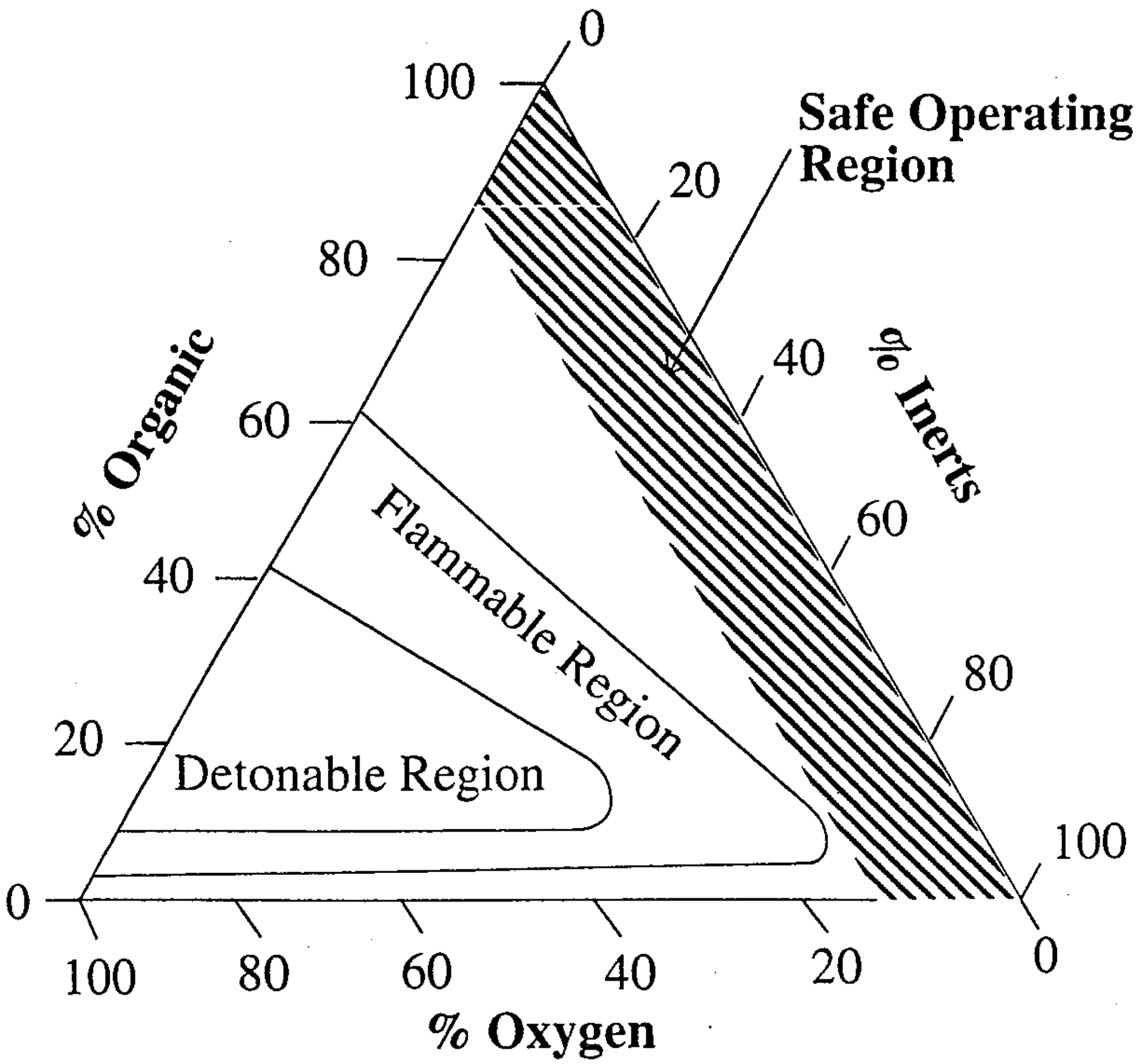


Figure 4. Acetone-Oxygen-Steam Gas Mixture Ignition Plot

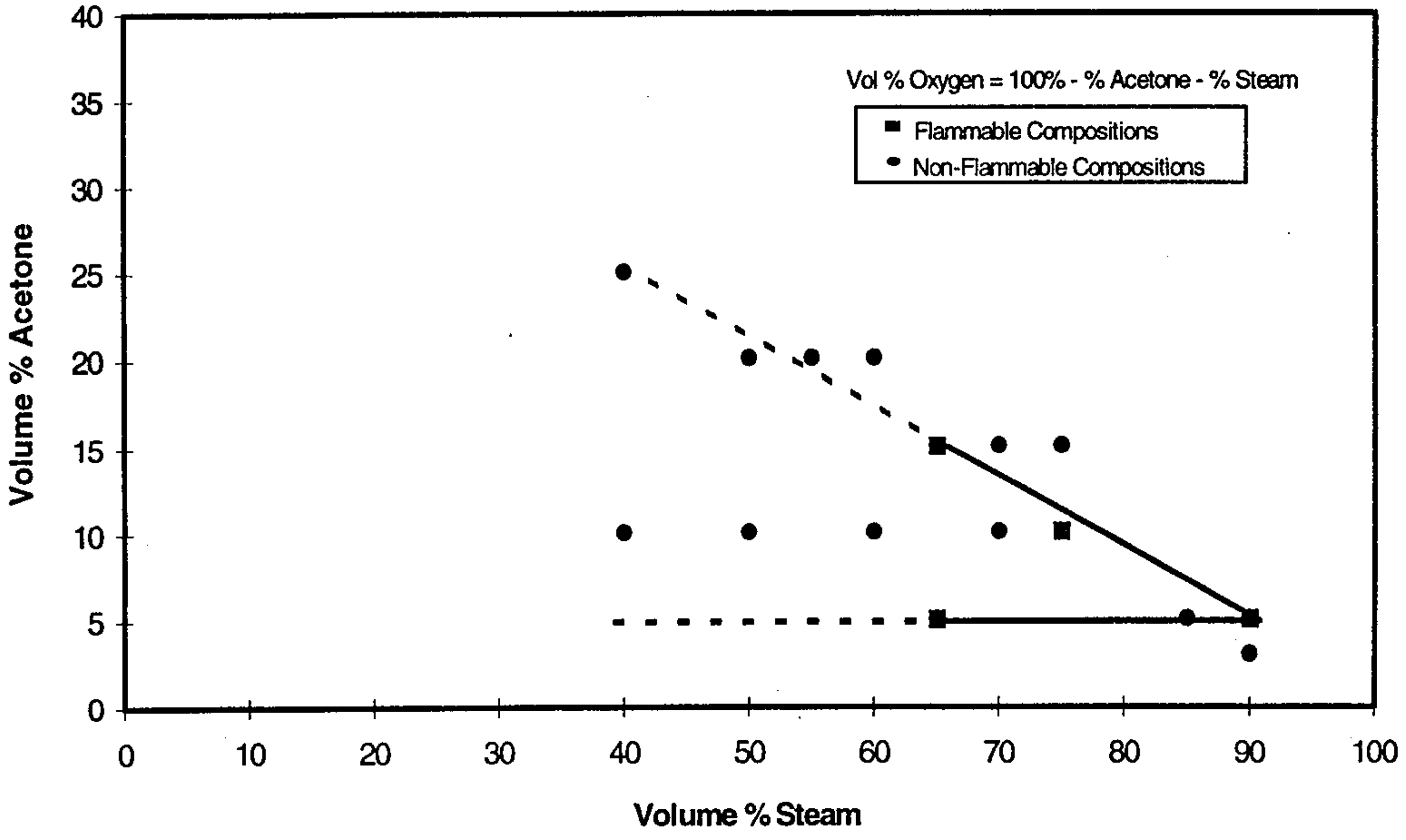


Table 1. Gas Mixture Ignition Results for the Acetone-Oxygen-Steam System

Experiment Identification Number	Calculated Acetone vol%	Calculated Oxygen vol%	Calculated Steam vol%	Initial Pressure (psia)	Final Pressure (psia)	Run Temperature (°C)	Ignition?
895238	3	7	90	84	79	201	N
895239	3	7	90	84	78	198	N
895162	5	5	90	92	90	201	N
895160	5	5	90	93	91	202	N
895150	5	5	90	80	91	201	Y
895153	5	5	90	93	90	199	N
895145	5	10	85	94	90	199	N
895156	5	10	85	98	96	200	N
895235	5	30	65	119	118	200	Y
895234	5	30	65	120	113	198	N
895154	10	15	75	110	121	201	Y
895149	10	15	75	106	115	199	Y
895151	10	20	70	120	118	201	N
895148	10	20	70	120	116	199	N
895146	10	30	60	170	163	202	N
895152	10	30	60	146	142	201	N
895241	10	40	50	155	145	200	N
895240	10	40	50	160	150	201	N
895245	10	50	40	188	181	200	N
895246	10	50	40	192	179	200	N
895173	15	10	75	101	98	202	N
895174	15	10	75	100	93	203	N
895168	15	15	70	116	111	202	N
895163	15	15	70	116	113	203	N
895158	15	20	65	126	123	202	N
895157	15	20	65	124	121	202	N
895167	15	20	65	126	193	202	Y
895170	15	20	65	118	114	202	N
895171	20	20	60	130	124	202	N
895172	20	20	60	127	123	200	N
895147	20	25	55	149	145	201	N
895155	20	25	55	146	141	201	N
895165	20	30	50	166	161	200	N
895169	20	30	50	155	149	200	N
895159	25	35	40	199	195	201	N
895161	25	35	40	196	192	201	N
895166	25	35	40	200	195	198	N

Toluene-Oxygen-Steam

Figure 5 is a plot of toluene concentration as a function of steam concentration for the toluene-oxygen-steam system. The concentration of oxygen for each composition can be found by subtracting both the toluene and steam concentrations from 100 %. Flammable compositions are shown with squares, and non-flammable points are shown with dots. Table 2 lists the results of all the experiments performed with toluene.

As with acetone, the flammable composition with the least amount of oxygen is 5 vol% toluene-5 vol% oxygen-90 vol% steam. In all, we found four flammable compositions. All were reproducible except for 5 vol% toluene-5 vol% oxygen-90 vol% steam. As with acetone, we observed reproducible non-flammable points within a region of the flammability curve that should have given flammable mixtures (Figure 5).

The final pressures for all non-flammable mixtures were less than the initial pressures by an average of 5 % (Table 2). A few of the flammable compositions either increased in pressure only slightly, or not at all. One flammable mixture (experiment number 895197) actually decreased in pressure by about 1 %.

Hexanes-Oxygen-Steam

Figure 6 is a plot of the concentration of a mixture of hexane structural isomers as a function of steam concentration for the hexanes-oxygen-steam gas mixture system. The concentration of oxygen for each composition can be found by subtracting both the hexanes and steam concentrations from 100 %. Flammable compositions are shown with squares, and non-flammable compositions are shown with dots. Table 3 lists the results of the hexanes experiments.

The composition with the least amount of oxygen that was still flammable was 5 vol% hexanes-10 vol% oxygen-85 vol% steam. In all, we found three flammable compositions. All of the flammable mixtures in Table 3 were reproducible except for 5 vol% hexanes-10 vol% oxygen-85 vol% steam. Again, we found mixtures of hexanes-oxygen-steam that were reproducibly non-flammable within a region of the flammability curve that should have given rise to flammable mixtures (Figure 6).

It can be seen in Table 3 that all of the non-flammable mixtures experienced decreases in pressure during testing. The average decrease was 7 %. Two flammable mixtures also had final pressures that were less than the initial pressures by an average of 2 %.

Figure 5. Toluene-Oxygen-Steam Gas Mixture Ignition Plot

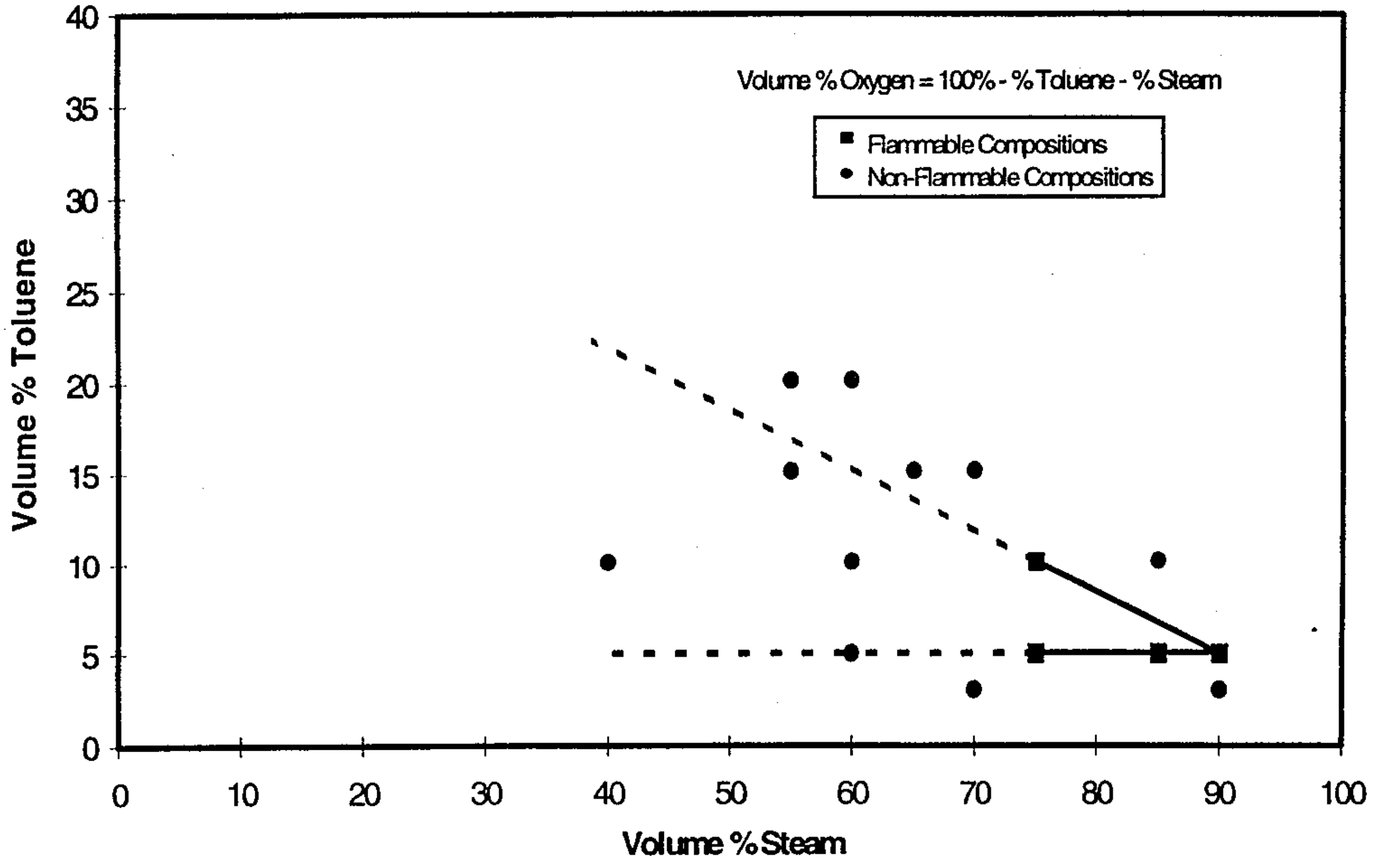


Table 2. Gas Mixture Ignition Results for the Toluene-Oxygen-Steam System

Experiment Identification Number	Calculated Toluene vol%	Calculated Oxygen vol%	Calculated Steam vol%	Initial Pressure (psia)	Final Pressure (psia)	Run Temperature (°C)	Ignition?
895202	3	7	90	91	86	201	N
895201	3	7	90	89	85	204	N
895203	3	27	70	114	107	201	N
895200	3	27	70	117	111	201	N
895183	5	5	90	83	80	204	N
895184	5	5	90	84	96	203	Y
895182	5	10	85	90	98	202	Y
895181	5	10	85	89	93	202	Y
895176	5	20	75	101	101	201	Y
895175	5	20	75	101	102	201	Y
895197	5	20	75	104	103	202	Y
895195	5	35	60	132	124	199	N
895192	5	35	60	135	128	202	N
895189	10	5	85	92	88	203	N
895196	10	5	85	93	89	204	N
895180	10	15	75	102	137	203	Y
895179	10	15	75	102	142	201	Y
895198	10	30	60	129	123	201	N
895199	10	30	60	132	125	203	N
895249	10	50	40	170	160	200	N
895250	10	50	40	183	169	199	N
895178	15	15	70	108	103	201	N
895177	15	15	70	108	104	201	N
895191	15	20	65	120	116	202	N
895190	15	20	65	121	116	202	N
895194	15	30	55	136	130	200	N
895193	15	30	55	141	134	202	N
895188	20	20	60	128	123	202	N
895187	20	20	60	127	124	202	N
895186	20	25	55	147	140	202	N
895185	20	25	55	142	137	201	N

Figure 6. Hexanes-Oxygen-Steam Gas Mixture Ignition Plot

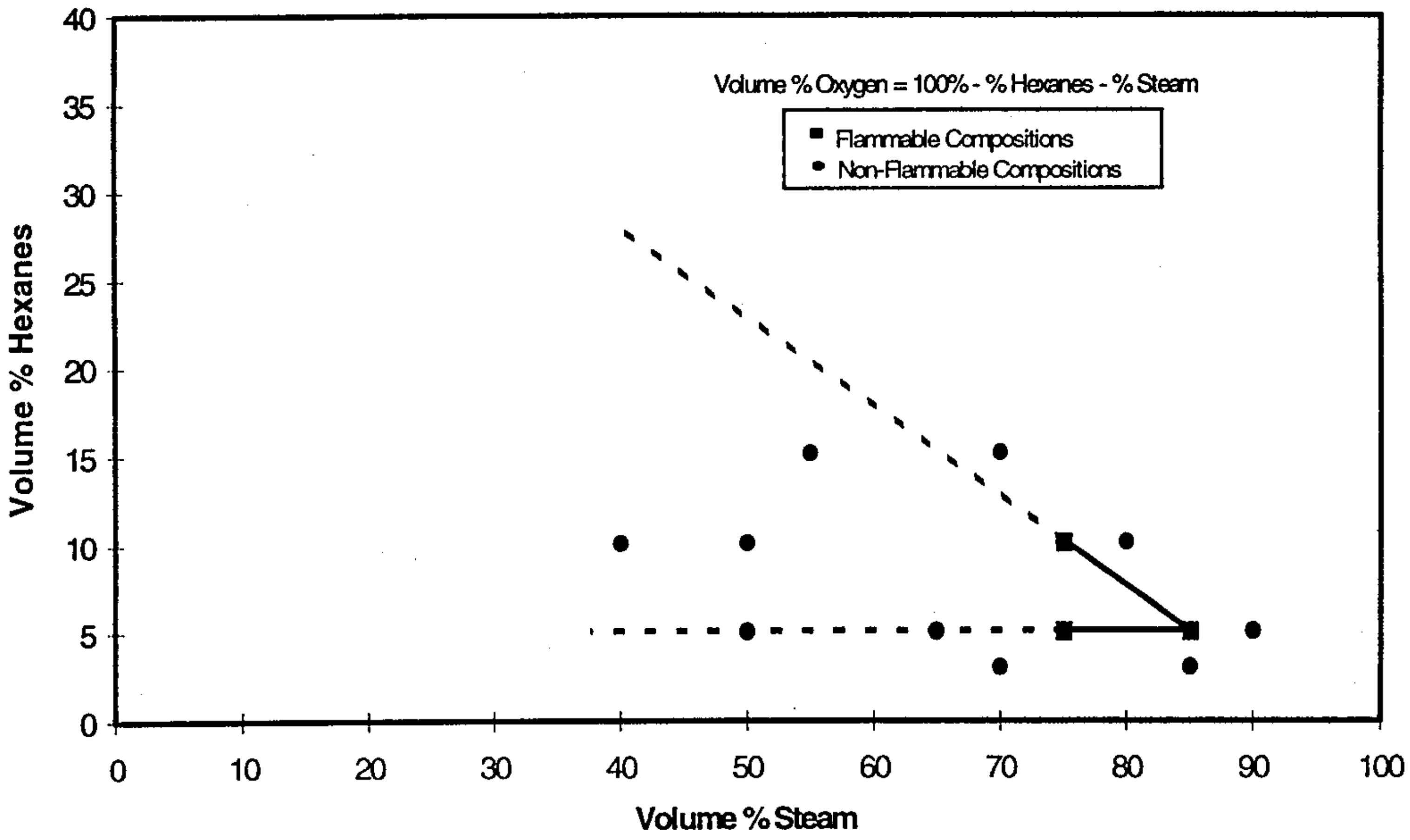


Table 3. Gas Mixture Ignition Results for the Hexanes-Oxygen-Steam System

Experiment Identification Number	Calculated Hexanes vol%	Calculated Oxygen vol%	Calculated Steam vol%	Initial Pressure (psia)	Final Pressure (psia)	Run Temperature (°C)	Ignition?
895223	3	12	85	89	83	200	N
895229	3	12	85	88	84	201	N
895231	3	27	70	109	103	200	N
895226	3	27	70	109	102	202	N
895219	5	5	90	85	80	199	N
895220	5	5	90	87	83	200	N
895221	5	10	85	93	98	201	Y
895216	5	10	85	93	84	200	N
895215	5	20	75	104	103	201	Y
895214	5	20	75	108	105	201	Y
895227	5	30	65	118	110	202	N
895228	5	30	65	117	110	200	N
895237	5	45	50	154	142	199	N
895236	5	45	50	153	144	200	N
895230	10	10	80	95	88	198	N
895222	10	10	80	92	86	201	N
895217	10	15	75	107	152	201	Y
895218	10	15	75	106	143	201	Y
895243	10	40	50	152	139	201	N
895242	10	40	50	151	140	201	N
895247	10	50	40	188	172	199	N
895248	10	50	40	187	170	201	N
895233	15	15	70	108	100	197	N
895232	15	15	70	109	102	199	N
895225	15	30	55	140	130	201	N
895224	15	30	55	143	133	200	N

DISCUSSION OF EXPERIMENTAL RESULTS

Maximum Safe Oxygen Concentration

In this series of experiments, our aim was to determine experimentally the minimum concentration of oxygen that would sustain combustion for the three systems of organic-oxygen-steam. We tried to model both the geometry of the headspace of the DETOX^{SM} catalytic chemical oxidation reaction vessel by using a right-regular cylindrical test chamber, and possible gaseous environments in the reaction vessel headspace by varying the concentration of organic, type of organic, and amount of oxygen while keeping the amount of steam constant. Furthermore, the temperature at which we conducted our experiments was the same as will be used in the demonstration reaction vessel. Our attempts to model both the physical environment of the gas mixtures and the conditions under which these gas mixtures could ignite were motivated by the knowledge that the flammability of a given gas mixture is affected by test chamber geometry and size (particularly with respect to wall quenching effects), gas mixture composition, pressure, temperature, direction of flame propagation, source of ignition, and degree of gas mixture homogeneity.[4,5]

Based on the design of the demonstration unit and our knowledge of the physical and chemical aspects of the DETOX^{SM} process, we believe that we have modeled the vapor space of the reaction vessel relatively accurately. The two aspects we could not model were the flow of gases out of the chamber and the source of ignition. Ignoring the flow of gases out of the chamber should not affect the validity of the model since under steady state operating conditions, the relative concentrations of organic, oxygen, and steam should remain constant regardless of whether the mixture is static, as in the test chamber, or flowing, as will be the case in the demonstration reaction vessel.[6] As for the source of ignition, as long as sufficient energy is present to ignite a mixture, the source of ignition should not matter. Therefore, concentrations of oxygen below the measured minimum necessary to sustain combustion should be safe for waste treatment processing on a continuous basis in the DETOX^{SM} catalytic chemical oxidation process.

For both the acetone and toluene systems, the flammable compositions with the least amount of oxygen were 5 vol% organic-5 vol% oxygen-90 vol% steam. The flammable composition with the minimum amount of oxygen in the hexanes system was 5 vol% organic-10 vol% oxygen-85 vol% steam. These compositions all proved to be non-reproducible. We believe the primary reason these compositions did not ignite reproducibly is because they lie on or near the line that separates flammable mixtures from non-flammable mixtures. We expect that these compositions would be very sensitive to reaction conditions. The limit of flammability is a borderline composition and a slight change in composition or environment can cause it to lie on either the flammable or non-flammable side of the border.[5] Small variations from experiment to experiment in the ratios of reactants, total pre-ignition pressure, the presence of soot deposits on the test chamber walls, and especially, small variations in gas mixture homogeneity could cause the mixtures to be alternately on the flammable side or the non-flammable side of the flammability curve. Therefore, we believe that these three compositions represent the "nose" tip compositions of their respective systems. The other non-reproducible flammable mixtures in the acetone system (Table 1) are also, we believe, borderline compositions.

Lower organic concentrations, i.e., 3 vol%, were tested for each system. In every case we found reproducible non-flammable behavior (see Tables 1, 2, and 3) even though higher oxygen amounts were always present. It has long been known that very low concentrations of organic are non-flammable regardless of the amount of oxidant present because below the threshold value of fuel (organic), not enough thermal energy is liberated in any given reacting layer to ignite the adjacent layer and propagate the combustion front.[5] Similarly, oxidant (oxygen) concentrations below a threshold value are insufficient to sustain fuel oxidation and thereby propagate the combustion front.[5] It is reasonable, then, to conclude that if the oxygen content of the headspace gases of the DETOX^{SM} demonstration reaction vessel are kept at or below 3 vol%, the process can be operated safely on a continuous basis.[7] Fortunately, the design operating conditions of the demonstration unit are predicated on a headspace oxygen concentration of 1–2 vol%. We feel confident that the DETOX^{SM} demonstration unit will prove to be continuously safe if operated under these conditions.

Pressure Behavior Upon Ignition

In each experiment, initial and final equilibrium chamber pressures, and any rapid increases in pressure due to ignition, were recorded. A rise in equilibrium pressure is a comparatively slow event, while a rapid rise in pressure upon ignition is a transient, pulsed event. The peak pressure ratio can be calculated from the ratio of the maximum in the transient pressure measured by the dynamic gages to the initial equilibrium chamber pressure measured by the static gage. The magnitude of this ratio gives an indication of the type of flammable behavior: deflagration versus detonation. The rise in equilibrium chamber pressure is given by the difference in initial and final pressures measured by the static gage. These quantities were calculated for each flammable event of each organic-oxygen-steam system and the results are listed in Table 4.

Initially, the rise in equilibrium pressure was of the most interest to us. We used the definition of propagation of flames as set forth in ASTM Standard E 918-83 to decide if the tested mixture was flammable.[8] This definition stated that if the equilibrium pressure rose by 7 % or more above the starting pressure (as measured by the static gage), the mixture must be taken as flammable (i.e., [final pressure/initial pressure] ≥ 1.07). Table 4 shows that, in over half the cases, the equilibrium pressure rise was greater than 7 %. There is no question that these compositions produced flammable mixtures. However, in a few cases, the equilibrium pressure rise was less than 7 %. In fact, in six cases there was a rise in equilibrium chamber pressure of 1 % or less (four of these six cases actually had slight decreases in pressure). According to the definition given by ASTM Standard E 918-83 these were not flammable events, yet we definitely recorded transient pressure pulses due to ignition. Thus, these six cases constitute what we are designating (for purposes of this study) as pressure-neutral flammable events.

In some combustion systems, such as hydrogen-air-steam, slight decreases in post-combustion pressures are typical. Equilibrium combustion chemistry calculations were performed in order to determine if the organic-oxygen-steam systems studied here should exhibit post-combustion

Table 4. Peak Pressure Ratios and Rise in Equilibrium Pressure for Flammable Compositions

Organic	Experiment Identification Number	Calculated Organic vol%	Calculated Oxygen vol%	Calculated Steam vol%	Initial Pressure (psia)	Dynamic Gage Peak Pressure (psia)	Peak Pressure Ratio	Equilibrium Pressure Rise[1] (%)
Acetone	895150	5	5	90	80	[2]	[2]	14
Acetone	895154	10	15	75	110	230	2.1	10
Acetone	895149	10	15	75	106	260	2.5	8.5
Acetone	895167	15	20	65	126	310	2.5	53
Acetone	895235	5	30	65	119	270	2.3	0
Toluene	895184	5	5	90	84	[2]	[2]	14
Toluene	895182	5	10	85	90	140	1.6	8.9
Toluene	895181	5	10	85	89	150	1.7	4.5
Toluene	895180	10	15	75	102	165	1.6	34
Toluene	895179	10	15	75	102	260	2.5	39
Toluene	895176	5	20	75	101	240	2.4	0
Toluene	895175	5	20	75	101	250	2.5	0.1
Toluene	895197	5	20	75	104	220	2.1	0
Hexanes	895221	5	10	85	93	190	2.0	5.4
Hexanes	895217	10	15	75	107	280	2.6	42
Hexanes	895218	10	15	75	106	260	2.5	35
Hexanes	895215	5	20	75	104	220	2.1	0
Hexanes	895214	5	20	75	108	240	2.2	0

[1] Difference between final and initial chamber pressures; taken from Tables 1, 2, and 3.

[2] No transient pressure pulse registered by dynamic pressure transducers.

pressures less than the pre-combustion pressures. We used the relationship for stoichiometric combustion provided by Zabetakis:[4]

$$C_nH_mO_yX_z + (n + \frac{m-z-2y}{4})\,O_2 \rightarrow n\,CO_2 + (\frac{m-z}{2})\,H_2O + z\,HX.$$

For all three systems, $z = 0$ (i.e., no halogens, X, were present). In performing the calculations, we made two assumptions:

- All gas mixtures could be treated as ideal (i.e., the Ideal Gas Law applied), and
- Each mixture burned to the maximum extent allowed by the amount of oxygen present.

Thus, in estimating pressures after combustion, we accounted for leftover organic (if any), excess oxygen (if any), carbon dioxide produced, and steam produced. In the case of steam, the pressure resulting from combustion was added to the initial pressure of steam (i.e., 90 psia). However, these calculations could not account for heat evolution.

Using the Ideal Gas Law and the stoichiometric combustion relationship, we found that in all three systems, for every organic and every composition, the pressure should have risen if the mixture had burned. Thus, we would not expect the post-combustion pressure to ever be less than the pre-combustion pressure. The most likely explanation for these pressure-neutral events is that they resulted from leaks in the test chamber (although other flammable events showed pressure increases).

On the other hand, it is possible that in each pressure-neutral event, the combined partial pressures of the residual starting gases and the product gases (carbon monoxide, carbon dioxide, and steam) were essentially equal to the combined partial pressures of the reactant gases at the start of each experiment.

Inspection of Tables 1, 2, and 3 shows that when the organic concentration is less than 5 vol%, flammable mixtures do not form. In addition, in all three gas mixture systems, the flammable compositions that gave the highest increases in equilibrium pressure had volumetric ratios of oxygen:organic of 1.3:1 to 1.5:1 (see Table 4). Thus, the compositions most likely to give large increases in equilibrium pressure following ignition have relatively significant amounts of organic present, whereas those that gave small or no increases in equilibrium pressure have relatively low concentrations of organic present (oxygen:organic volumetric ratios of 2.0:1 to 4.0:1). These findings have significance for the long-term safety of the DETOX^{SM} process, for they suggest that there are two methods to prevent the formation of flammable mixtures in the headspace of a DETOX^{SM} reaction vessel. The most effective, and the most easily instituted, is to control the amount of oxidant (oxygen) entering the system. If this is not possible, or not practical in certain circumstances, then the amount of organic entering the system can be restricted. This study was not explicitly directed toward answering the question of what is the threshold level of organic necessary to sustain combustion, but our results demonstrate that at 3. vol% organic or less, flammable mixtures do not form.

Increases in equilibrium chamber pressure resulting from combustion ranged from 0 to 53 % (see the last column in Table 4). Each flammable mixture displayed one of the following characteristics: 1) greater than 7 % rise in equilibrium pressure accompanied by a pressure pulse; 2) greater than 7 % rise in equilibrium pressure with no pressure pulse; 3) less than 7 % rise in equilibrium pressure accompanied by a pressure pulse; and 4) no increase in equilibrium pressure accompanied by a pressure pulse. These observations forced us to expand our definition of flammability. We decided that a flammable mixture had to exhibit either:

- A rise in equilibrium pressure greater than 7 %, even without an accompanying pressure pulse (the ASTM definition of flammability), or
- Transient pressurization, due to a pressure pulse, greater than 7 % of the initial pressure, with or without an accompanying increase in equilibrium pressure.

With respect to the first definition, we noted that in two cases (experiment numbers 895150 and 895184, Table 4) a rise in test chamber equilibrium pressure of greater than 7 % was recorded, but the dynamic gages failed to register a pressure pulse. We believe that in these two cases, the combustion fronts traveled too slowly for the dynamic gages to recognize them as transient pressure pulses.

Peak pressure ratios were calculated for all of the flammable compositions that registered a pressure pulse (Table 4). In all cases, the ratios were less than 3. These results, when considered along with the absolute peak pressures given in Table 4, indicate that the flammable events were all deflagrations rather than detonations. A deflagration is the subsonic propagation of a reacting flame front. Typically, deflagrations have peak pressure ratios of 8:1 or less, whereas in a detonation, the supersonic propagation of a reacting front, peak pressure ratios of 40:1 or more are typically observed.[4] The peak pressure ratios observed in these experiments averaged 2.4:1 for acetone, 2.1:1 for toluene, and 2.3:1 for hexanes. This suggests that the combustion front velocities in these three systems were comparatively slow.

The spatial relationship between the ignition source (spark plug) and the dynamic pressure transducers in the test chamber (see Figure 1) was such that when ignition occurred, the initial front of expanding gases would have grazed the surface of the transducers and no pressure increase would have registered. As the combustion front expanded in a roughly hemispherical front, it would have been reflected off the chamber walls and floor back toward the transducers. The pressure pulse experienced by the transducers, then, would have been the result of constructive interference among these reflected waves. The distance traveled by the combustion front would be roughly twice the vertical height of the chamber, or 12 in. (1 ft.). The time it would take the front to travel the 12 in. can be read from the pressure traces as the maximum in the rise time of the dynamic gages following ignition. Dividing the distance traveled by the rise time, then, gives us a rough estimate of the combustion front velocity. Obviously, we have ignored a lot of parameters in estimating this velocity, such as the thermal diffusivity, the chemical reaction rate, the heat of combustion, presence of turbulence, etc., but this velocity gives us a rough order of magnitude estimate of the speed of propagation and, hence, allows us to roughly distinguish between deflagration and detonation. Combustion front velocities were estimated for the flammable experiments listed in Table 4. These estimates are presented in Table 5.

Table 5. Estimates of Combustion Front Velocities for Flammable Compositions

Organic	Experiment Identification Number	Calculated Organic vol%	Calculated Oxygen vol%	Calculated Steam vol%	Dynamic Gage Rise Time (ms)	Estimated Combustion Front Velocity (ft/s)
Acetone	895150	5	5	90	†	†
Acetone	895154	10	15	75	75	13
Acetone	895149	10	15	75	65	15
Acetone	895167	15	20	65	125	8.0
Acetone	895235	5	30	65	70	14
Toluene	895184	5	5	90	†	†
Toluene	895182	5	10	85	180	5.6
Toluene	895181	5	10	85	135	7.4
Toluene	895180	10	15	75	320	3.1
Toluene	895179	10	15	75	335	3.0
Toluene	895176	5	20	75	40	25
Toluene	895175	5	20	75	25	40
Toluene	895197	5	20	75	35	29
Hexanes	895221	5	10	85	125	8.0
Hexanes	895217	10	15	75	260	3.8
Hexanes	895218	10	15	75	190	5.3
Hexanes	895215	5	20	75	80	13
Hexanes	895214	5	20	75	45	22

† No transient pressure pulse registered by dynamic pressure transducers.

It can be seen in Table 5 that the speed of propagation of the combustion front for all cases is quite slow, far below what could reasonably be expected to be the speed of sound in the unburned gases in the chamber (the speed of sound in air at 1 atm is 1116.5 ft/s; see Reference 9). (Deflagrations have burn speeds below the speed of sound in the unburned gases.) Since our estimated velocities are only about 1% (on average) of the speed of sound in air at 1 atm, we believe that the flammable events in the three systems of organic-oxygen-steam were comparatively slow deflagration events. This is important for the long-term safety of the DETOX^{SM} process because if a flammable event were to occur in the reaction vessel, the probability of that event resulting in a deflagration to detonation transition is very low.[10]

The pressure pulses for the different systems generally ranged from about 150 psia to 300 psia with a maximum of 310 psia (Table 4). These are not particularly excessive pressures and, in any event, they are transient in nature. In an earlier calculational study,[1] it was found that detonation pressure pulses could have magnitudes of about 30,000 psia and result in equilibrium vessel pressures of about 4200 psia, corresponding to an increase in vessel equilibrium pressure of 1767 %. We observed in this study a maximum rise in equilibrium vessel pressure of 53 %.

The peak pressures, equilibrium pressures, and pulse widths recorded during the flammable events in this study are important for assessing the likelihood of a flammable event in the demonstration unit resulting in rupture of the reaction vessel. The impulse imparted to the vessel by the pressure pulse is the integral of pressure over time.[1] The more narrow the pulse width, the less time the vessel has to respond to the pulse. Very narrow pulses do not exert pressure on a vessel for a sufficient time for the vessel to respond by deforming and thus compromising its structural integrity. Hence, even fairly large magnitude pressure pulses don't represent a concern for vessel rupture as long as the pulse duration is short (< 50–100 μs). We can surmise, then, that relatively wide pressure pulses also pose no threat to the integrity of the reaction vessel so long as the pulse magnitude is small. It is this latter situation that we believe is operative for our three gas mixture systems. Pulse widths were measured (full width at half-maximum peak height) from the dynamic pressure gage recordings. These measurements are presented in Table 6. The pulse widths are comparatively large, ranging from 30 ms to 300 ms. Fortunately, the peak pressures are small. We conclude that the impulses experienced by the test chamber in these experiments were small and their potential for damage is also small.

We recognize that extrapolating these results obtained in a small chamber under controlled laboratory conditions to a large vessel under field operating conditions can lead to large errors. Nevertheless, if we assume that the percentage rise in pressure remains fairly constant with the volumetric ratios of organic to oxygen to steam, then the final equilibrium pressure inside the DETOX^{SM} demonstration reaction vessel following a flammable event can be estimated. The nominal operating pressure of the reaction vessel will be about 115 psig. The worst case rise in equilibrium pressure after combustion was measured as 53 % (Table 4). We estimate, then, that the demonstration reaction vessel would experience a final equilibrium pressure of about 170 to 200 psig if a flammable event similar to those studied here were to occur inside the vessel. Furthermore, it is our judgment that the peak pressure of such a flammable event would be less than 1000 psig and the pulse width would be large, on the order of several hundred milliseconds. This pulse is wide enough to impart a relatively significant impulse to the reaction vessel. But,

Table 6. Pressure Pulse Widths for Flammable Compositions

Organic	Experiment Identification Number	Calculated Organic vol%	Calculated Oxygen vol%	Calculated Steam vol%	Dynamic Gage Peak Pressure (psia)	Pulse Width[1] (ms)
Acetone	895150	5	5	90	[2]	[2]
Acetone	895154	10	15	75	230	86
Acetone	895149	10	15	75	260	60
Acetone	895167	15	20	65	310	70
Acetone	895235	5	30	65	270	95
Toluene	895184	5	5	90	[2]	[2]
Toluene	895182	5	10	85	140	200
Toluene	895181	5	10	85	150	120
Toluene	895180	10	15	75	165	300
Toluene	895179	10	15	75	260	85
Toluene	895176	5	20	75	240	40
Toluene	895175	5	20	75	250	30
Toluene	895197	5	20	75	220	40
Hexanes	895221	5	10	85	190	140
Hexanes	895217	10	15	75	280	200
Hexanes	895218	10	15	75	260	150
Hexanes	895215	5	20	75	220	75
Hexanes	895214	5	20	75	240	35

[1] Full width at half maximum peak height.

[2] No transient pressure pulse registered by dynamic pressure transducers.

since the reaction vessel shell will be constructed of commercially pure titanium with a yield strength of about 18,000 psi at 316 °C (Reference 11), we believe that the vessel walls will maintain their structural integrity in response to the pressure pulse.[1]

As the pressure pulse dissipates, the equilibrium vessel pressure will rise. There are three pathways by which the reaction vessel can relieve pressure: burst disk rupture, seal failure, and controlled venting. The vessel design pressure is 300 psig, while the rupture disk is set to relieve at 225 psig. We note also that the rupture disk is contained inside a housing that is slightly offset from the head of the vessel (typical for chemical process vessels). A pressure pulse due to a flammable event would have to travel through a tube before it reached the disk. In this situation, it is unlikely that the rupture disk would blow due to the pressure pulse. Instead, it is much more likely to relieve only in response to a significant rise in equilibrium pressure.

Recognizing that our estimate of final equilibrium pressure in the vessel is only approximate, a flammable event inside the DETOX^{SM} reaction vessel could result in blowing the rupture disk. Thus, if a flammable event were to occur, our results suggest that the structural integrity of the reaction vessel would not be compromised due to the pressure pulse (i.e., the vessel walls would not be breached), and the rise in equilibrium pressure could be sufficient to blow the rupture disk. The vessel seals should not fail, though, because the rupture disk should relieve before the vessel design pressure was reached. If, on the other hand, the final equilibrium pressure were less than 225 psig, there should be enough time for the operator to initiate controlled release of the excess pressure. In any case, in our judgment, it is unlikely that a flammable event would result in catastrophic failure of the reaction vessel.

Non-Flammable Behavior of Interior Points

If we extrapolate the lines that define the "nose" regions in Figures 4, 5, and 6 back to the organic axes, we find that there are numerous tested compositions that lie within what should be flammable regions for these three systems. But, all of these points are reproducibly non-flammable. We do not understand this behavior. Without performing additional experiments and, most likely, redesigning the experimental protocol, we do not wish to speculate in depth on the possible causes of this behavior. However, we have identified five experimental variables that could have contributed to this behavior, either singly or in combination. These five variables are:

- The spark energy requirements of each system, which would likely vary with composition,
- The power density,
- Appropriateness of the ignition source (spark versus fuse wire) for the compositions we tested,
- Variation in initial gas mixture pressure, and
- Gas mixture homogeneity.

Ignition (spark) energy is a particularly important parameter since even flammable mixtures will fail to ignite if the energy of the initiation source is insufficient to be within the ignitibility limits of the composition under study.[4] The maximum electrostatic spark energy, E_{spark}, is given by the formula for electrostatic discharge:[12]

$$E_{spark} = \frac{1}{2} CV^2$$

where C is the capacitance (4 μF, see Figure 2) and V is the input voltage (300 VDC, see Figure 2). Using the above relationship, we estimated the maximum spark energy of our ignition source to be 180 mJ. This is a relatively small energy content.[4] In reality, there are energy losses due to internal resistances, the majority of which probably occur at the car coil in the fireset. Therefore, it is more likely that the actual spark energy was about 125–150 mJ (losses of 30–15%).

Many flammable mixtures can be ignited with just such a small amount of energy if the power density is relatively large.[4] We estimated the power density of our spark source from the measured lifetime of the spark, the measured spark gap of 0.030 in., and an estimated electrode diameter of 0.150 in. The spark gap and electrode diameter gave us an estimated spark volume of 8.7×10^{-3} cm^3. Using this value and the maximum spark energy, we estimated a maximum spark energy density of 20.7 J/cm^3. The average lifetime of a spark (measured from the spark signal recordings) was about 31. μs, thus the power density probably ranged from about 0.5 MW/cm^3 to a maximum of about 0.7 MW/cm^3. This is a significant power density; greater than 1 MW/cm^3 is often taken as a large power density.[4] Furthermore, the spark was essentially a compact point source,[12] so most of its energy was usable for ignition (as opposed to ignition by a diffuse electrostatic discharge which typically has a much higher energy requirement[4]). Therefore, we believe that there was sufficient energy for ignition of the various organic-oxygen-steam compositions.

Another issue is that of the appropriateness of the ignition source. It is not uncommon to observe inconsistent performance and gas mixture ignition failures when highly conductive or very high ignition energy materials are being tested using a spark ignition source.[13] It may be necessary in future testing to employ a fusible wire ignition source.[14] It is difficult to say whether the compositions we tested were high ignition energy materials, but generally, halogenated organics, such as methylene chloride and 1,1,1-trichloroethane, are considered to be high ignition energy materials.[4,5]

Another variable that may have contributed to the non-flammable behavior is variation in initial pressure. We designed this study to have a constant amount of steam in the test chamber. This was done to mimic the steam content of the DETOX^{SM} demonstration reaction vessel. The DETOX^{SM} oxidation solution has a vapor pressure of 90 psia at 200 °C and the vapor consists primarily of steam. The test compositions with higher organic and oxygen concentrations tended to have higher initial pressures; that is, the initial pressures varied with composition. It is known that flammability limits vary with temperature and pressure and that these variables need to be held constant.[4,5] We held the temperature constant (201±1 °C), but by design we allowed the pressure to vary depending on the composition of the mixture being tested. It is possible that allowing the initial pressure to vary caused the interior points of Figures 4, 5, and 6 to become non-flammable by decreasing (narrowing) the flammability limits. The effect of increasing pressure is dependent upon the individual gas mixture—the limits can either widen or narrow.[5] We do not know which effect was operative in our systems. However, we tend to discount the concept that varying the initial pressure caused the interior points to become non-flammable because those interior points lie across a broad range of compositions, many of which have high oxygen concentrations. Increased oxygen concentration almost always increases the limits of flammability.[4,5,12]

Gas mixture homogeneity was probably not a problem at low organic and oxygen concentrations since we included a feature in the test chamber that should have ensured good gas mixing, and we obtained reproducible behavior for many of the flammable compositions using the same test procedure. Under these conditions, mixing of small amounts of organic and oxygen to give a dilute solution of these components in steam should have been relatively facile. However, as the organic and oxygen concentrations increased, mixing may have become more difficult compared to the dilute system. Among the reasons for increased mixing difficulty are:

- The concentration gradient of any one component relative to another is different,
- The mass transport properties of the system are different, and
- The viscosity of the system is different.

Each of these variables would be expected to change the kinetics of mixing by increasing the amount of time necessary to achieve complete homogeneity. Hence, we conclude that in the mixtures with large amounts of organic and oxygen, complete homogeneity may not have been attained.

Determining the reasons behind the non-flammable behavior of the compositions in the interiors of Figures 4, 5, and 6 will require a careful examination of the combustion and detonation physics literature, so that a redesigned experimental system can be developed. We believe that such an effort would certainly be worthwhile, not only so that we could investigate the reasons for these gas mixture ignition failures, but also to contribute understanding to an area of combustion and chemical process safety research that has not been well investigated. There is little data in the open literature concerning the combustion behavior of organic-oxidant (air or oxygen)-inert gas mixture systems at elevated temperature and pressure, probably because performing experiments under these conditions is very difficult. Such an investigation would be relevant to increasing operating safety in many sectors of the chemical processing, oil and petroleum, and hazardous waste treatment and environmental remediation industries.

Nonetheless, we believe that having interior points in Figures 4, 5, and 6 that are non-flammable does not affect our conclusion about the maximum safe level of oxygen in the DETOX^{SM} catalytic chemical oxidation demonstration reaction vessel. Moreover, our objective was to determine the maximum safe oxygen concentration by measuring the minimum concentration of oxygen necessary to sustain combustion. We believe that we have achieved this objective. In all three systems, we found flammable compositions that describe nose regions and we demonstrated that non-flammable compositions lie outside of these regions. This behavior is consistent with a large body of data in the combustion literature.[4,5,12] We believe that in the three organic-oxygen-steam systems, the behavior of the interior points is not coupled to the behavior of the nose points and that our determination of these nose points is valid. Therefore, it is our belief that the demonstration reaction vessel can be safely operated continuously as long as the oxygen level is kept at or below 3 vol%.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

- Non-flammable organic-oxygen-steam gas mixture compositions are produced when either the oxygen concentration or the organic concentration is no more than 3 vol%. Thus, the maximum safe oxygen concentration in the DETOX^{SM} catalytic chemical oxidation process is 3 vol%.

- The largest increases in equilibrium vessel pressure upon ignition of organic-oxygen-steam gas mixtures are associated with higher concentrations of organic.

- Deflagrations in the organic-oxygen-steam systems studied here were comparatively slow. Thus, the probability of a flammable event transitioning from a deflagration to a detonation is low.

- If a deflagration were to occur in the DETOX^{SM} demonstration reaction vessel, the pressure pulse is anticipated to be small enough that the vessel walls would not rupture. The resulting rise in equilibrium pressure, though, could blow the rupture disk. If the rupture disk holds, then there should be enough time for the operator to relieve excess pressure in a controlled manner. In any case, it is unlikely that a flammable event would result in catastrophic failure of the reaction vessel.

Recommendations

- The DETOX^{SM} process must always be operated so that the oxygen concentration in the reaction vessel headspace gases is kept at or below 3 vol%.

- Provisions should be included in the DETOX^{SM} unit operating procedures to restrict the flow of organic feed into the reaction vessel, since gas mixtures with organic concentrations at or below 3 vol% are also non-flammable. This would be a backup position in cases where restricting the oxygen content is not possible or practical.

- It would be valuable to perform a redesigned, expanded combustion study to define the flammability curves for a larger number of gas mixture systems, including determining the area of the explosive region. These gas mixture systems should be based on organics likely to be found in the greatest abundance in US Department of Energy waste streams. Also, a comprehensive review of the literature should be performed to determine the most likely causes of gas mixture ignition failures and inconsistent performance so that the experimental variables that affect these problems can be controlled.

REFERENCES AND NOTES

[1] Robertson, D. T. *Summary of DETOX^{SM} Process Safety Study: Detonability at Operating Conditions*; Summary Report, Delphi Research, Inc.: Albuquerque, NM, December 2, 1994.

[2] We can be reasonably sure that at least a majority of the contents of the sample cylinder were in their gaseous state because the measured pressure of the test chamber after introduction of the organic-steam mixture always agreed to within 10 - 15 % of that calculated using the Ideal Gas Law.

[3] See for example, Megyesy, E. F. *Pressure Vessel Handbook*, 4th Ed.; Pressure Vessel Handbook Company: Tulsa, OK, 1977.

[4] Zabetakis, M. G. *Flammability Characteristics of Combustible Gases and Vapors*; U. S. Bureau of Mines Bulletin No. 627, US Department of the Interior: Washington, D. C., 1965.

[5] Coward, F.; Jones, G. W. *Limits of Flammability of Gases and Vapors*; U. S. Bureau of Mines Bulletin No. 503, US Department of the Interior: Washington, D. C., 1952.

[6] Gas flow out of the reaction vessel could slightly affect the flammability limits of a given gas mixture by inducing turbulence. Judging from the literature, turbulence would tend to make ignition more difficult by slightly raising the lower flammability limit (i.e., increasing the minimum amount of organic necessary to sustain combustion).[5] Thus, the static conditions in our model should approach worst-case conditions. Furthermore, if gas flow out of the DETOX^{SM} reaction vessel was interrupted, the vapor space environment would approach our static conditions (particularly as the time of interruption becomes long and turbulence is allowed to dissipate).

[7] This is a statement of our belief that the flammability limits of these organic-oxygen-steam mixtures will not widen appreciably as the volume of the vessel increases from that of the test chamber (2.78 L) to that of the DETOX^{SM} demonstration reaction vessel (806 L), hence it is reasonable to extrapolate these results to the larger vessel and conclude that continuously safe processing can be performed at oxygen (and organic) concentrations of 3 vol% or less. Wall quenching effects are of concern when flammability determinations are made in small vessels since they tend to narrow the measured flammability limits for a given gas mixture.[4,5] In larger vessels, wall quenching effects are weak, the flammability limits can expand to their maximum extent, and a truer measure of flammability can be made. Coward and Jones indicate that wall quenching effects are minimal in cylindrical test vessels with diameters greater than approximately 2 inches.[5] Thus, we infer that the volume of our test chamber was sufficiently large to preclude the introduction of any significant wall quenching effects into our flammability determinations, and that the flammability limits will widen only slightly (≤ 0.5 %), or not at all, in the DETOX^{SM} reaction vessel.

[8] *Standard Practice for Determining Limits of Flammability of Chemicals at Elevated Temperature and Pressure*; ASTM Standard E 918-83 (Reapproved 1988), American Society for Testing and Materials: Philadelphia, PA, 1988.

[9] *CRC Handbook of Chemistry and Physics*, 60th Ed.; Weast, C.; Astle, M. J., Eds.; CRC Press, Inc.: Boca Raton, FL, 1980; F-127.

[10] The potential for detonation will always be an issue of concern when mixtures of organic and oxygen ignite within an enclosed vessel. First of all, it is possible for a deflagration to transition to a detonation as a result of the reacting layer heating and compressing gases in an adjacent layer.[4] The resulting detonation can be especially devastating. Secondly, the detonable region of a gas mixture lies within the flammable region (see Figure 3) and the distance between the flammable curve and the edge of the explosive envelope is mixture-dependent. Thus, for some mixtures there is a narrow window of flammability and it is easy to move into the detonable region. We attempted to find a composition within the explosive envelope for toluene with little success. We did experience an explosive event with toluene (approximate composition: 10 vol% toluene-50 vol% oxygen-40 vol% steam) under conditions that were poorly defined. We recorded a minimum peak pressure of 10,600 psig with a pulse width of about 8 µs. We also determined that the fiber-type gaskets we had installed in the test chamber failed at about 950 psig, allowing the vessel contents to vent to atmosphere (we replaced them with the steel gaskets). Unfortunately, we failed to reproduce this explosive event under well-defined conditions of environment and gas mixture composition. Interestingly, the very narrow pulse width is consistent with results obtained in the earlier calculational study.[1]

[11] *Titanium Basic Design Guide*; RMI Titanium Company: Niles, OH, not dated.

[12] Kuchta, J. M. *Investigation of Fire and Explosion Accidents in the Chemical, Mining, and Fuel-related Industries - A Manual*; U. S. Bureau of Mines Bulletin No. 680, US Department of the Interior: Washington, D. C., 1985.

[13] *Standard Test Method for Concentration Limits of Flammability of Chemicals*; ASTM Standard E 681-85 (Reapproved 1991), American Society for Testing and Materials: Philadelphia, PA, 1991.

[14] An argument against employing a fusible wire ignition source in any future testing is that it is highly unlikely that wires or wire bundles will be found inside a chemical reactor operating in the field, hence, it is highly unlikely that gas mixture ignition inside such a vessel would occur by a mechanism similar to that of a fusible wire source. It is much more likely that ignition would occur by an electrostatic discharge, as would be found with a spark source. Furthermore, a fusible wire source emits volatilized metal which would eventually deposit on the walls of the test chamber. These foreign metal deposits could then catalyze unwanted side reactions of the organic compounds being tested and affect the identification of valid flammable compositions.

Appendix

Headspace Gas Flammability Test Procedure

Headspace Gas Flammability Test Procedure

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243-3111
June 27, 1995

All steps in this procedure will conform to Building 907 SOP No. SP 472093

1. Pre-Testing Notes.

Before beginning testing, make sure that the test chamber temperature is at the desired run temperature. If it is not, turn on heating bands and allow the chamber to come to run temperature; equilibrium is assumed if the test chamber temperature remains constant for 30 min. Next, place a thermocouple at the center of sample cylinder and secure it with KAPTON™ tape. Wrap cylinder with heating tape. Insulate the cylinder with a phenolic tube. Also, make sure the thermocouple on the injection stem is secured with KAPTON™ tape. Remember, the solenoid valves get hot quickly and fail closed, so don't leave the valves open any longer than necessary. Finally, it might be necessary to pressure the test chamber with oxygen in order to accurately set the oxygen regulator to deliver the desired oxygen charge. Do this at the time the test chamber is being purged following the previous run. It would be a good idea, even necessary, to clean the chamber every couple of runs by firing a 5 % toluene-20 % oxygen-75 % steam mixture to burn off any carbonaceous deposits.

2. Begin building firing sequence.
3. Secure wrapped and insulated sample cylinder to the injection port tee on top of chamber.
4. Set oxygen pressure regulator on oxygen cylinder according to composition on run sheet.
5. Check to ensure that all valves to the test chamber are closed.
6. Turn on vacuum pump.
7. Open valve to vacuum pump.
8. Begin evacuating the test chamber.
9. Clear pad, account for all personnel, secure the door.
10. Record the test chamber temperature on the run sheet.

11. Begin heating sample cylinder to run temperature.

12. Go to building firing sequence.

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13. Check that sample cylinder is at run temperature and that the contents have vaporized. Record the temperature on the run sheet.

14. Verify that the injection port tee temperature is > 100°C.

15. Assume test chamber is < 1 Torr if foreline pressure is stable at ≤100 mTorr for 5 min.

16. Shut valve to vacuum pump. Open valve to the static gage.

17. Inject oxygen into test chamber to the pressure specified on run sheet for the given organic-steam-oxygen composition (provided by Delphi).

18. Close valve to oxygen cylinder. Record the measured oxygen pressure on the run sheet.

19. Allow the oxygen to heat for 5 min. (oxygen has a fairly low thermal conductivity).

20. Open the manual valve to the sample cylinder by turning the rod until it stops.

21. Allow pressure to equilibrate.

22. Close the manual valve to the sample cylinder.

23. Turn off sample cylinder heating tape.

24. Record the equilibrium pressure in the test chamber on the run sheet.

25. Close valve to static pressure gage.

26. Go to building firing sequence and perform test.

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27. After test, record equilibrium chamber pressure on run sheet.

28. Vent the test chamber to atmospheric pressure.

29. Vent the sample cylinder to atmospheric pressure by turning rod to open manual valve.

30. Shut vent valve.

31. Evacuate the test chamber and sample cylinder for 5 min.

32. Close valve to vacuum pump.

33. Open vent valve.

34. Allow test chamber and sample cylinder to come to atmospheric pressure.

35. Close the manual valve to sample cylinder.

36. Perform the following purge sequence as many times as necessary to clean out the chamber:

    1) check that vent valve is open,
    2) remove clean out bolt from bottom of the chamber,
    3) using house air, clean out the interior of the chamber,
    4) reinstall the clean out bolt, and close the vent valve,
    5) open vacuum valve and evacuate chamber,
    6) check vacuum pump foreline pressure, it should fall to less than 100 mTorr when the chamber is clean,
    7) if chamber is not clean, close valve to vacuum pump,
    8) open vent valve,
    9) go to 5),
    10) if chamber is clean, close valve to vacuum pump,
    11) open vent valve and allow chamber to come to atmospheric pressure.

37. Remove empty sample cylinder.

38. Remove sample cylinder insulation, heating tape, and thermocouple.

39. Go to 2.

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40. After last test of the day and after the chamber has been cleaned and is at atmospheric pressure, allow the chamber to remain open to the atmosphere.

41. Allow the chamber to remain at run temperature.

42. Secure appropriate equipment.

Summary of DETOX^{SM} Process Safety Study: Detonability at Operating Conditions

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December 2, 1994

I. Introduction

This report summarizes the results of a calculational study conducted jointly by Kevin W. Boyack of the Facilities Safety and Risk Analysis division of Sandia National Laboratories and Delphi Research, Inc. as part of the DETOX^{SM} process development program at Delphi. DETOX^{SM} is a patented wet oxidation process using ferric iron to oxidize the organic constituents of hazardous and mixed wastes to carbon dioxide and water at about 200°C and 200 psig total reactor pressure. In this process, organics are in contact with small amounts of oxygen inside the oxidation reactor. The potential exists, then, to form a flammable oxygen / organic mixture in the reactor - particularly if large concentrations of oxygen are attained. The engineering design for a pilot scale continuous processing unit includes operating conditions and controls and many safety features and interlocks to prevent the attainment of high concentrations of oxygen and organic in the reactor vessel. However, Delphi recognizes that we must have data to validate that the oxygen levels anticipated under nominal operating conditions (typically 1 - 5 vol% O_2) are themselves safe.

In order to insure continuous safety at operating conditions, we required three types of data: 1) estimates of the maximum pressures (at different organic / oxygen loadings) to be expected if a detonation were to occur in the reaction vessel, 2) an estimate of the response of the reaction vessel to the detonation pressure pulse (i.e., would the detonation be containable?), and 3) measurements of the flammability limits to be expected under operating conditions - these measurements would define the safe operating limits for oxygen in the reactor.

This report will summarize the calculational estimates of the maximum pressures to be expected in detonations that take place in both a 1 gallon vessel (model of our 1 gallon laboratory reactor) and in a 250 gallon vessel (model of our pilot scale reactor). We also summarize the results of finite element (FE) calculations that simulate the responses to internal detonations that occur in these vessels. Finally, we will show that these calculational results 1) strongly indicate that safe oxygen levels are attainable under operating conditions, and 2) that actual measurements of flammability limits under operating conditions are necessary to validate the calculations and define as precisely as possible safe oxygen levels for our process.

II. Conclusions and Recommendations

A. Conclusions

1. Calculational results and extrapolations of flammability data at room temperature and atmospheric pressure strongly indicate that safe continuous operation can be achieved at low (< 10 vol%) oxygen levels.

2. These calculational results and extrapolations must be validated with experimentally measured flammability limits of mixtures of organic / oxygen / steam at 200°C and 200 psi total reactor pressure (i.e., anticipated operating conditions in a pilot scale reactor).

3. Steam generated from hot DETOX^{SM} liquid is an inherent inerting material.

4. The higher the operating temperature, the more steam that is availbale for inerting the vapor space in the reactor. Thus, higher operating temperature correlates with safer operating conditions since less volume is available for oxygen and fuel to occupy.

5. The yield strength of the material used to fabricate the reactor vessel shell is an important factor in determining the ability of the vessel to resist a detonation and maintain its structural integrity.

6. If reactor vessel integrity is compromised as a result of an internal detonation, failure will likely occur first at the edges where the reactor head and vessel wall meet.

B. Recommendations

1. Experimental measurements of the flammability limits of organic / oxygen / steam mixtures should be made at anticipated reactor operating onditions to validate the calculations and extrapolations described in this report and to define as precisely as possible safe oxygen levels for the DETOX^{SM} process.

2. A titanium α,ß-alloy (such as Ti-6Al-4V) containing 0.05 wt% palladium should be used to construct the pilot reactor vessel shell because of its large yield strength and corrosion resistance properties.

III. Methodology

A. Vessel Geometries

1. 1 gallon Laboratory Reactor

The 1 gallon lab reactor is a standard Parr Instrument Co. autoclave (vertical axisymmetric cylinder) constructed out of titanium grade 2 (commercially pure) containing a glass insert that has a diameter of 14 cm and a height of 19 cm. The bottom of the reactor head is flat. DETOX^{SM} liquid is held in the glass insert during testing. For the calculations, the cylinder was assumed to contain 1.5 L of DETOX^{SM} liquid (as in the actual reactor) and the liquid surface was assumed to behave as if it were a wall (incompressible fluid). Thus, for both the thermodynamics calculations and the finite element simulations (see descriptions below) the cylinder in which the detonations take place has a diameter of 14 cm and a height of 9.5 cm giving a (vapor space) volume of 1.5 L.

2. 250 gallon Pilot Reactor

The design of the pilot reactor calls for a cylindrical vessel with ASME dished heads having a straight-side height of 122 cm (48 in.), a diameter of 91 cm (36 in.), and an internal volume of 1000 L (264 gal.). The vessel material is titanium grade 7 (commercially pure with 0.2 wt% palladium addition). For both the thermodynamics calculations and the finite element simulations (see descriptions below), the vessel was assumed to be about half-full with DETOX^{SM} liquid and the liquid surface was assumed to behave as if it were a wall (incompressible fluid). We further assumed that the reactor head was flat (as in the 1 gallon case) and that the vapor space volume would be 473 L (125 gal.). Thus, the cylinder in which the detonations take place has a diameter of 91 cm and a height of 73 cm.

B. Gas Mixture Compositions

Benzene and cylcohexene (as a model for *n*-hexane) were investigated, both were assumed to be completely vaporized at the nominal operating conditions. The compound most frequently studied was benzene. Two different temperatures were also investigated - 150°C and 200°C - with 200°C being used most often.

Various organic loadings and O_2 mole fractions were studied to determine the dependence of detonation pressure on equivalence ratio[1]. For most of this work, steam pressure was specified at the measured vapor pressure of DETOX^{SM} liquid at the temperature of interest. For example, most of the thermodynamics calculations were run at 200°C. The vapor pressure of DETOX^{SM} liquid at that temperature is 90 psia (at 150°C it's 20 psia).

For the 1 gallon reactor, oxygen percentages varied from 30 to 76 vol%, all at 200°C. In the 250 gallon case, oxygen percentages varied from 70 to 86 vol% at 150°C and from 37 to 53 vol% at 200°C.

Gas mixture compositions were determined by fixing the mole fractions of steam and organic at the desired temperature; the residual mole fraction was then assigned to oxygen. The total pressure at the desired temperature was then calculated. In some instances, the total pressure was fixed along with the mole fractions of steam and organic before assigning the residual mole fraction to oxygen. Typically, in the 1 gallon case the total reactor pressure was 200 psig and the temperature was 200°C. In the 250 gallon case, the total pressure varied from 193 to 227 psig at 150°C and from 196 to 225 psig at 200°C

C. Calculations

1. Thermodynamics

The CET89 code[2] was used to perform the thermodynamics calculations. This code is capable of handling a variety of fuel / oxidant / inert gas mixtures in estimating peak detonation pressures. The code calculates various properties of the detonation state (known as the CJ state), such as CJ and reflected CJ pressure (the sum of these two pressures is the estimated peak detonation pressure), the detonation temperature, the detonation velocity, and the AICC (adiabatic isochoric complete combustion) pressure and temperature. The AICC pressure and temperature are the maximum pressure and temperature expected to be found at equilibrium following the detonation. In practice, the AICC pressure and temperature are not realized since the vessel in which the detonation took place loses some heat to its surroundings.

The results of the CET89 calcualtions were then used to calculate refined detonation pressure histories using the CTH code[3]. The output of the CTH code is a plot of pressure (psia) against time (µs) and gives the pressure histories to be expected at a wall and at an edge in the vessel. This code also approximates the pressure histories and these simplified histories are used in the finite element simulations (see below). The approximate pressure histories are compared to the refined pressure histories to justify their use in the simulations.

The point at which detonation initiation occurs can have an affect on the calculated detonation pressure pulse. The locus of initiation (point source) was fixed at the geometric center of the vessel at just above the surface of the DETOX^{SM} liquid (bottom of the vessel). This gave the maximum travel distance in all directions for the nascent detonation front which results in the maximum impulse at the vessel wall. We also assumed that no obstructions, such as a thermowell, a stirrer shaft, baffles, etc., were present. These obstructions will tend to attenuate the peak pressure experienced at the vessel walls[4].

2. Finite Element (structural dynamics) Simulations

Finite element (FE; PRONTO code) simulations were carried out for four conditions: one for the 1 gallon case and three for the 250 gallon case. The code used the results from the CTH calculations to simulate the dynamic responses of the two vessels to detonation. Approximate pressure histories (see above) are used to simplify the calculations and minimize computer time.

The code uses a mesh generator to approximate the vessel geometry (vertical axisymmetric cylinder) and simulate the movement (if any) of the walls and lid of the vessel as it experiences the sudden impulsive loading of an internal detonation.

For the 1 gallon case, the physical properties of titanium grade 2 were used. A three part approximation of the vessel (wall, head, and split rings) was studied. The wall thickness was asssumed to be 0.375 in., the head thickness was assumed to be 1.0 in., and the split rings were assumed to be 0.375 in. all around. The gas composition was 7.5 vol% benzene, 32.5 vol% oxygen, and 60.0 vol% steam at 200°C and 200 psig initial pressure. The peak detonation pressure was 10,400 psia with a pulse width of 22.5 µs. The final equilibrium pressure was 2,100 psia.

For the 250 gallon case, two different titanium materials (titanium grade 7 and Ti-6Al-4V alloy) and two different peak detonation pressures were studied. Titanium grade 7 has the same physical properties as Ti grade2 (but has increased corrosion resistance) while Ti-6Al-4V has three times the yield strength. . A three part approximation of the vessel (wall, head, and split rings) was studied. The wall thickness was asssumed to be 0.625 in., the head thickness was assumed to be 2.0 in., and the split rings were assumed to be 1.0 in. all around. The gas composition, temperature, and initial pressure were assumed to be essentially the same as for the 1 gallon case above. For each titanium material, peak detonation pressures of 10,400 psia (final equilibrium pressure of 2,100 psia) and 5,000 psia (final equilibrium pressure of 1000 psia) and pulse widths of 169.9 µs and 214.9 µs were studied. The pulse widths were longer to account for the larger volume of the vessel as compared to the 1 gallon case.

IV. Results and Discussion

A. 1 gallon Laboratory Reactor

1. Thermodynamics Calculations

The condition we were most interested in was the detonability of the nominal gas composition we were using in the lab reactor for process development testing. We chose benzene as the organic and performed three calculations in which the equivalence ratio was varied from 1 04 to 1.75. In addition, the reactor temperature, reactor pressure, and steam content were kept constant at 200°C, 200 psig, and 60 vol%, respectively. The results of these calculations are presented in Table 1 below.

Table 1. CET89 Results for Benzene in the 1 gallon Reactor

	Case 1	Case 2	Case 3
Initial temperature (°C)	200	200	200
Initial pressure (psia)	200	200	200
Benzene fraction (vol%)	10	7 5	5
Oxygen fraction (vol%)	30	32.5	35
Steam fraction (vol%)	60	60	60
Equivalence ratio	1.75	1 38	1.04
Peak detonation pressure (psia)	9,284	10,664	9,810
Peak detonation velocity (m/s)	2027	2154	2117
AICC pressure (psia)	1,347	1,510	1,451
AICC temperature (°C)	1,851	2,520	2,860

It can be seen that the peak detonation pressure comes at an equivalence ratio of 1.38 (Case 2). In fact, the conditions of Case 2 were routinely attained under batch operating conditions in our lab reactor. These results have prompted us to modify our lab operating conditions to significantly reduce the amount of oxygen present in the reactor during testing. We should mention that this reduction in oxygen content at elevated temperature is only possible in a tantalum-lined reactor. Titanium is unable to withstand low oxygen levels at elevated temperatures in the presence of DETOX^{SM} liquid and vapors.

We used these results to calculate the pressure history in the 1 gallon vessel using the CTH code. The resulting pressure history plot showed that the peak detonation pulse would be experienced at the vessel edge - that is, where the reactor head contacts the cylinder wall - and that the peak pressure at the wall would be about 7,635 psia. The CTH results also estimated the pulse width (the width of the peak detonation pressure pulse) to be about 35 us. This is a very narrow pulse.

The factor that determines the loading the vessel edges and walls will see is the cumulative impulse. The impulse is the integrated area over time of the peak detonation pressure pulse. Thus, very high detonation pressures do not necessarily guarantee that the vessel will fail. Large detonation pressures with narrow pulse widths may have small cumulative impulses. It is the cumulative impulse that the vessel walls and edges experience which will determine if the vessel will fail. The way to estimate the cumulative impulse and its effects on the vessel is to use these results in finite element simulations.

2. Finite Element Simulation

The FE simulation of the dynamic response of the 1 gallon vessel to a 10,400 psia detonation pressure pulse (approximation of Case 2 thermodynamics results) showed that the reactor head would become slightly disengaged from the cylinder, but not to the extent that venting of reactor contents would occur - the detonation was contained. This FE simulation confirmed the CTH prediction that the edge (where the head contacted the cylinder wall) would experience more explosive power than the wall[4]. Since the pulse width was small, the total impulse was small and the vessel didn't bulge much: the equilibrium displacement of the wall after detonation is only about 0.012 in. outward from the starting condition. However, this result in no way obviated our need and desire to modify the lab reactor operating conditions: even a containable detonation is unacceptable.

B. 250 gallon Pilot Reactor

1. Thermodynamics Calculations

In an initial investigation, results of CET89 calculations showed that the more reduced the organic fuel was, the greater the detonation pressure pulse. For example, at 150°C cylcohexene had a slightly larger detonation pressure than benzene. This is shown in Table 2. Then, we varied the concentration of cyclohexene at 150°C keeping the pressures of oxygen and steam constant (the total reactor pressure varied from 193 - 227 psia). We found that the largest detonation pressures (on the order of 30,000 psia) were achieved when the gas mixtures were relatively fuel-rich (equivalance ratio of about 2.0). We saw approximately this same behavior in the 1 gallon case.

We also found that detonation pressures were higher for benzene at lower temperature. This behavior is due to lower steam pressure at lower temperature resulting in less steam (inert gas) being present to displace benzene and oxygen. Thus, more total potential energy is present at lower reactor temperature (higher fuel and oxygen concentrations) to be released during a detonation. These results are summarized in Table 2.

Next, we intended to vary the oxygen concentration to gauge its affect on detonation pressure. In looking at the flammability limits of benzene, cyclohexene, and *n*-hexane in air at 25°C and 1 atm. pressure[5], it seemed unlikely that flammable mixtures would exist at oxygen concentrations below about 10 - 20 vol%. However, our concern was with the flammability limits at elevated temperature and pressure in the presence of an inerting material (steam).

Table 2 CET89 Results for Benzene and Cyclohexene in the 250 gal. Reactor

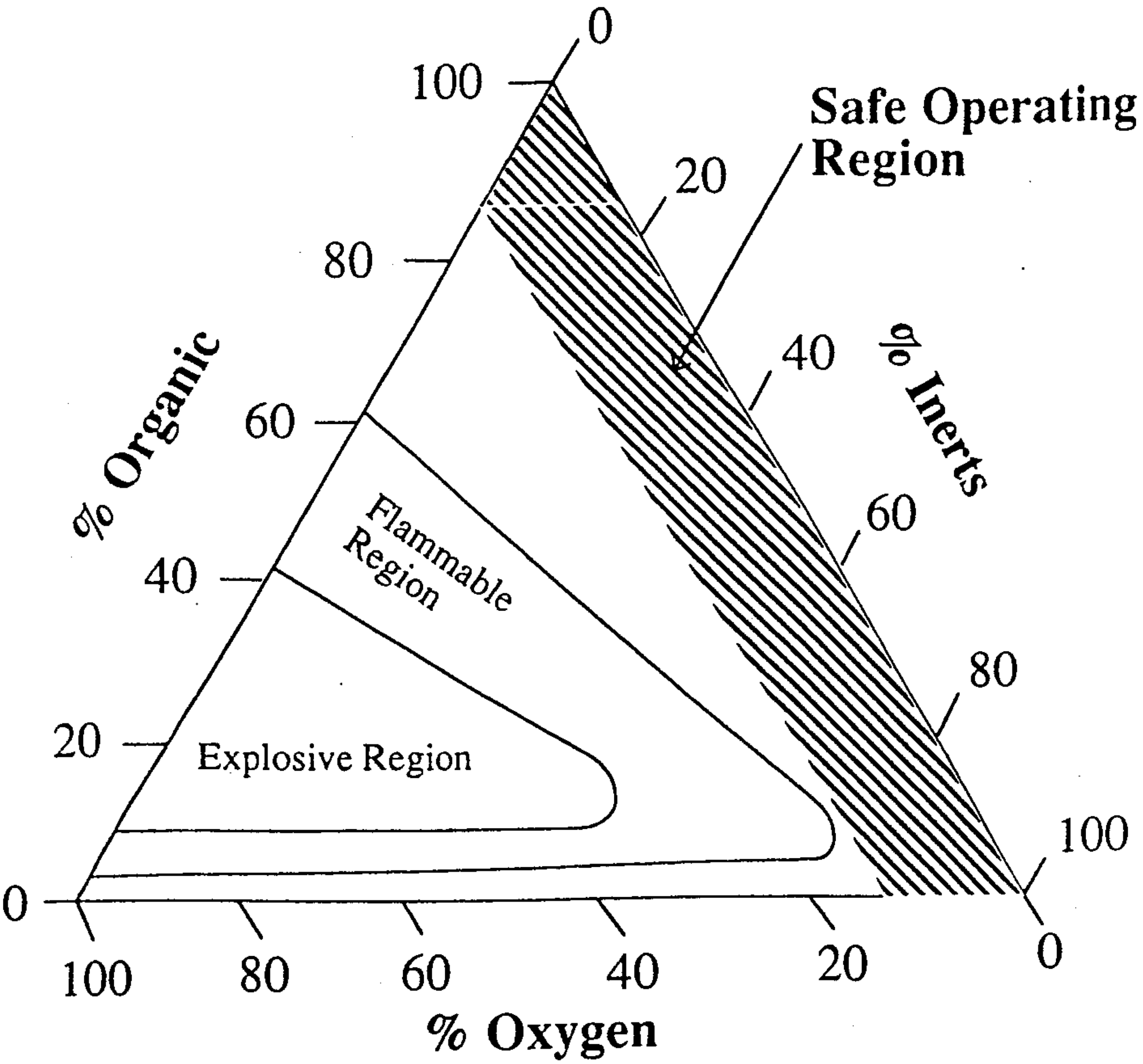
Organic	Case 1 Benzene	Case 2 Benzene	Case 3 Cyclohexene
Initial temperature (°C)	200	150	150
Initial pressure (psia)	236	244	238
Organic fraction (vol%)	13.1	22.1	16.0
Oxygen fraction (vol%)	48.8	69.7	75.6
Steam fraction (vol%)	38.2	8.2	8.4
Equivalence ratio	1.73	2.30	1.76
Peak detonation pressure (psia)	17,572	28,980	29,498
Peak detonation velocity (m/s)	2,357	2,536	2,624
AICC pressure (psia)	2,524	4,130	4,205
AICC temperature (°C)	2,948	3,160	3,642

In general, the effect of increased temperature is to widen the flammability limits: the lower limit decreases slightly and the upper limit increases, sometimes greatly[6]. Increased pressure has the same effect, and also slightly decreases the effectiveness of an inert gas[6]. Addition of inert gas tighens the flammability limits: the lower limit is raised slightly and the upper limit is decreased, sometimes considerably[6]. Also, in all cases the explosive region lies within the flammable region. This means that if we prevent a flammable mixture from forming, we've also prevented a detonable mixture from forming (but not *vice versa*). By using data from Yeaw and Shnidman[illegible], Kuchta[8], and Coward and Jones[6] an estimate was made of the flammability range for benzene in the DETOX℠ process. We estimated that at 10 vol% oxygen, 40 vol% benzene, 50 vol% steam the gas mixture is *not* detonable. Furthermore, the result of a CET89 calculation for 23 vol% benzene, 37 vol% oxygen, and 40% steam gave 10% solid carbon in the wake of the detonation - the calculation failed to converge to a solution for detonation pressure. We interpret this result to mean that this condition is too fuel-rich to be detonable. Thus, we believe that it is possible to delineate operating conditions in the pilot reactor in which it will be continuously safe to mix organic and small amounts of oxygen. We believe that as long as we keep the oxygen levels low enough (say < 10 vol%) we will not form a flammable - and therefore an explosive - mixture in our process. This is shown conceptually in Figure 1. We stress, however, that these flammability limit estimations are extrapolations of data at atmospheric pressure near 25°C and contain relatively large uncertainty in their accuracy. This makes it absolutely imperative to experimentally measure the flammability limits of mixtures of organic, oxygen, and steam at 200°C and 200 psi pressure to validate these extrapolations and define as precisely as possible safe oxygen levels for our process.

2. Finite Element Simulations

FE simulations were performed for three conditions; these conditions were described in part C of section III. above. We used the same approximate detonation pressure (10,400 psia) as for the 1 gallon reactor FE simulation but a longer pulse width to account for the larger vessel volume. The results fo this simulation were disappointing. At 0.6 μs after detonation the seal between the

Figure 1 - Generic Flammability Curve



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head and the wall completely disengages (by more than 1 in.). The reactor contents would be vented to atmosphere and the split ring would probably dislodge. Also, the wall is significantly bowed outward. This situation worsens by 1.0 µs after the detonation and it is very likely that the vessel wall would have ruptured (unfortunately, we did not include fracturing in the simulation).

Since the anticipated operating conditions for the pilot reactor will keep the amount of organic and oxygen to low levels, we decreased the detonation pressure and final equilibrium pressure to about half of the initial values (5,000 psia and 1,000 psia, respectively) This necessitated increasing the pulse width because of the resulting decrease in detonation velocity at the lower detonation pressure. All other parameters remained the same. We feel that this is a more realistic condition in the event of an upset leading to increased oxygen levels in the reactor. The FE simulation of this condition showed the wall bowing outward to a final displacement of about 0.75 in. from start. The head comes uncomfortably close to completely disengaging from the vessel wall (i.e., it is possible that the seal would be compromised). Thus, with Titanium grade 7 as the vessel shell material it is possible that even a relatively small detonation could lead to limited venting of reactor contents to atmosphere. → [illegible] occur, [illegible] inside the vessel ?

There are other grades of titanium that have higher structural strengths. For example, titanium grade 5 (Ti-6Al-4V) has a yield strength at room temperature of 120,000 psi. Commercially pure titanium grades 2 and 7 have room temperature yield strengths of 40,000 psi[9]. The physical properties of titanium grade 5 were used along with the detonation pressure of 5,000 psia and pulse width of 214.9 µs to simulate the response of a 250 gal. vessel made of this material. The result was only a slight bowing of the wall (final displacement of about 0.10 in.) and maintenance of the seal between the reactor head and wall - the detonation was contained. Thus, it appears that the yield strength of the material used to fabricate the vessel is important in determining the ability of the vessel to resist a detonation. It would be prudent to fabricate the vessel shell from a titanium α,ß-alloy because if its higher yield strength. However, Ti grade 7 was chosen as the material of construction for the pilot unit because of its superior corrosion resistance (due to the addition of palladium[10]) as compared to Ti grade 2. We recommend investigating the possibility of using a Ti-6Al-4V alloy that has some palladium added (about 0.05 wt%) to increase the corrosion resistance as a preferred material of construction for the pilot reactor vessel shell.

V. References

1. The equivalence ratio of a fuel - oxygen mixture is a measure of how fuel-rich or feul-lean the mixture is. An equivalence ratio of 1.0 is the stoichiometric ratio of fuel (organic) to oxygen for complete combustion. Fuel-rich mixtures of equivalence ratios > 1.0.

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Appendix D – Operations Sensitivity Data for DETOX^{SM} Solutions

Operations Sensitivity Analysis Data for DETOX^{SM} Solutions

1.0 Introduction

Properties of DETOX^{SM} solutions from an operating environment are not well characterized. The purpose of these experiments and measurements was to gather realistic information to be used in the demonstration of DETOX^{SM} technology at DOE's Savannah River Site.

The solubilities and physical properties of selected metals and metal salts in the DETOX^{SM} solution were measured. The metals and metal salts used in these experiments were copper, copper chloride, aluminum, aluminum chloride, potassium chloride, sodium chloride, nickel chloride, zinc, zinc chloride, chromium oxide, and 316 stainless steel. The solubilities of sulfate and calcium sulfate in the DETOX^{SM} solution were measured. Solubility curves versus temperature in DETOX^{SM} solution were generated for aluminum chloride, calcium chloride, nickel chloride, and sodium chloride. Also studied were the mechanisms by which metals are oxidized in DETOX^{SM} solution, ferric ion reduction, and hydronium ion reduction.

2.0 Experimental

2.1 Metals solubilities in DETOX^{SM} solution.

The solubilities of metals and metal salts in the DETOX^{SM} solution were determined at 318. K. An excess amount of metal or metal salt was loaded into a 125. mL unstirred reaction bomb containing 50. mL of DETOX^{SM} solution. The bombs were sealed, pressurized to 35. psig with oxygen, and heated at 473. K for 16. hours. After cooling, each sample of the DETOX^{SM} solution was reheated to 308. K and filtered using a 1.2 µm glass fiber filter. The resulting saturated solutions were then analyzed using a Perkin-Elmer Model 603 atomic absorption spectrophotometer. Each test was performed in duplicate and the results were averaged.

2.2 Specific gravity.

The specific gravities of the metal solubility studies solutions were measured at 313. K, 333. K, and 353. K. A quantity of each solution was added to a preweighed 5. mL glass volumetric flask to give 5. mL volume of sample. The flask was immersed in a water bath to maintain the experimental temperature. The flask was then dried and weighed again. Each measurement was repeated twice for each of the two solutions from the metal solubility tests, and the results averaged.

2.3 Surface tension.

The surface tensions of the solutions from the metal solubility studies were measured using a Fisher Model 20 Surface Tensiometer. Surface tension was measured at 313. K, 333. K, and 353. K. Each measurement was repeated six times for each of the two solutions from the metal solubility tests, and the results were averaged.

2.4 Viscosity.

The viscosities of the solutions from the metal solubility studies were measured using an Ostwald viscometer. The viscometer was immersed in a water bath to maintain the experimental temperatures (313. K, 333. K, and 353. K). The time for a fixed volume of sample to flow between two reference marks in a vertical capillary tube was measured. Each measurement was repeated twice for each of the two solutions from the metal solubility tests, and the results averaged.

2.5 Metals oxidation.

The metal oxidation studies were conducted in Parr Instrument Company 23. mL, general purpose, Teflon^{TM}-lined reaction bombs. A weighed amount of metal or alloy (typically 100. mg) was loaded into the bomb along with 10. mL of DETOX^{SM} solution. The bombs were sealed and heated for 4. hours at 473. K. The bombs were cooled and the resulting solutions were diluted with deionized water to 100. mL in a volumetric flask. Duplicate aliquots of each solution were titrated with a standardized cerium (IV) solution to determine the amount of iron (II) produced when the metal or alloy was oxidized.

2.6 Sulfate anion solubility in DETOX^{SM} solution.

Sulfate anion (as sodium bisulfate) was tested for extent of solubility in the DETOX^{SM} solution. Varying amounts (200-400 µL) of a 1.02 M sodium bisulfate solution were loaded into bombs containing 10. mL of DETOX^{SM} solution. Parr Instrument Company (PIC) Model 4749 23. mL Teflon^{TM}-lined reaction bombs were used as test vessels. The bombs were heated for 16. hours at 473. K. After cooling, the contents of the each bomb were filtered through a 1.2 µm glass fiber filter in a preweighed Gooch crucible. The filter was rinsed with acetone and dried at 523. K to a constant weight. Duplicate tests were run with each sulfate loading.

2.7 Calcium sulfate solubility in DETOX^{SM} solution.

Calcium sulfate was tested for extent of solubility in the DETOX^{SM} solution. Varying amounts (200. - 500. µL) of a 1.0M sodium bisulfate solution were loaded into bombs containing 10. mL of DETOX^{SM} solution and 1.0M calcium. PIC Model 4749 23. mL Teflon^{TM}-lined reaction bombs were used as test vessels. The bombs were heated for 16 hours at 473. K. After cooling, the contents of the each bomb were filtered through a 1.2 µm glass fiber filter in a preweighed Gooch crucible. The filter was rinsed with acetone and dried at 523. K to a constant weight. Duplicate tests were run with each sulfate loading.

2.8 Solubilities of metal salts in DETOX^{SM} solution versus temperature.

Aluminum chloride, calcium chloride, nickel chloride, and sodium chloride were tested for extent of solubility in the DETOX^{SM} solution versus temperature. An excess of each salt was loaded into a 40. mL glass flask containing 20. mL of DETOX^{SM} solution. The flask was sealed and then immersed in a water bath to maintain the experimental temperature. After 30. minutes in the water bath, the solutions were sampled using an Eppendorf pipette and diluted in volumetric flasks with deionized water. The resulting solutions were then analyzed for the appropriate metal using a Perkin - Elmer Model 603 atomic absorption spectrophotometer. Duplicate tests were run with each material at 313. K, 333. K, 353. K and 365. K.

3.0 Results and Discussion

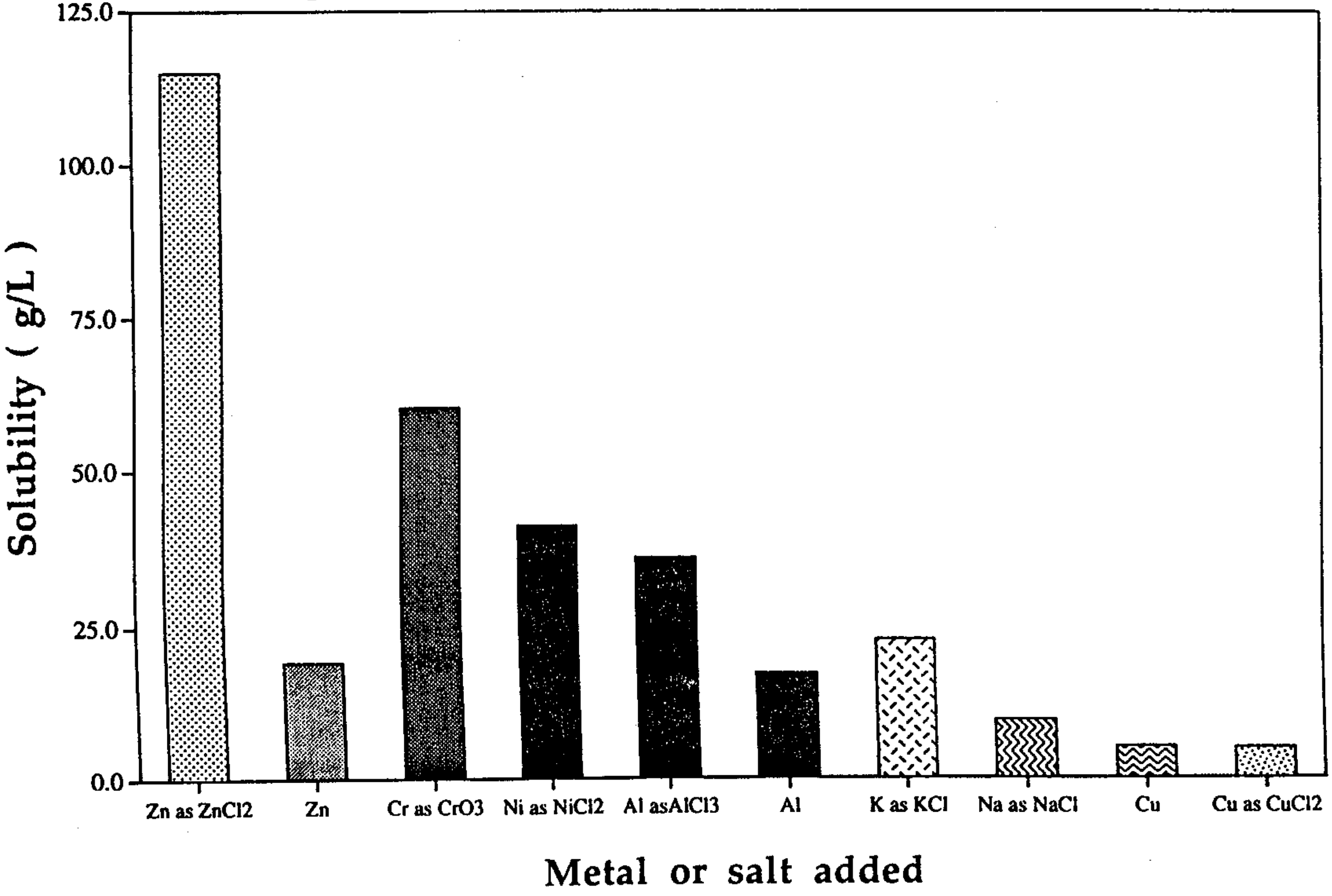
3.1 Metals' solubilities in DETOX^{SM} solution.

The metal solubility results are summarized in Table I and are compared graphically in Figure 1. As seen in Figure 1, the solubilities of metals in the DETOX^{SM} solution range from 115. g/L for zinc as zinc chloride to 5.2 g/L for copper as copper chloride. It is important to note that the solubilities of aluminum and zinc as the chlorides (37.7 and 115. g/L, respectively) were several times greater than the solubilities of the metals (17.8 and 19.5 g/L). This is believed to be due to reduction of the acidity of the DETOX^{SM} solution when metals are dissolved. However, the solubilities of copper and copper as copper chloride were nearly identical (5.4 and 5.2 g/L). The solubility of chromium as chromium oxide in the DETOX^{SM} solution reached approximately 60.4 g/L at 308. K temperature. Chromium (VI) in the DETOX^{SM} process is reduced to chromium (III). The solubility of the components of 316 stainless steel were 3.6 g/L for nickel and 12.4 g/L for chromium, although these are not the actual solubilities for these metals in the DETOX^{SM} solution.

Table I - Metal Solubility in DETOX^{SM}

Metal	Loading (g/L)	Solubility at 308. K (g/L)
316 Stainless Steel	60.	Ni - 3.6 ; Cr - 12.4
Aluminum	20.	17.8
Aluminum as $AlCl_3$	60.	37.7
Chromium as CrO_3	114.	60.4
Copper	40.	5.4
Copper as $CuCl_2$	19.	5.2
Potassium as KCl	157.	23.4
Sodium as NaCl	118.	10.0
Nickel as $NiCl_2$	190.	41.3
Zinc	20.	19.5
Zinc as $ZnCl_2$	140.	115.

Figure 1 - Metal Solubility in DETOX^{SM}



3.2 Specific gravity versus temperature for metals in DETOX^{SM} solution.

The results from the specific gravity measurements are summarized in Table II. Figures 2-5 illustrate the experimental measurements of specific gravity versus temperature for a group of metals in the DETOX^{SM} solution. The data were extrapolated to 473. K using a linear regression. It should be noted that extrapolation of specific gravities to temperatures less than the freezing point of the DETOX^{SM} solution is meaningless. The correlation coefficients are reasonably good for all of the extrapolations, with the exception of chromium oxide (Figure 4, $r^2 = 0.915$). The specific gravity results for the 316 stainless steel solution (Figure 4) were consistently lower than those of pure a DETOX^{SM} solution at each experimental temperature. The explanation for this remains unknown. The specific gravities for the other metals and salts in the DETOX^{SM} solution were generally higher than those of pure DETOX^{SM} at each temperature. The results for zinc as zinc chloride (Figure 5) are much greater at each temperature than those of the other metals. The reason for this is that the solubility for zinc as zinc chloride is much higher than the solubilities of the other metals and metal chlorides.

Table II - Specific Gravity Results

Solution	Specific Gravity		
	313. K	333. K	353. K
DETOX^{SM}	1.6344	1.6093	1.5741
DETOX^{SM} w/ 316 SS	1.6100	1.5743	1.5404
DETOX^{SM} w/aluminum	frozen	1.6195	1.6054
DETOX^{SM} w/Al as $AlCl_3$	1.6624	1.6412	1.6209
DETOX^{SM} w/Cr as CrO_3	1.6527	1.6495	1.6391
DETOX^{SM} w/copper	1.6563	1.6441	1.6247
DETOX^{SM} w/Cu as $CuCl_2$	1.6405	1.6119	1.5840
DETOX^{SM} w/ K as KCl	1.6224	1.6058	1.5905
DETOX^{SM} w/ Na as NaCl	1.6483	1.6357	1.6175
DETOX^{SM} w/ Ni as $NiCl_2$	1.6494	1.6288	1.5971
DETOX^{SM} w/zinc	1.6465	1.6336	1.6263
DETOX^{SM} w/Zn as $ZnCl_2$	1.7641	1.7354	1.7001

Specific gravity affects bubble size and agitator energy use. Higher specific gravity causes smaller bubbles and increased energy use.

Figure 2 - Specific Gravity vs. Temperature for Potassium Chloride and Sodium Chloride in DETOX^{SM}

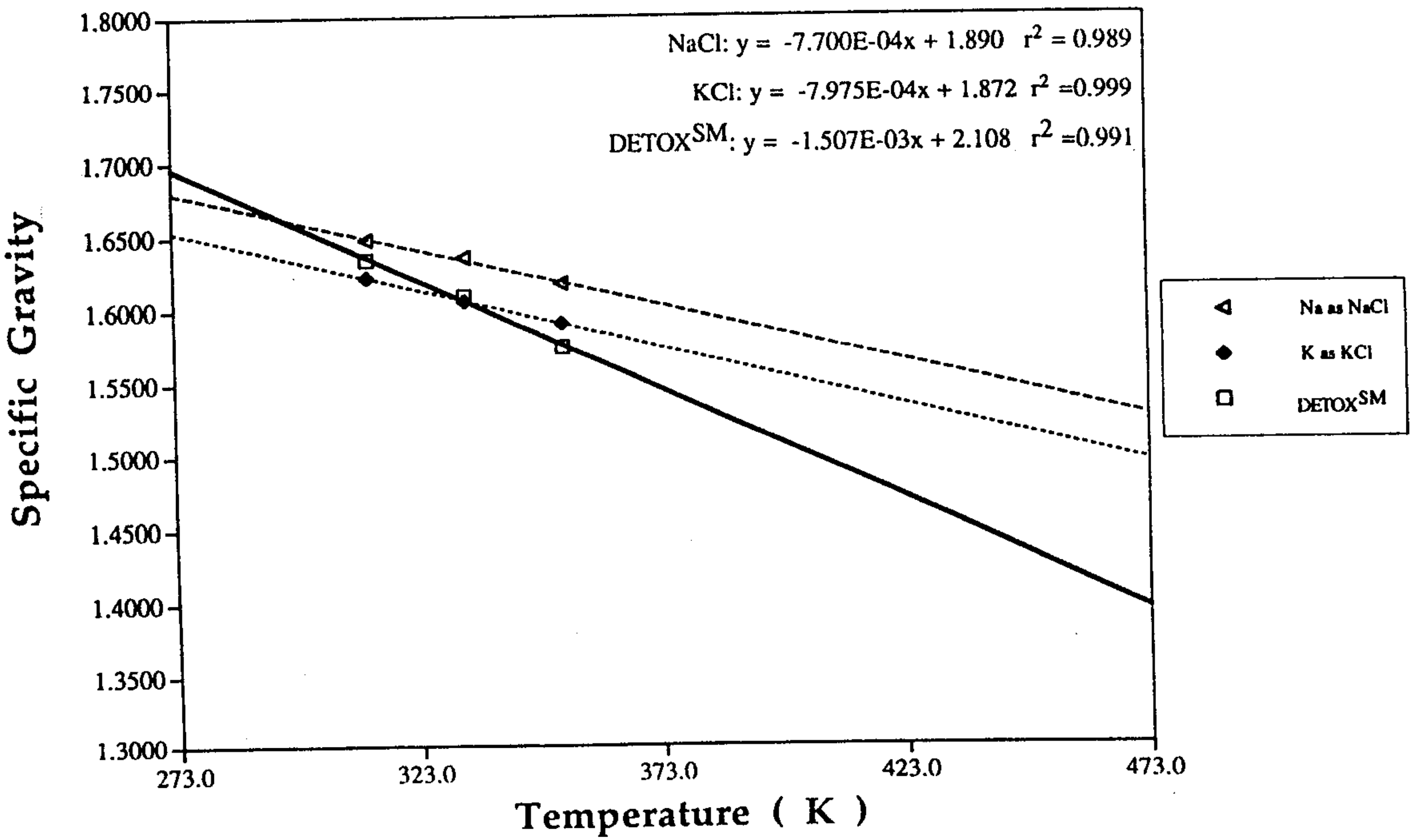


Figure 3 - Specific Gravity vs. Temperature for Copper and Copper Chloride in DETOX^{SM}

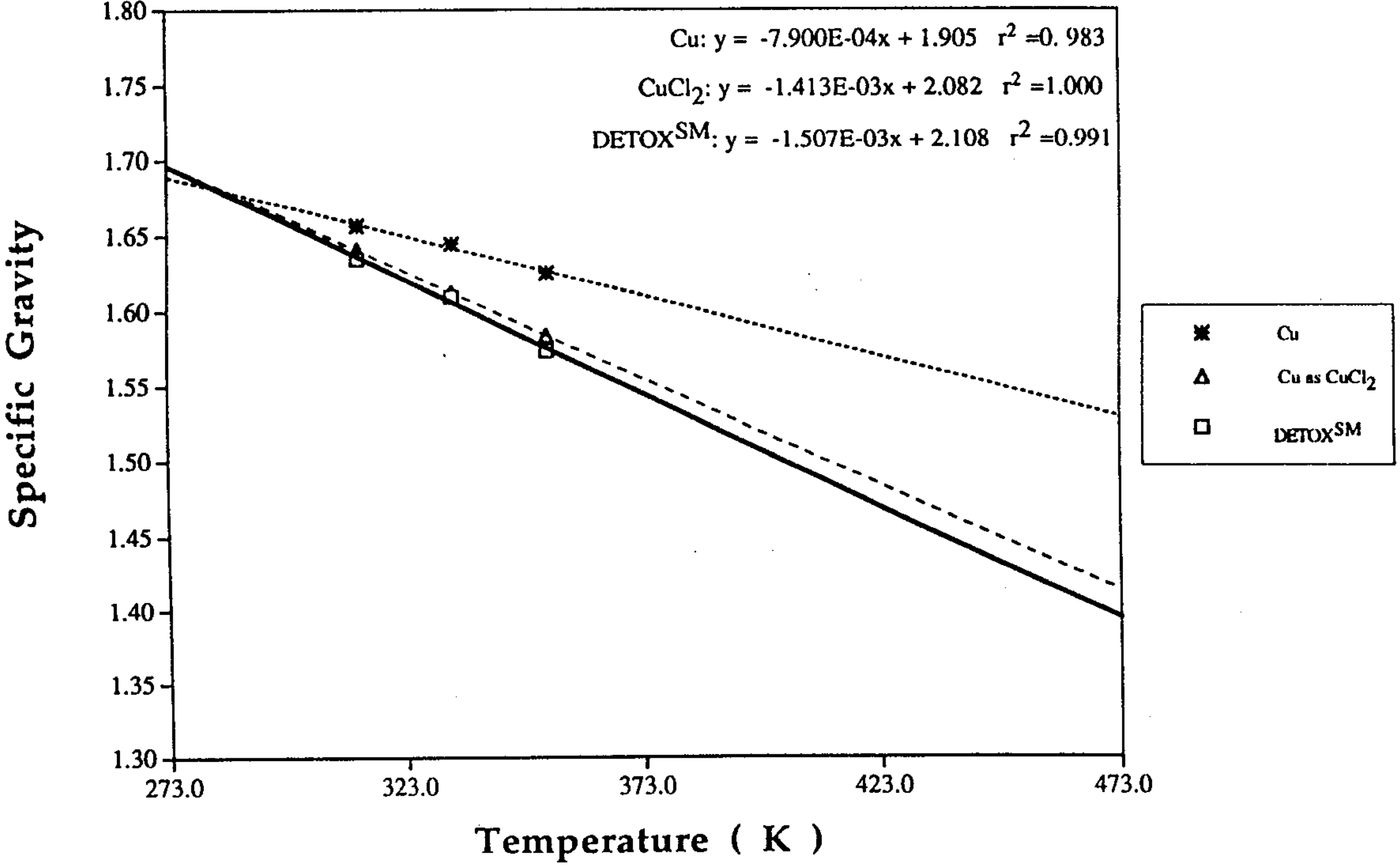


Figure 4 - Specific Gravity vs. Temperature for 316 Stainless Steel and Components in DETOX^{SM}

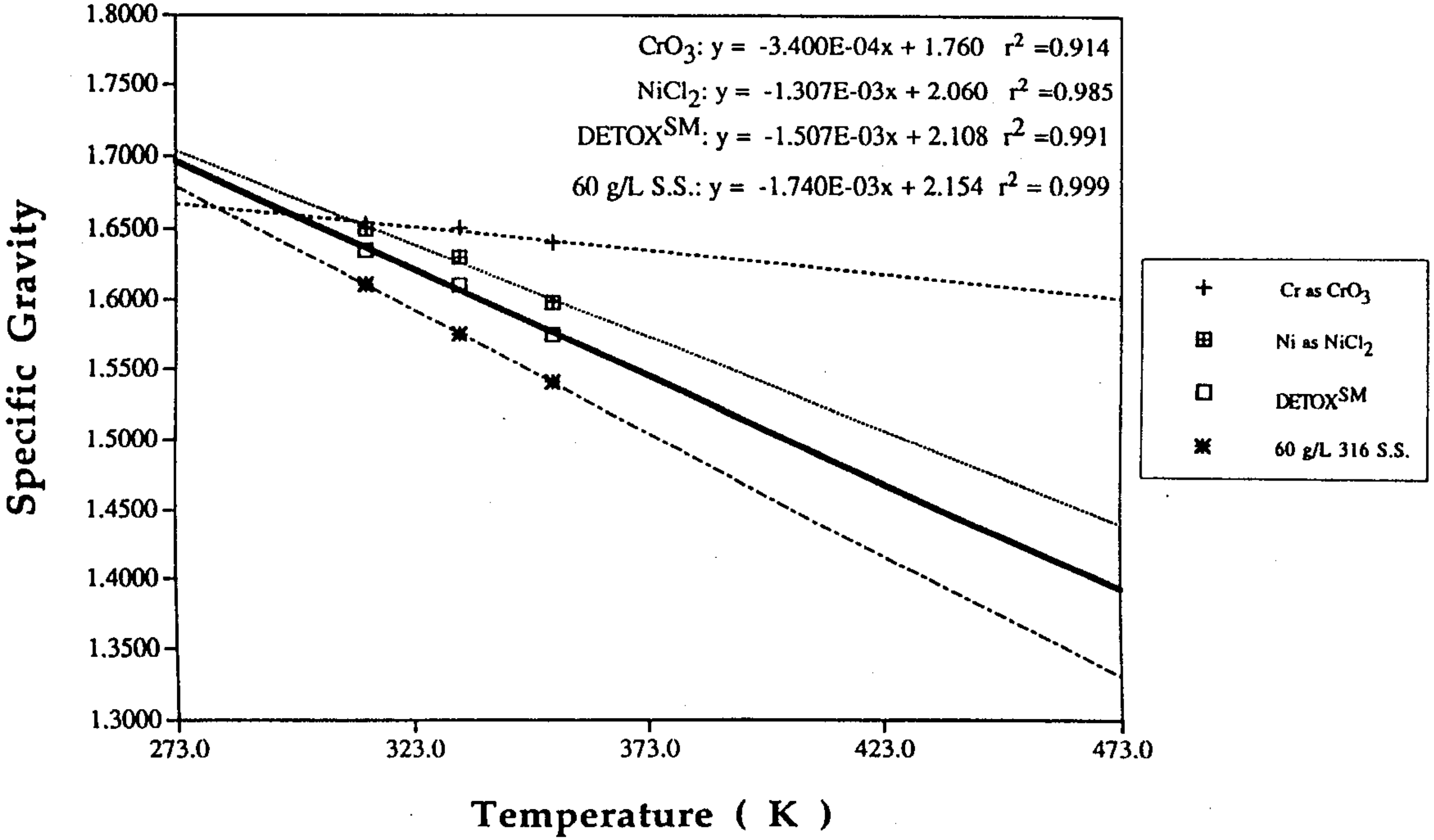
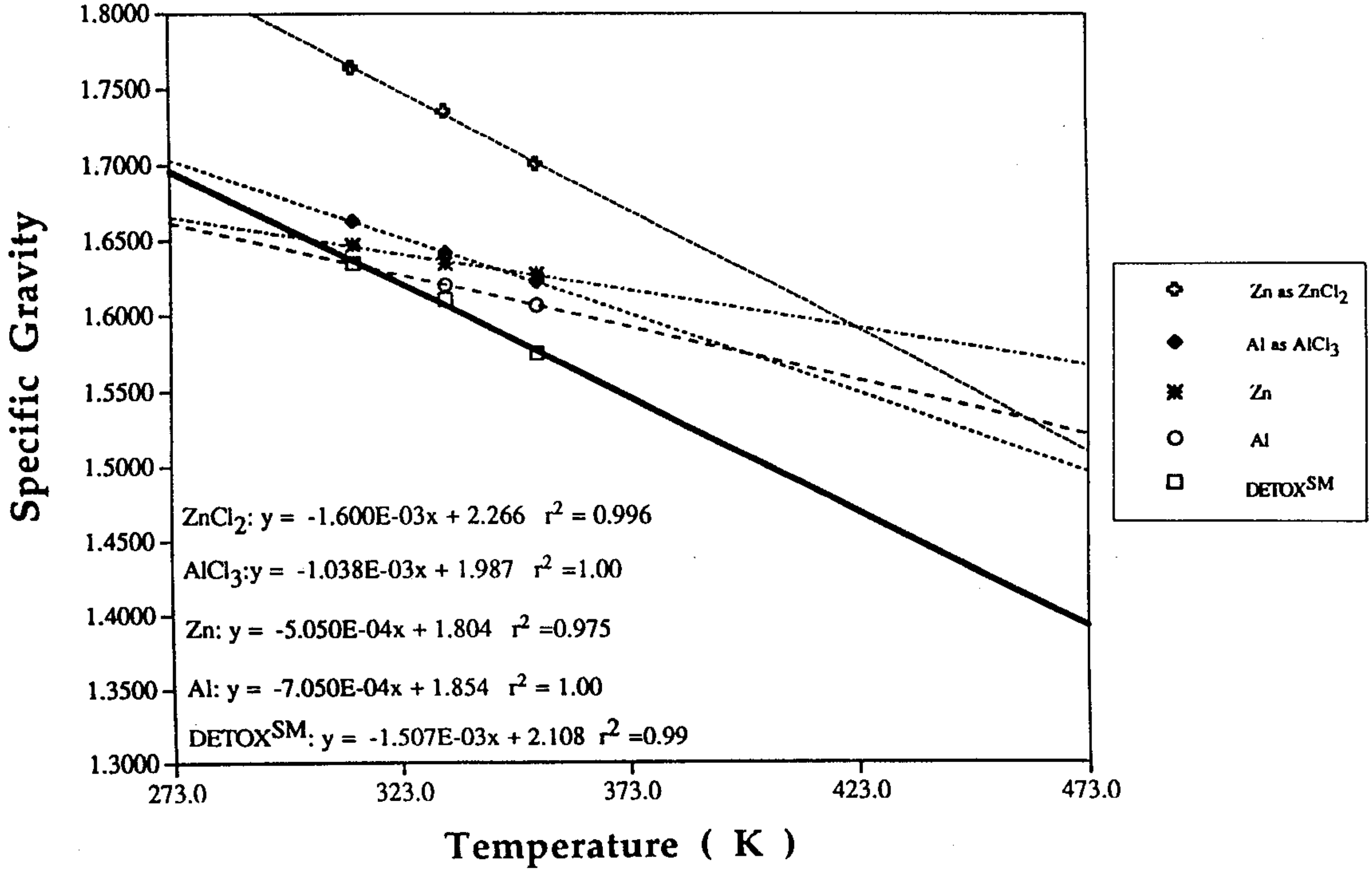


Figure 5 - Specific Gravity vs. Temperature for Aluminum, Aluminum Chloride, Zinc, and Zinc Chloride in DETOX^{SM}



3.3 Surface tension versus temperature for metals in DETOX^{SM} solution.

The results from the surface tension measurements are summarized in Table III. Figures 6-9 illustrate the experimental measurements of surface tension versus temperature for a group of metals in the DETOX^{SM} solution. The data were extrapolated to 473. K using a power curve regression. It should be noted that extrapolation of surface tensions to temperatures less than the freezing point of the DETOX^{SM} solution is meaningless. The curve fit correlation coefficients for all of the salt solutions are greater than that for the pure DETOX^{SM} solution ($r^2 = 0.940$).

As with the specific gravity results, the surface tension results for the 316 stainless steel solution (Figure 8) were lower than those of the DETOX^{SM} solution, while the results for the other metals were higher. The results for the zinc as zinc chloride solution (Figure 9) were again greater than those of the other metals.

Table III - Surface Tension Results

Solution	Surface Tension (dynes/cm)		
	313. K	333. K	353. K
DETOX^{SM}	66.24	63.04	61.92
DETOX^{SM} w/ 316 SS	63.08	59.05	57.37
DETOX^{SM} w/aluminum	69.58	67.64	66.59
DETOX^{SM} w/Al as $AlCl_3$	68.49	67.37	66.41
DETOX^{SM} w/Cr as CrO_3	73.31	71.02	69.34
DETOX^{SM} w/copper	70.12	67.36	65.50
DETOX^{SM} w/Cu as $CuCl_2$	69.13	66.98	64.93
DETOX^{SM} w/ K as KCl	70.41	68.89	66.83
DETOX^{SM} w/ Na as NaCl	69.68	67.01	62.51
DETOX^{SM} w/ Ni as $NiCl_2$	70.57	68.94	68.01
DETOX^{SM} w/zinc	67.91	66.28	64.45
DETOX^{SM} w/Zn as $ZnCl_2$	75.75	75.43	71.25

Surface tension affects bubble size and tendency to foam. Water has a surface tension of about 72 dynes/cm at room temperature. Decreased surface tension causes larger bubbles and more tendency to foam.

Figure 6 - Surface Tension vs. Temperature for Potassium Chloride and Sodium Chloride in DETOX^{SM}

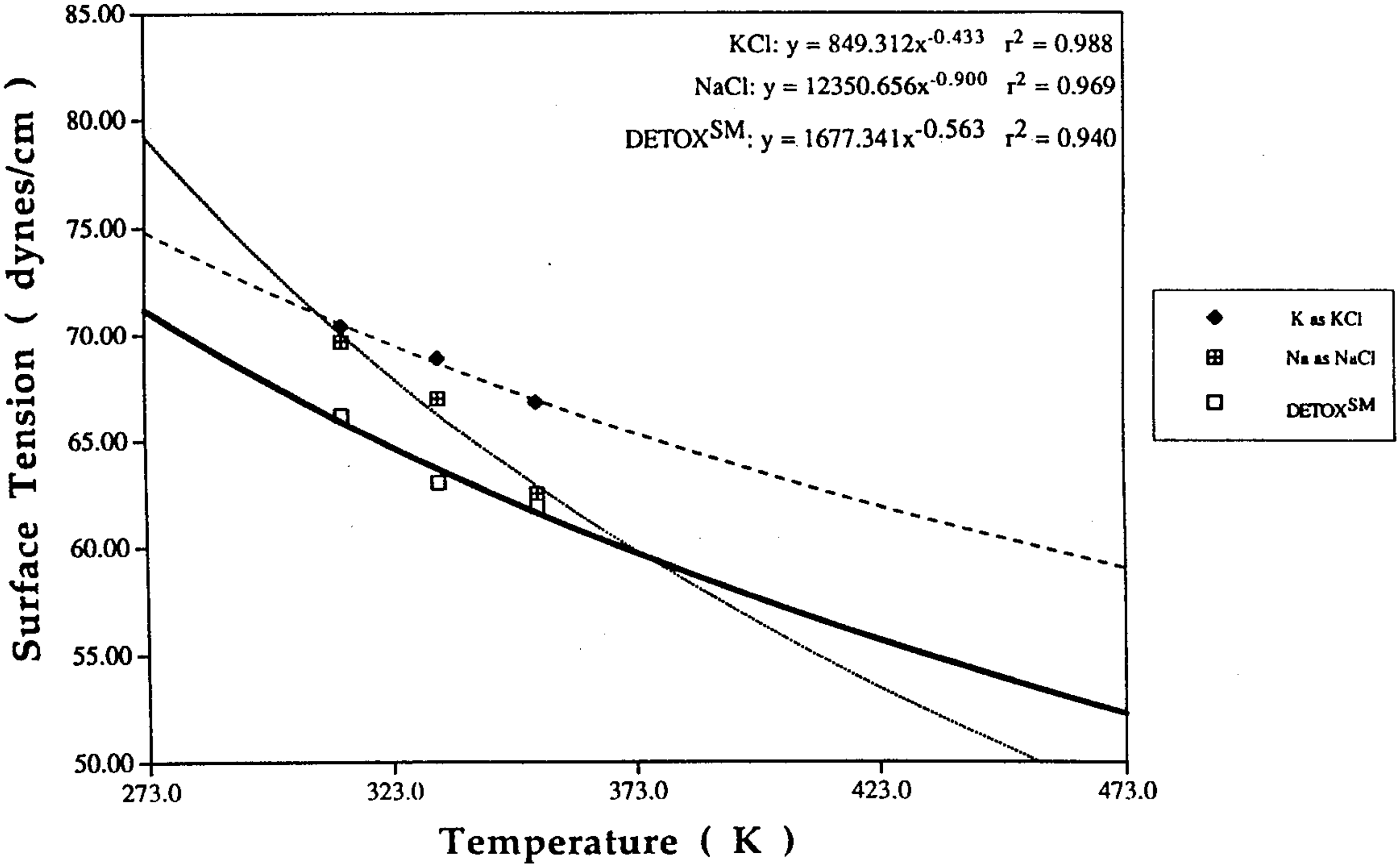


Figure 7 - Surface Tension vs. Temperature for Copper and Copper Chloride in DETOX^{SM}

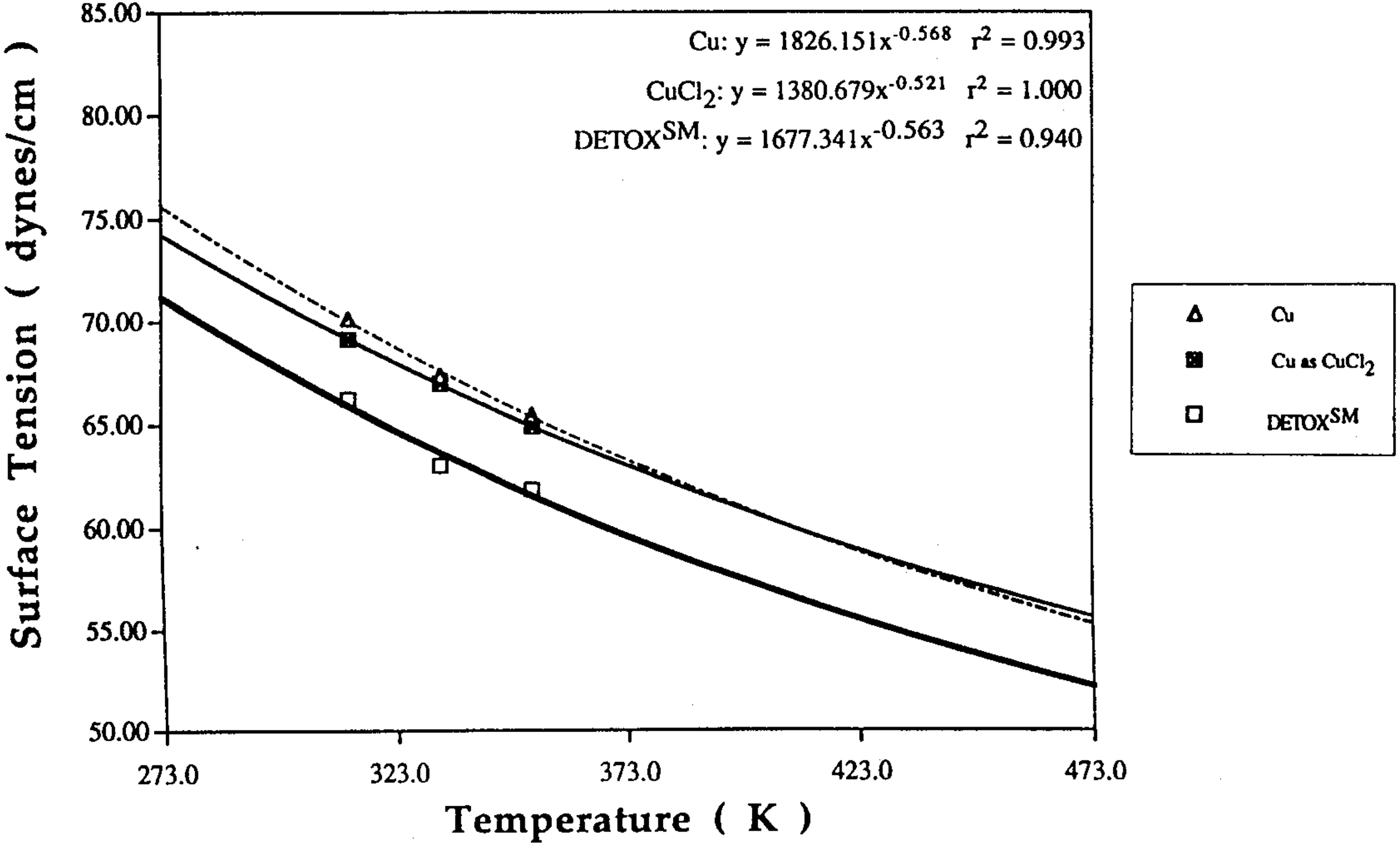


Figure 8- Surface Tension vs. Temperature for 316 Stainless Steel and Components in DETOX^{SM}

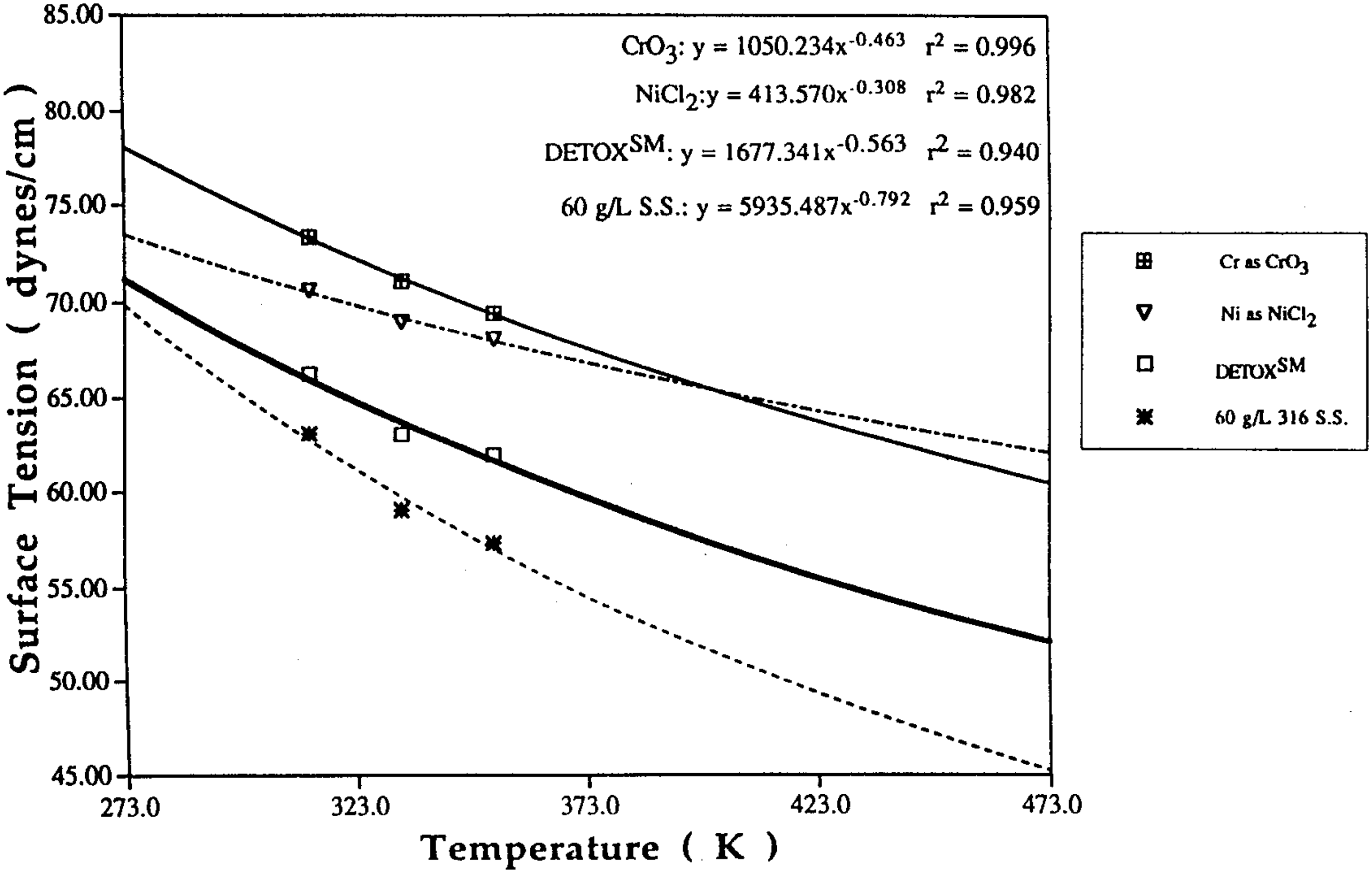
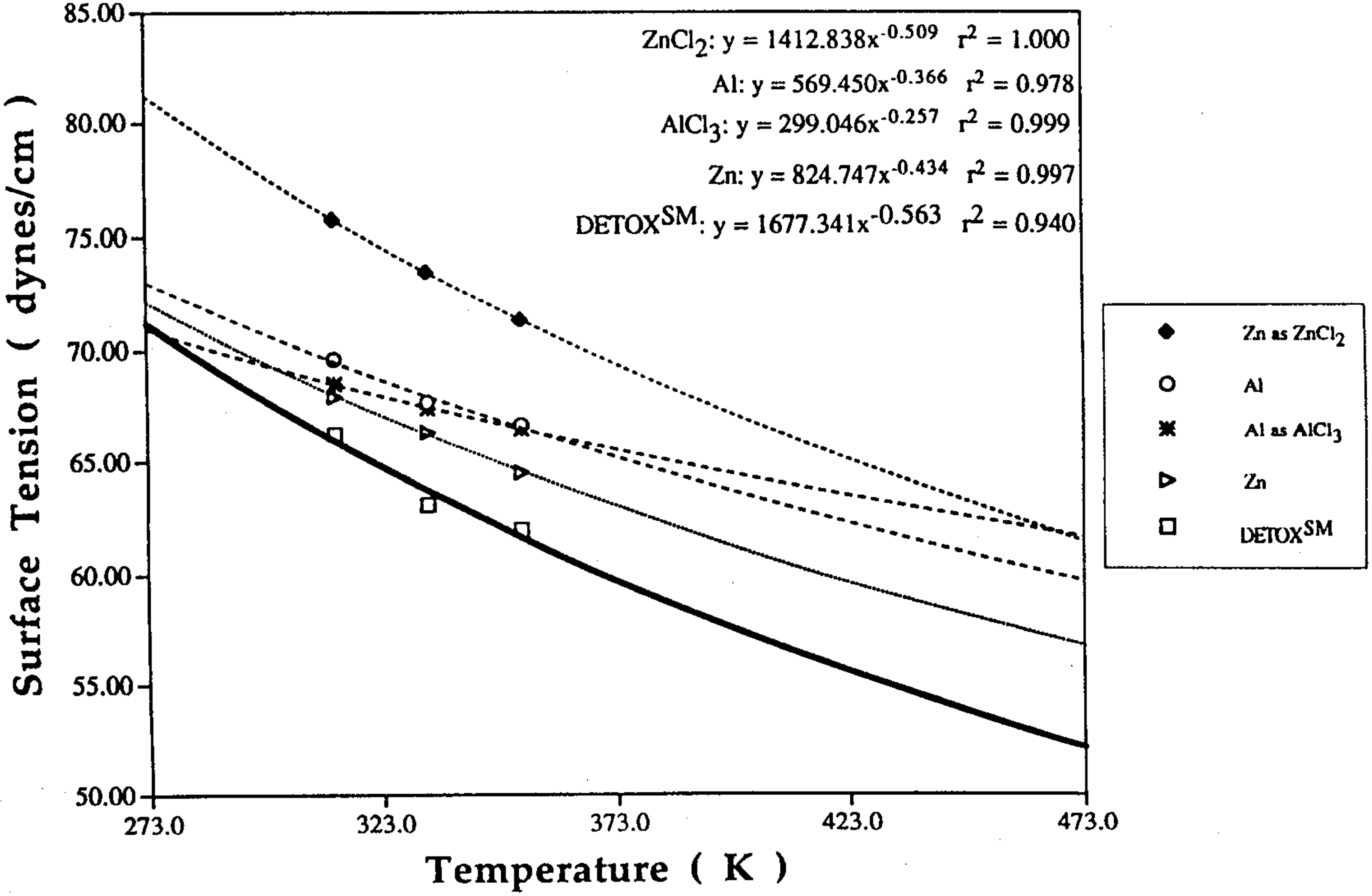


Figure 9 - Surface Tension vs. Temperature for Aluminum, Aluminum Chloride, Zinc, and Zinc Chloride in DETOX^{SM}



3.4 Viscosity versus temperature for metal solubility in DETOX^{SM} solution.

The results from the viscosity measurements are summarized in Table IV. Figures 10-13 illustrate the experimental measurements of viscosity versus temperature for a group of metals in the DETOX^{SM} solution. The data were extrapolated to 473. K using an exponential curve regression. It should be noted that extrapolation of viscosities to temperatures less than the freezing point of the DETOX^{SM} solution is meaningless. The correlation coefficients for all of the extrapolations are greater than that of the DETOX^{SM} solution ($r^2 = 0.981$).

At 313. K , the viscosities for solutions of copper as copper chloride, potassium, sodium, nickel and zinc in the DETOX^{SM} solution are lower than that of the pure DETOX^{SM} solution. At the higher temperatures, the results for all of the salt solutions in DETOX^{SM} are equal to or higher than those of the pure DETOX^{SM} solution. As with the specific gravity and surface tension results, the viscosity results for the zinc as zinc chloride solution (Figure 13) were much greater than those of the other metals.

Table IV - Viscosity Results

Solution	Viscosity (centipoise)		
	313. K	333. K	353. K
DETOX^{SM}	9.91	4.02	2.32
DETOX^{SM} w/ 316 SS	10.13	4.29	2.38
DETOX^{SM} w/aluminum	11.72	5.41	3.26
DETOX^{SM} w/Al as $AlCl_3$	13.44	8.47	5.64
DETOX^{SM} w/Cr as CrO_3	17.90	8.48	4.20
DETOX^{SM} w/copper	11.08	5.00	2.84
DETOX^{SM} w/Cu as $CuCl_2$	7.91	4.17	2.46
DETOX^{SM} w/ K as KCl	9.57	4.46	2.45
DETOX^{SM} w/ Na as NaCl	8.76	4.07	2.40
DETOX^{SM} w/ Ni as $NiCl_2$	7.91	4.00	2.46
DETOX^{SM} w/zinc	9.69	5.41	3.01
DETOX^{SM} w/Zn as $ZnCl_2$	22.64	10.62	5.61

Viscosity affects bubble rise times in the DETOX^{SM} solution, tendency of the solution to bump, and agitator energy use. Water has a viscosity of approximately 1.0 centipoise at room temperature. Higher viscosity causes longer bubble rise times, increases agitator energy use, and increases the tendency of the solution to bump.

Figure 10 - Viscosity vs. Temperature for Sodium Chloride and Potassium Chloride in DETOX^{SM}

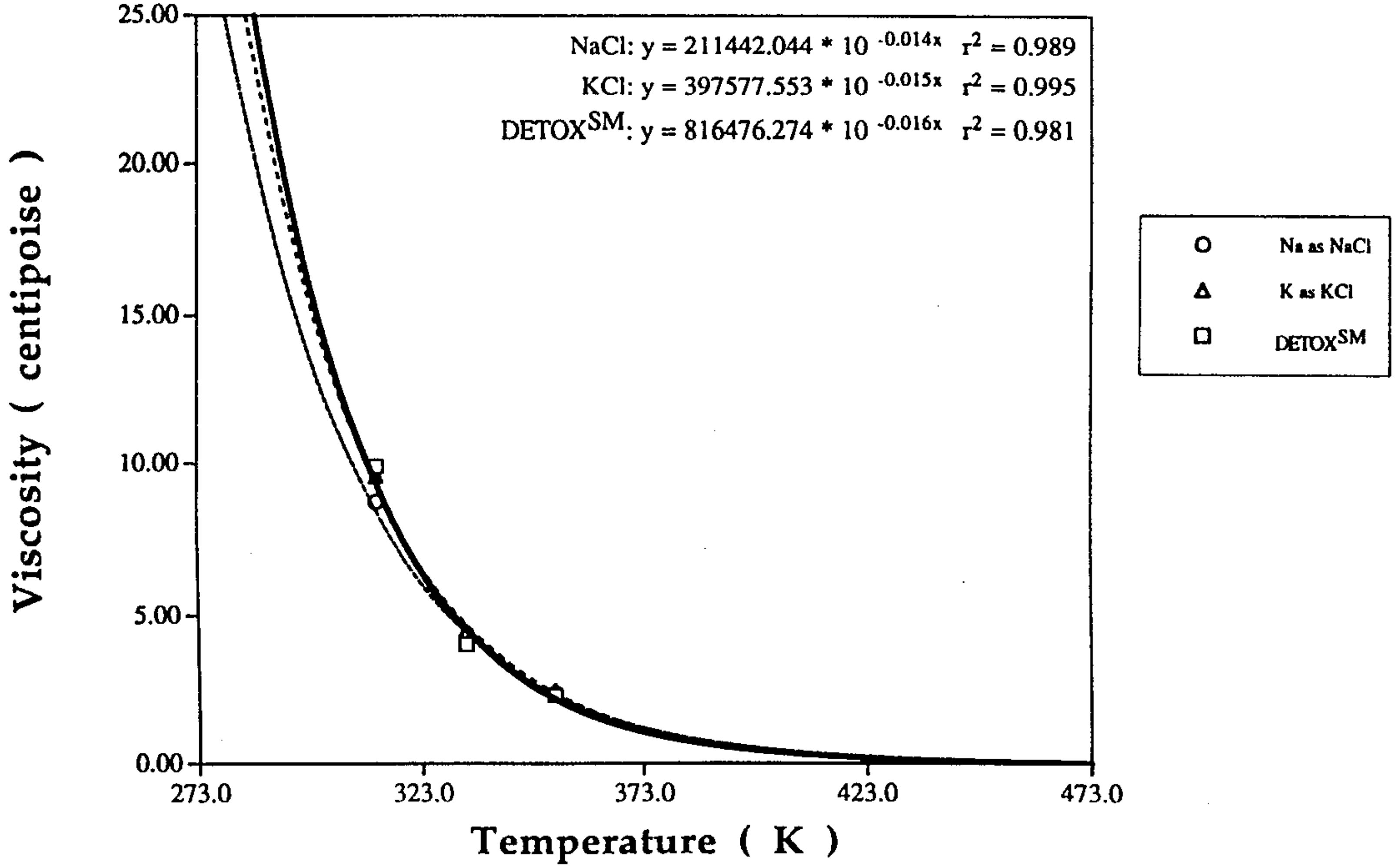


Figure 11 - Viscosity vs. Temperature for Copper and Copper Chloride in DETOX^{SM}

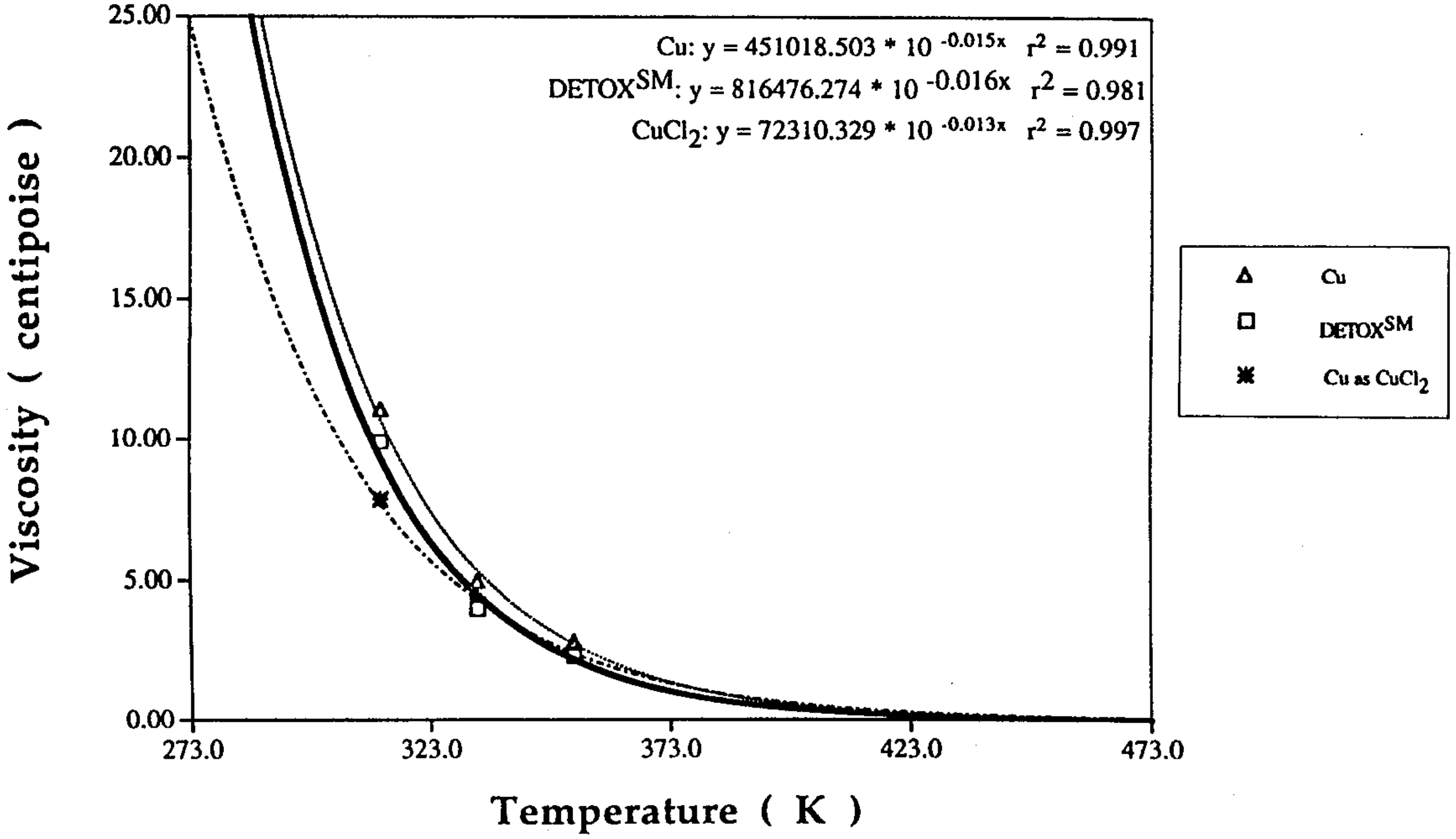


Figure 12 - Viscosity vs Temperature for 316 Stainless Steel and Components in DETOX^{SM}

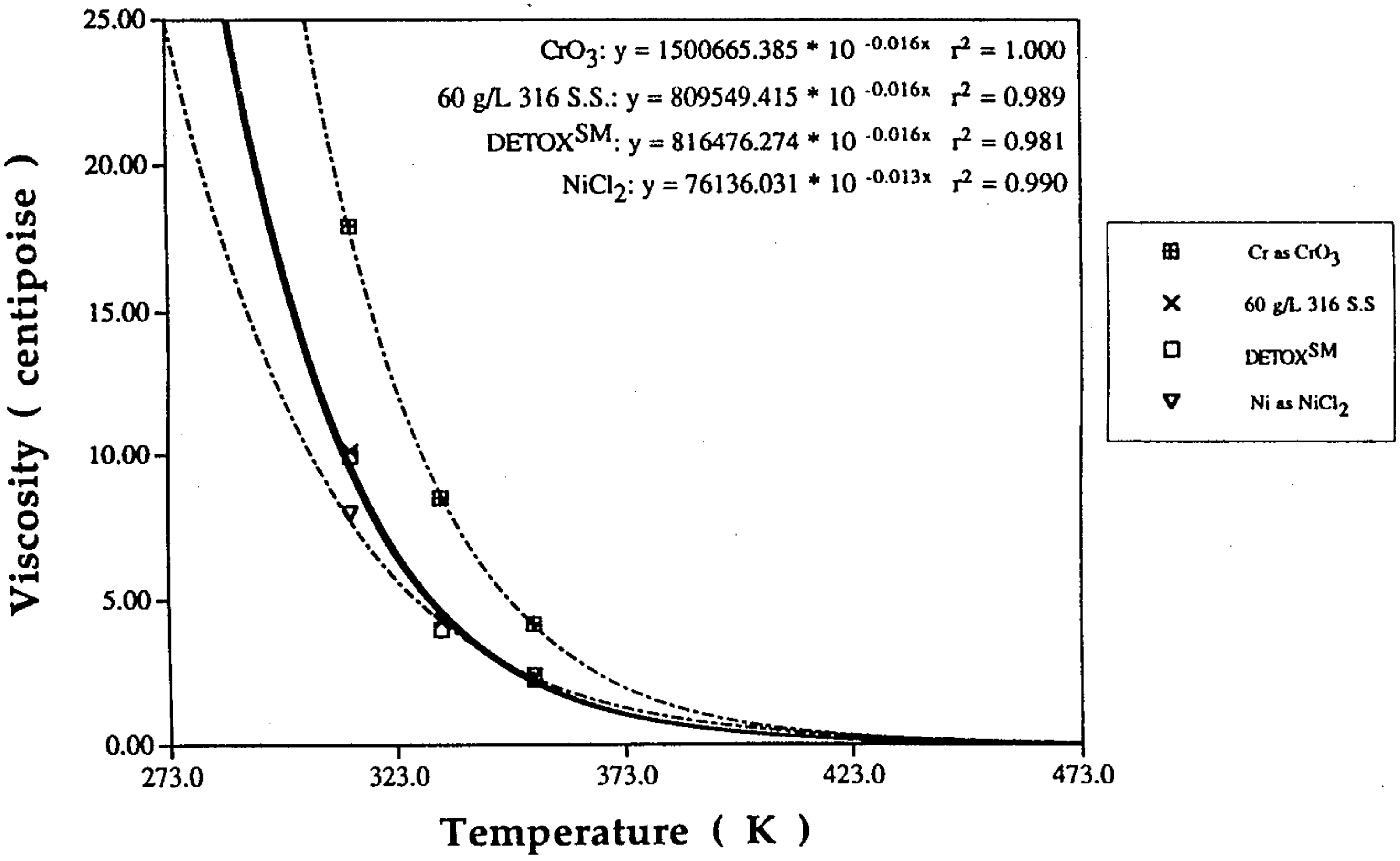
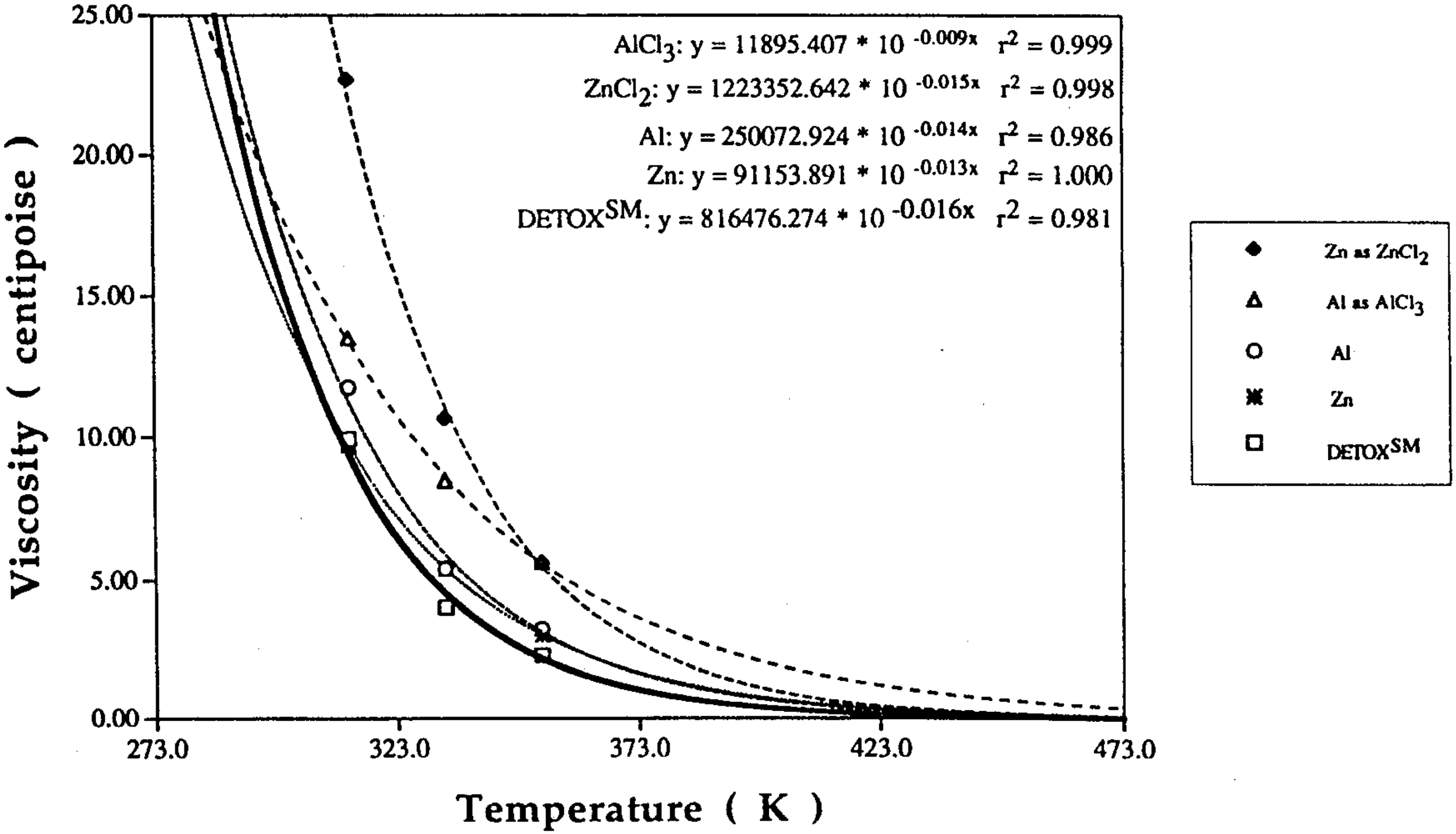


Figure 13 - Viscosity vs. Temperature for Aluminum, Aluminun Chloride, Zinc, and Zinc Chloride in DETOX^{SM}



3.5 Metals Oxidation.

Metals introduced into the process solution can be oxidized by either reducing ferric iron to ferrous iron, or by reducing hydronium ion to hydrogen gas. Three metals and two alloys were chosen to determine the relative preferences for these two reduction pathways. These metals and alloys represent those most likely to be seen in waste streams from the possible demonstration sites. The difference between the number of moles of electrons necessary for complete oxidation of the added metal or alloy and the number of moles of electrons used in forming ferrous iron gives the number of moles of electrons that reduced hydronium ion to hydrogen gas. These values give the fractions of ferric and hydronium ion reduced by the added metal or alloy and are summarized in Table V.

Table V - Metal Oxidation Results

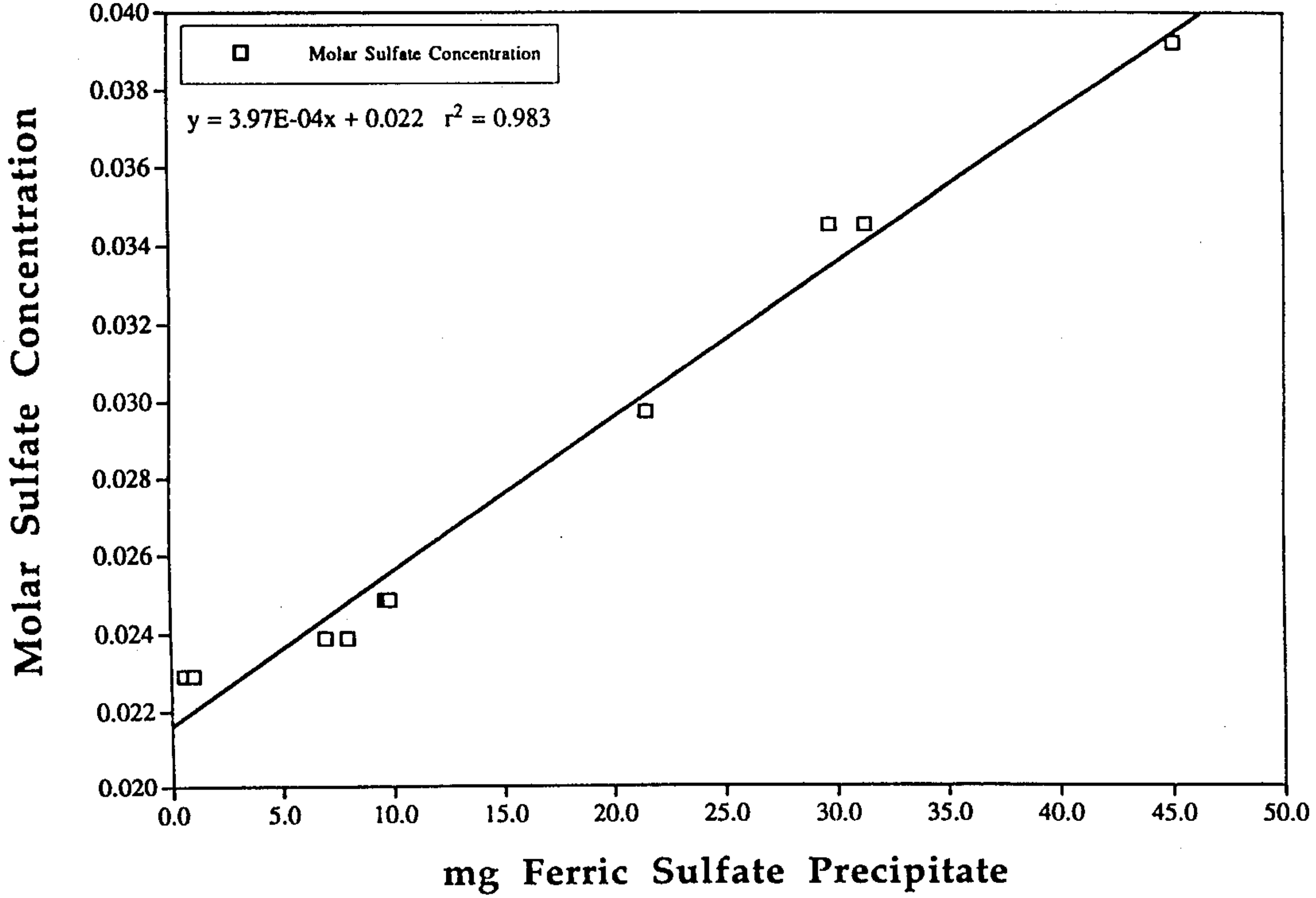
Metal or Alloy	Percent Ferric Iron Reduced by Metal	Percent Hydronium Ion Reduced by Metal
Aluminum	94.1	5.9
Copper	92.3	7.7
Zinc	94.8	5.2
316 Stainless Steel	83.6	16.4
Carbon Steel	88.2	11.8

The data show that the majority of the metal or alloy is oxidized by reducing ferric iron to ferrous iron. This is preferred from an operational standpoint because the amount of hydrogen generation and hydrochloric acid use are minimized during waste processing. Embrittlement of the tantalum liner in the reactor by hydrogen absorption is not expected to occur. The above data, showing low hydrogen generation, eliminate much of the risk to the tantalum liner associated with hydrogen absorption.

3.6 Sulfate anion solubility in DETOX^{SM} solution.

Figure 14 illustrates sulfate concentration in the DETOX^{SM} solution versus milligrams of ferric sulfate precipitate. The data was extrapolated to 0.0 milligrams of precipitate using a linear regression. The y-intercept, 0.022, gives the molar concentration of sulfate at 0.0 milligrams precipitate, or in other words, the solubility of sulfate in DETOX^{SM} solution. Sulfate is fairly insoluble in the DETOX^{SM} solution, and would precipitate the iron catalyst out of the solution.

Figure 14 - Sulfate Solubility in DETOX^{SM}



3.7 Calcium sulfate solubility in DETOX^{SM} solution.

The results from the calcium sulfate solubility tests are summarized in Table VI. At a concentration of 1.0 M calcium in DETOX^{SM}, (the concentration to be used in the demonstration), three of the four tests showed at least 98.5% efficiency in scavenging sulfate. The result from sample #2, 60.3%, was attributed to loss of precipitate during filtration. The results show that very low levels of sulfate can be efficiently scavenged using 1.0 M calcium in DETOX^{SM} solution.

Table VI - Calcium Sulfate Solubility Results

Sample	[Calcium] (moles/L)	Moles Sulfate Added	Moles Sulfate as Precipitate ($CaSO_4$)	Percent Sulfate Precipitated as $CaSO_4$
#1	1.0	2.04E-04	2.01E-04	98.5
#2	1.0	2.04E-04	1.23E-04	60.3
#3	1.0	5.10E-04	5.10E-04	100.0
#4	1.0	5.10E-04	5.17E-04	101.4

Calcium sulfate is much less soluble than iron sulfate. Thus, calcium can be used to scavenge sulfate from the DETOX^{SM} solution. A 1.0 M concentration of calcium will be effective in scavenging sulfate, and will also scavenge fluoride.

3.8 Solubilities of metal chlorides in DETOX^{SM} solution versus temperature.

Figures 15-22 illustrate experimental solubility data for nickel chloride, calcium chloride, sodium chloride and aluminum chloride in the DETOX^{SM} solution versus temperature. The data were extrapolated to 473. K.

In general, the logarithm of solubility is directly proportional to the temperature raised to the -1.5 power. The experimental data is fitted by theoretical solubility curves in Figures 15, 17, 19 and 21. The theoretical solubility relationship is represented by a linear regression. The experimental data of solubility versus temperature for the metal chlorides in the DETOX^{SM} solution in Figures 16, 18, 20 and 22 are more conservatively fitted by logarithmic curves. The correlation coefficients are not significantly different (< 5%) between the theoretical curves and the experimental curves for each metal chloride. The solubility of each metal chloride in DETOX^{SM} increases as temperature increases from 313. K to 473. K. At each experimental temperature, the highest solubility is that of aluminum chloride, followed by calcium, nickel and sodium chlorides.

Solubilities of these metals as chlorides increase with temperature, and so saturated solutions of these metal chlorides will precipitate as their temperature is decreased. Thus, they will be filtered out of the process solution after it is cooled from 473. K to 373. K en route to the unit's filter. Build-up of salts in the DETOX^{SM} process solution will be limited to their solubilities at approximately 373. K.

Figure 15 - Theoretical Solubility Curve for Aluminum Chloride in DETOX^{SM}

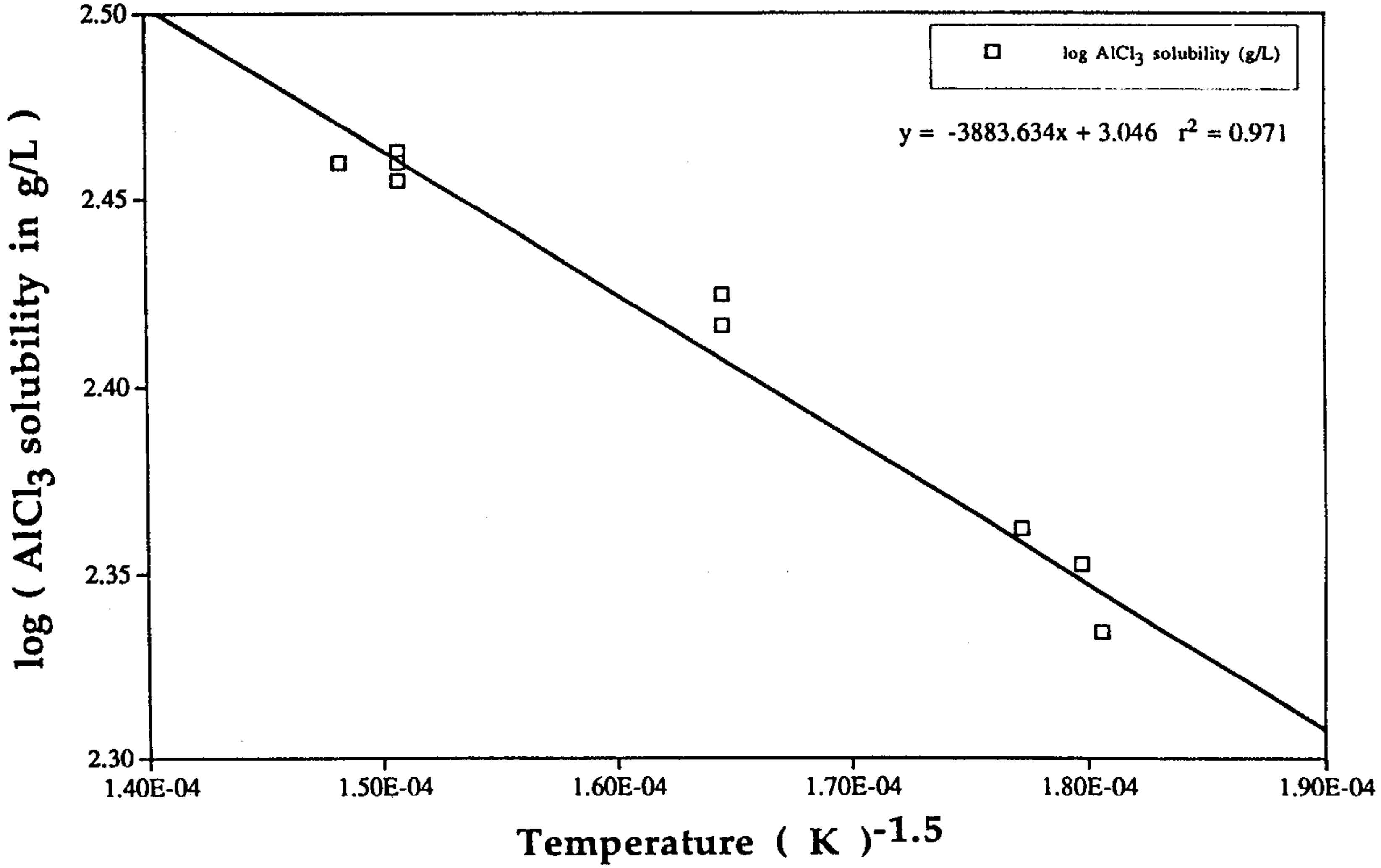


Figure 16 - Experimental Solubility Curve for Aluminum Chloride in DETOX^{SM}

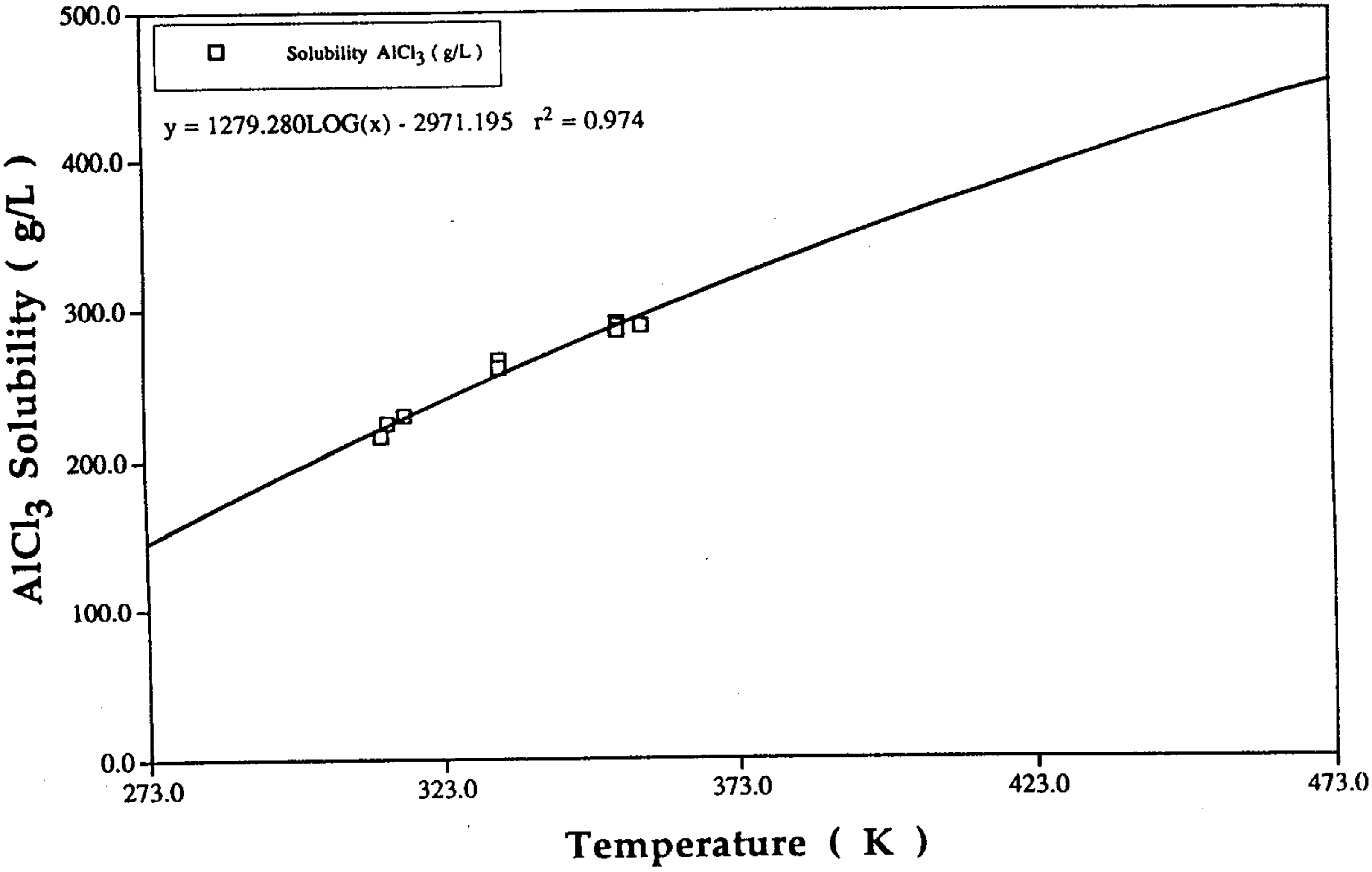


Figure 17 - Theoretical Solubility Curve for Calcium Chloride in DETOX^{SM}

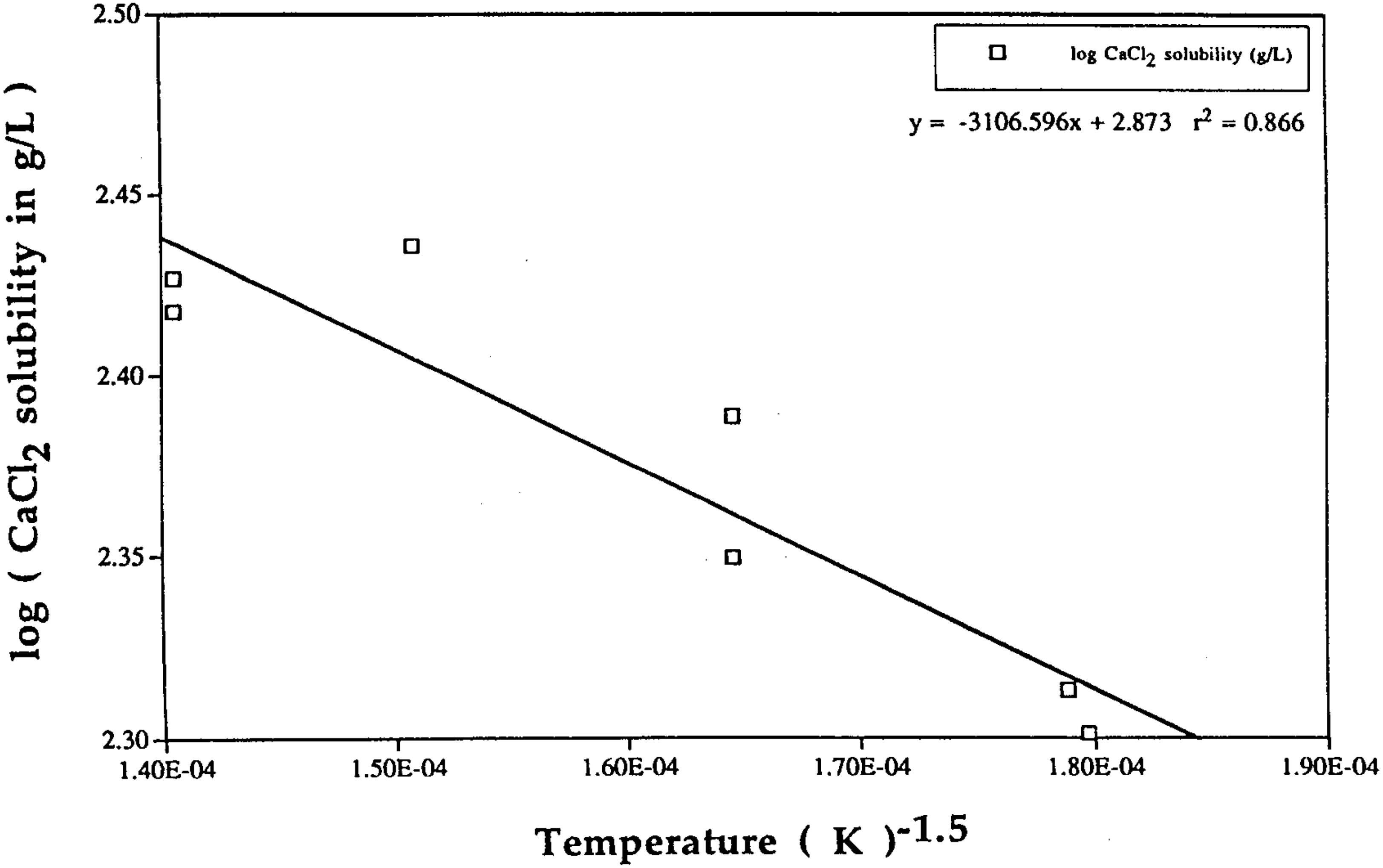


Figure 18 - Experimental Solubility Curve for Calcium Chloride in DETOX^{SM}

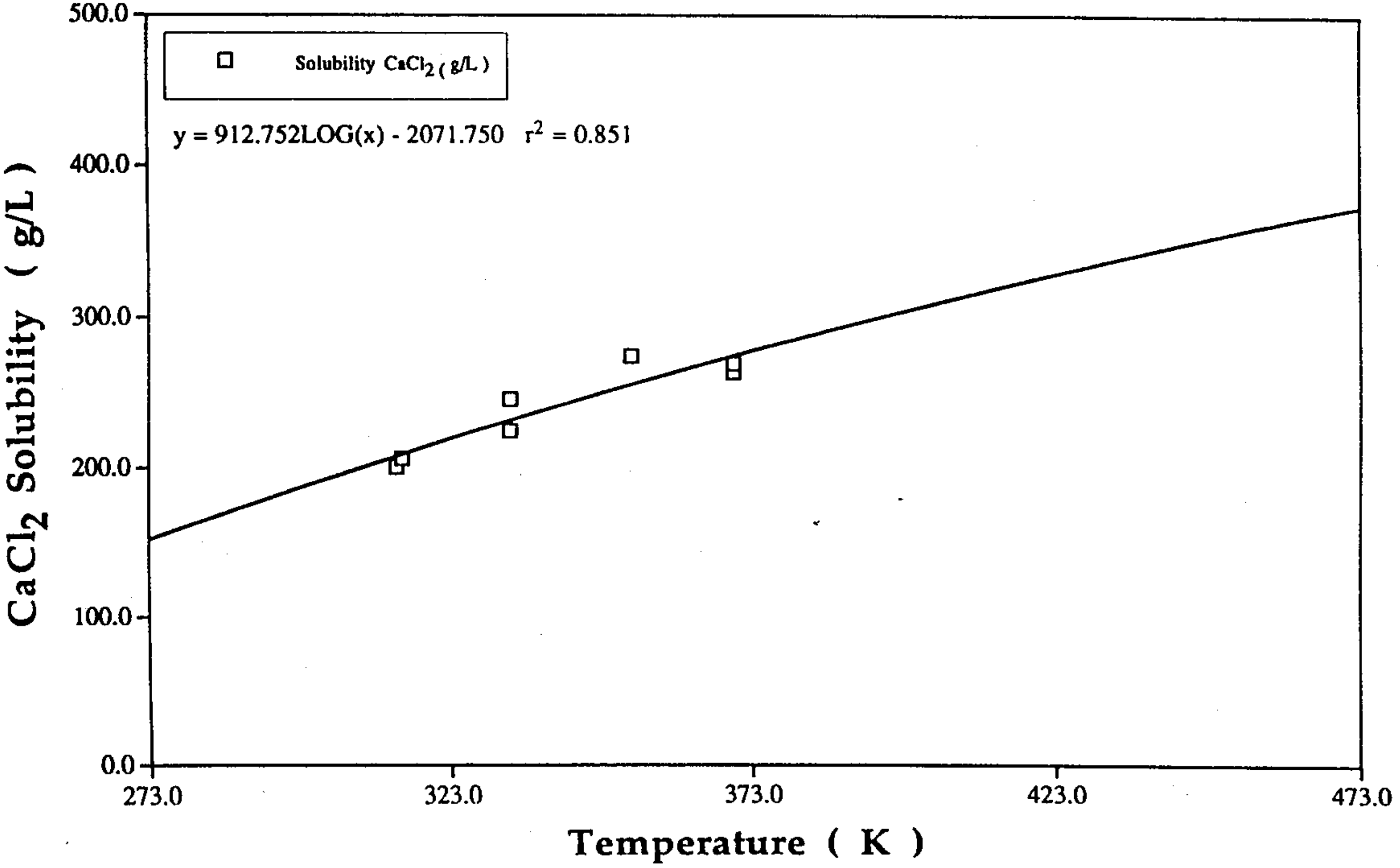


Figure 19 - Theoretical Solubility Curve for Nickel Chloride in DETOX^{SM}

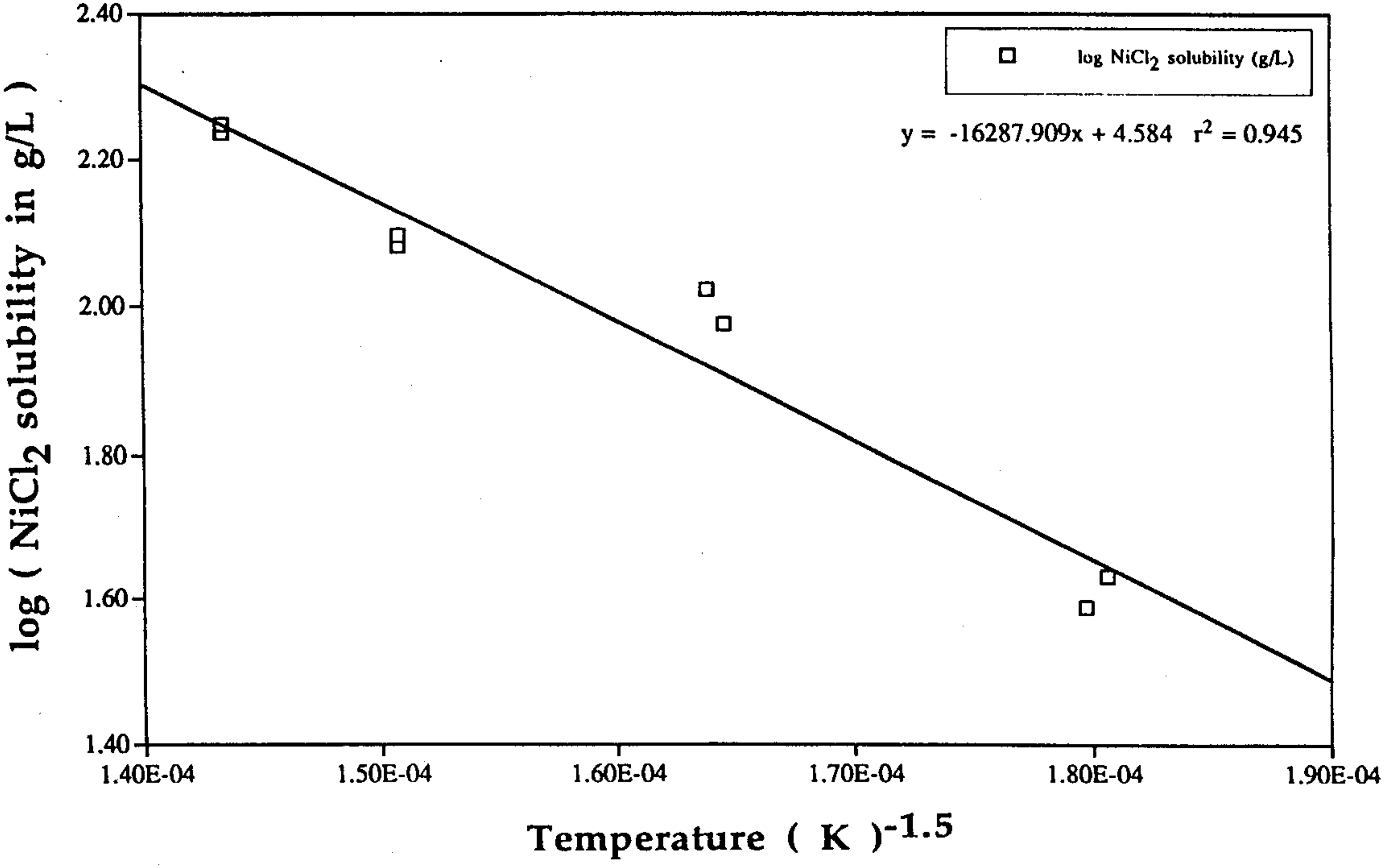


Figure 20 - Experimental Solubility Curve for Nickel Chloride in DETOX^{SM}

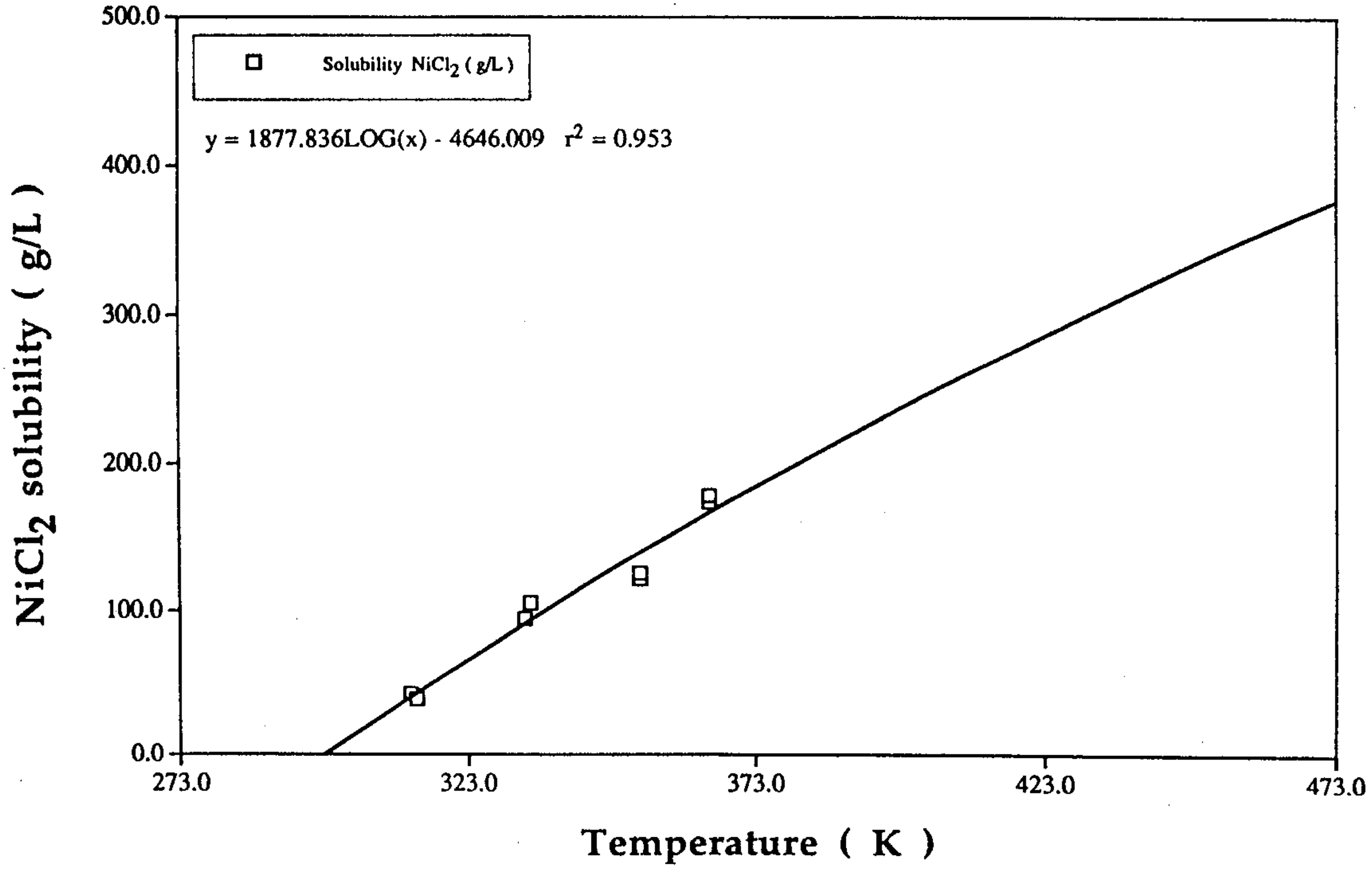


Figure 21 - Theoretical Solubility Curve for Sodium Chloride in DETOX^{SM}

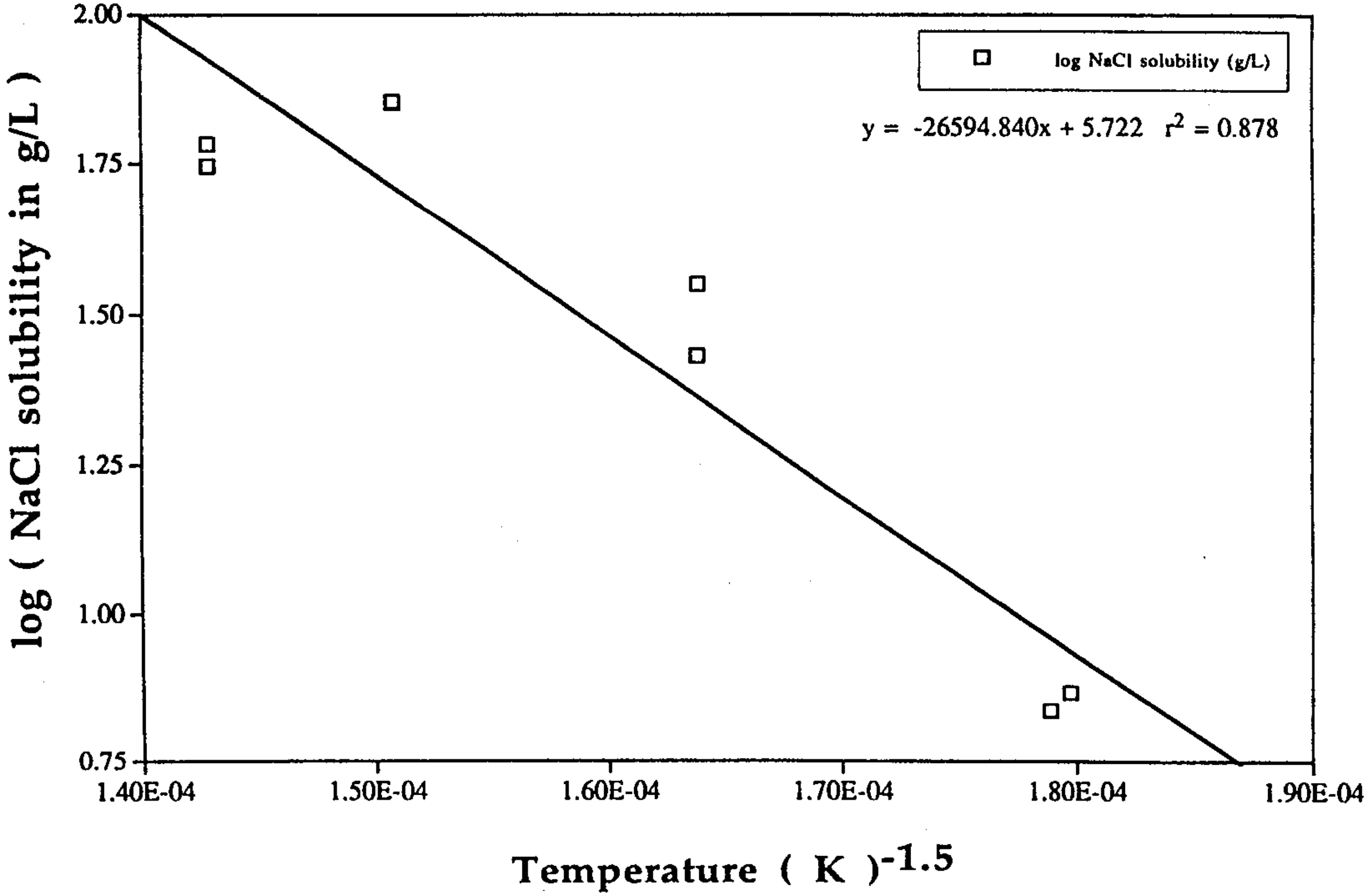
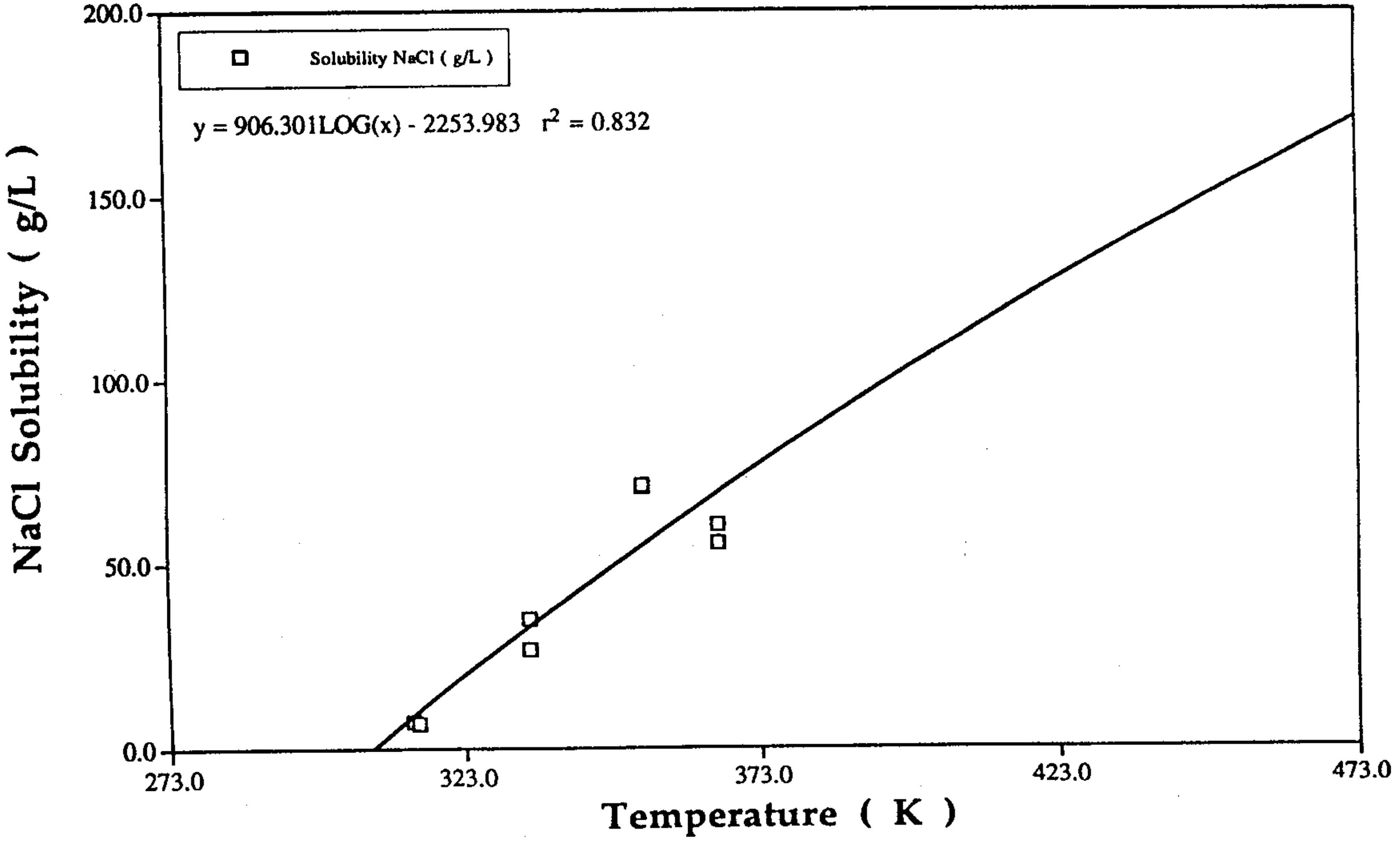


Figure 22 - Experimental Solubility Curve for Sodium Chloride in DETOX^{SM}



4.0 Conclusions

The solubilities of several metals in the DETOX^{SM} solution, and some of the physical properties of the resulting solutions, were successfully measured.

In general, the specific gravity, surface tension, and viscosity of a DETOX^{SM} solution saturated with a metal or metal chloride are functions of the concentration of the metal or metal chloride.

Specific gravity, surface tension, and viscosity tended to be greater for the salt solutions than for pure DETOX^{SM} solution. Overall, the effects of soluble materials in the DETOX^{SM} solution should be smaller bubble sizes, increased stirrer energy use, and longer bubble residence times.

The majority of any metal or alloy added to the DETOX^{SM} solution is oxidized by ferric ion, thus minimizing hydrogen generation during processing of wastes containing metals or alloys.

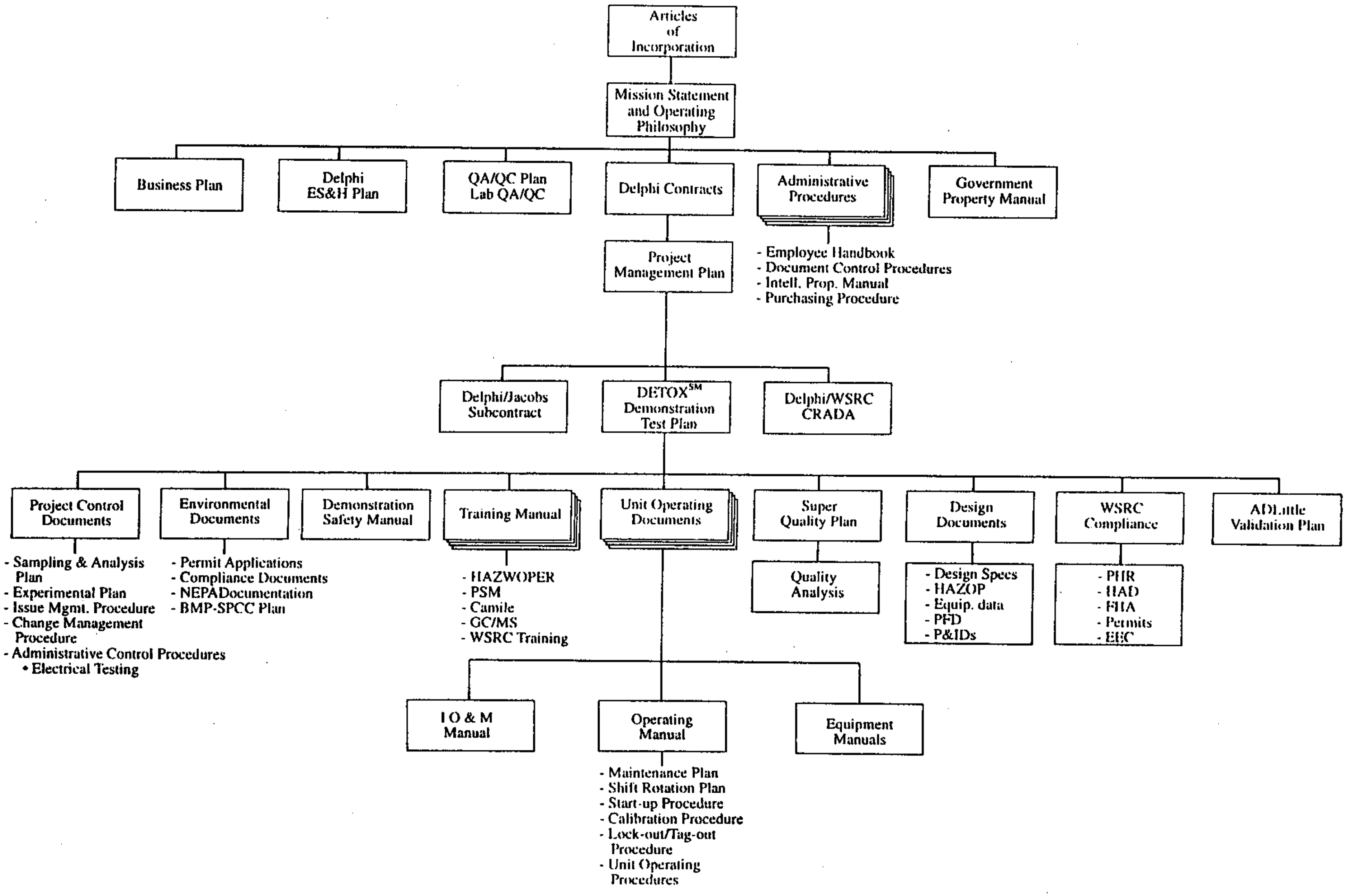
The solubility of sulfate anion in DETOX^{SM} is 0.022 $\underline{M}$. Low levels of sulfate contaminants can be efficiently precipitated from a 1.0 $\underline{M}$ calcium/DETOX^{SM} solution as calcium sulfate.

Of the four metal chlorides tested for solubility versus temperature, aluminum chloride had the highest solubility, followed by calcium chloride, nickel chloride, and sodium chloride. The solubilities at 373. K (the temperature at which the DETOX^{SM} process solution will be filtered) are approximately 319., 276., 183., and 77. g/L, respectively. All of the four chloride salts' solubilities increased with increasing temperature.

Appendix E – Delphi Document Hierarchy

10/28/97
Rev-9

Delphi Research, Inc.
Document Hierarchy



Appendix F – Process Hazards Review

PROCESS HAZARDS REVIEW REPORT
- PREOPERATIONAL -

Delphi DETOX(sm) Unit

May 7, 1997

REVISION LOG

Revision	Selection(s) Affected	Summary of Revision	Effective Date
0	All	Original Issue	5/7/97

Keywords: Delphi, Wet, Oxidation
Retention: Lifetime

APPROVAL SHEET

Delphi DETOX(sm) Unit
PRE-OPERATIONAL PROCESS HAZARDS REVIEW

APPROVER	SIGNATURE	DATE
C. J. Hampton Review Team Leader	C.J. Hampton	8/16/96
M. A. Fachada Project Manager		5/6/97
D.L. Booth TNX Facility Manager	RE [illegible] for D.L. Booth	5/8/97
E. B. Donner PSM Coordinator	MCF for EB Donner via Telcon	5/8/97

TABLE OF CONTENTS

1.0 INTRODUCTION

This report documents Preoperational Process Hazards Review (PPHR) of process safety hazards associated with the installation and operation of the Delphi DETOX(sm) waste treatment unit demonstration (see Appendix A). The purpose of this PPHR is to ensure that adequate protective measures are in place for all process hazards which may occur during setup and operation of this waste treatment R & D demonstration at TNX test facilities.

1.1 Basis for the Project

In an effort to deal with the increasing quantities of hazardous and mixed waste produced by the DOE complex, approval was given to test and demonstrate the viability of various waste treatment/volume reduction technologies as alternatives to incineration. The Delphi DETOX(sm) system was chosen to be developed to treat mixed waste with a catalyzed wet oxidation treatment process. TNX at SRS was selected as the demonstration site. This project includes installation and operation of the self-contained DETOX(sm) system and personnel in building 673-T as well as the safe operation of the system. Over a 10 month period, various chemical simulants will be used during the demonstration as well as liquid and solid organic hazardous materials produced at SRS. Based on the results of this validation process, the technology will be commercialized and used widely for waste management, pollution prevention, and other environmentally beneficial applications.

1.2 Description of the Delphi DETOX(sm) Demonstration Unit

The demonstration unit consists of several transportable sections that are field connected by process piping and structural connections. Process equipment includes:

- A solid waste hopper and shredder located at the top of the unit for input into the process.
- Primary and secondary reactor vessels of 232 gallons and 162 galllons respectively. An agitator is a part of the primary reactor designed to help ensure proper mixing of organics and the DETOX(sm) solution. Offgases from the primary reactor are routed to the secondary reactor

- A helical filter to remove precipitates/solid residues
- A granular activated carbon bed to remove volatile organic compounds
- HEPA filters to remove particulate
- An overhead distillate receiver tank that feeds to a waste water collection and neutralization tank.
- Process flow pumps

Various spill prevention features include:

- Unflanged vessel bottoms
- Tantalum-lined titanium vessels
- Tantalum, titanium, and TEFLON lined pipes
- Placement of the entire unit in an impermeable containment basin sized to accommodate the total volume of the largest vessel
- Elevated pipes and vessels
- Placement of liquids to be pumped into the primary reactor in the containment basin
- Placement of the process portion of the unit and containment basin in a building to prevent rainfall from contacting and exacerbating a spill
- Placement of spill response kits, equipped specifically for potential DETOX(sm) releases, at strategic locations around the equipment.

A system schematic of the equipment is presented in Appendix A, System Schematic.

1.3 Description of Process Operation

The DETOX(sm) demonstration unit uses wet oxidation to transform complex organic compounds into carbon dioxide and water. Liquids, sludges and solids are introduced to the process via pumps or hoppers. Dry solids are shredded or otherwise rendered suitable for introduction into the system. Wet Oxidation reaction takes place in the pressurized primary reactor 392 F and 110 psig. The oxidation of organics is performed by ferric iron with platinum and ruthenium acting as co-catalysts. Offgases from the primary reactor are routed through a second oxidation reactor that operates at similar conditions of temperature and pressure and provides treatment in a manner similar to the primary reactor. Overheads from the secondary reactor are condensed and captured for storage, neutralization and batch release to the waste water treatment system. Waste water, prior to discharge, is neutralized with aqueous caustic and pH tested. Tail gases are vented to atmosphere after passage through a carbon bed.

1.4 Scope of Review

See Appendix A for a schematic representation of the DETOX(sm) unit. This PPHR includes, but is not limited to, potential process safety hazards associated with unit setup and operations.

2.0 SUMMARY

The possibility of exceeding the following PHR Accident Criteria exists if the Action Items in this report are not implemented:

Criteria
1. Single Fatality/Permanent Injury

A "What-If" method was used to review hazards associated with the DETOX(sm) Unit. Potential hazards were reviewed against the criteria listed in Appendix C. Action items and recommendations have been identified to mitigate hazards. Appendix D details the hazards reviewed by the PHR team. Although not indicated in the Screening Process Hazards Review, one hazard was determined by the PHR team to have the possibility of exceeding the above PHR accident criteria. Unless stated otherwise within an action item, all action items listed in this report must be completed before the DETOX(sm) Unit is placed in service.

The hazards postulated in this process hazards review have the potential of exceeding the following accident criteria:

A. Bodily Injury (fatality)

The following Accident Criteria are not expected to be exceeded and can be omitted:

B.1. Radiation Exposure (500 mrem - Off-plant)
B.2. Radiation Exposure (5 rem - On-plant)
C. Loss of Equipment or Facilities (> $1,000,000 SRS Only)
D. Loss of Production (> six months)
E.1. Toxic material - (IDLH - Off-plant).

E.2. Toxic material - (Emergency Action Level - On-plant).
F. Nuclear Safety

3.0 REVIEW DETAILS

The review method selected for this PPHR was the "What-If" Examination methodology. Action Items were identified for hazards that may exceed SRS Accident Criteria. Recommendations were identified for potential hazards which would not exceed the accident criteria. Details of the hazards and associated Action Items/Recommendations identified by the PPHR teams are presented in Section 3.1.

3.1 DETOX(sm) Demonstration Unit

A PPHR was conducted in June, 1996 on the setup and operation of the DETOX(sm) system at TNX located at SRS. In addition to the PPHR, a Design Hazard and Operability Review was conducted by Delphi Research, Inc., in cooperation with Jacob's Applied Technology - the company responsible for the fabrication of the DETOX(sm) components. Representatives from TNX Operations, Solid Waste, SRTC, EPD, Projects, and Delphi Research formed the PPHR team.

Appendix A of this report provides a schematic diagram representative of the DETOX(sm) Unit.

The Preoperational PHR review team identified the following hazards, and subsequent action items and recommendations. What-If questions that are associated with the action items or recommendations are grouped and discussed for each hazard (see Appendix D for a complete summary of all What-Ifs considered). Action items and recommendations are intended to suggest functions which, when operational, will satisfy the action items and recommendations. The manner of the solution is not detailed here. It is left up to the responsible organization to find the best and most cost effective way to perform the function. Also, where there are action items and recommendations which show more than one group responsible, the recommendations and action items may not apply to all parties equally. The hazards are summarized in the following table:

HAZARDS	CRITERIA EXCEEDED	ACTION ITEMS	RECOMMENDATIONS
1. Hazardous Chemicals	A1	DD-A1	DD-R1, DD-R2
2. Chemical Splash	None	None	DD-R3
3. Steam	None	None	DD-R4, DD-R5
4. Lightning	None	None	DD-R6
5. Fire	None	None	DD-R7
6. Grounding	None	None	DD-R8
7. Lack of Communication	None	None	DD-R9,R10,R11,R12
8. Darkness	None	None	DD-R13, DD-R14

Hazard #1: **Hazardous Chemicals**
What-If #1: Chemicals mix to form a non-evaluated chemical?

Most of the chemicals to be used during the 10 months of testing will be stored in the TNX chemical storage facility. Chemicals already in storage at TNX could potentially mix with the Delphi stored chemicals and form a new chemical. The effect of this new chemical would be unevaluated and could potentially cause a vapor cloud or other hazard that could lead to a fatality.

Action Items

(DD-A1) Ensure that the list of chemicals to be used during the DETOX(sm) demonstration is evaluated prior to new chemical additions to the existing chemical inventory.

What-If #2: Process chemicals spill producing a harmful vapor or gas?

According to the chemical inventory provided by Delphi Research, a potential hazard was identified in an accident involving process chemicals, especially HCL that could cause personnel injury if a vapor cloud were to form and spread throughout the process building.

Recommendations

(DD-R1) WSRC to provide spill kit to Delphi operators.
(DD-R2) Evaluate building ventilation requirements.

Hazard #2: Chemical Splash

What-If #3: There is low flow or low pressure on DETOX(sm) skid safety showers?

One section of the DETOX(sm) Unit has three levels with a height of 30 ft. Water supply for the unit will be provided by TNX. One safety shower is built into the skid on each level. If water supply provided is not sufficient, safety showers could have low flow or pressure and could be ineffective in the event of an emergency in which an operator does not have time to come down to a lower level.

Recommendation

(**DD-R3**) Ensure that sufficient flow and pressure is available and tested.

Hazard #3: Steam

What-If #4: A steam leak develops?

Process piping connects the various systems of the DETOX(sm) Unit. Steam for the unit is provided by TNX with the existing connections to 673-T. Although the process piping is insulated and very little steam is used in the process, an accident scenario could involve a steam leak near on one of the upper levels of the skid; creating a situation where an operator may not be able to prevent steam loss and personnel injury.

Recommendations

(**DD-R4**) Ensure that remote steam shut-off is available.
(**DD-R5**) WSRC to provide steam trap and drain.

Hazard #4: Lightning

What-If #5: Lightning strikes the process building or exterior equipment?

Although taller buildings located in TNX have lightning suppression devices, 673-T does not, leaving an opportunity for an accident. Outside equipment, such as an oil skid/heat exchanger and other piping might also be in danger of a lightning strike that could cause injury and equipment damage.

Recommendation

(**DD-R6**) Have technical experts determine the need for lightning suppression for building and outside components.

Hazard #5: Fire

What-If #6: Fire starts anywhere in the process building?

A potential for fire requires sufficient fire suppression devices and emergency response opportunities. Personnel injury and equipment damage could result from any fire ignited by the process or any other source.

Recommendation

(**DD-R7**) Perform Fire Hazards Assessment and respond accordingly

Hazard #6: Grounding

What-If #7: Insufficient grounding exists causing a voltage potential between the skid and the process building?

Originally identified during the Delphi Research/Jacob's Applied Engineering Design Hazard and Operability Review, it was left to WSRC to provide adequate grounding for the building in which the DETOX(sm) unit would be demonstrated. Proper grounding for the skid itself is resolved as recommended by the previously mentioned Design Hazard and Operability Review.

Recommendation

(**DD-R8**) WSRC responsible for adequate grounding

Hazard #7: Lack of Communication

What-If #8: A situation arises requiring interaction with emergency personnel for evacuation, shelter, or accountability?

Within 673-T, the DETOX(sm) Unit will by housed along with SRTC R&D Activities . Delphi operators will conduct the DETOX(sm) demonstration from a control trailer located outside the process building. Besides a P.A. system present in 673-T, there is no other existing form of communication/interaction between the Delphi operators and other TNX personnel. In the event of an emergency involving the DETOX(sm) demonstration, the additional TNX project housed in 673-T, or any other location on site, personnel injury could occur if proper action is not taken in response to situations requiring evacuation, shelter protection and accountability.

Recommendations

(**DD-R9**) Require Delphi operators to attend TNX Ops. meetings and brief them on TNX projects housed within or near 673-T (and vice-versa).
(**DD-R10**) Provide radios, phones and more P.A. speakers

What-If #9: A situation arises requiring immediate action between the control trailer and the unit?

This accident scenario is similar to the one described in the previous "What If" Situation. With the DETOX(sm) control trailer located in a different room than the process equipment and there being no "hard" communication connecting the two, there is the potential for a situation to arise causing injury or equipment damage due to a lack of interface.

Recommendation

(**DD-R11**) Ensure battery powered egress lighting
(**DD-R12**) Require Delphi operators to carry flashlights

Hazard #8: Darkness

What-If #10: There is a loss of electrical power and personnel cannot find egress?

A loss of electrical power invites personnel injury due to a lack of emergency lighting within 673-T. Descent from an upper level of the skid could be hazardous.

Recommendation

(DD-R13) Same as (DD-R10)

(DD-14) Conduct a readiness review prior to the start of testing

4.0 NEXT REVIEW DATE

The process hazards discussed in this report are associated with the Delphi DETOX(sm) Unit. Due to the limited demonstration time of this system, a review of this PHR will only be necessary in the event of an incident investigation at the request of the Principal Investigation or facility manager.

5.0 CONTRIBUTORS

The following hazard review team members participated in this PPHR and contributed to the preparation of the report:

Team Members	Organization	
Russ Ferrara	TNX Ops	
Marie Berry	TNX Ops	
Dave Boyce	TNX Ops	
Edmund B. Donner	SWE	
Jermaine Hampton	SWE	Team Leader
Jeff Siler	SRTC	
Lynn Martin	EPD	
Mark Fachada	SW PROJECTS	
Frank Lustig	PROJECTS	
Scott Goldblatt	DELPHI RESEARCH	
Scott Anderson	ROCKY FLATS	

6.0 REFERENCES

1. Process hazards review manual

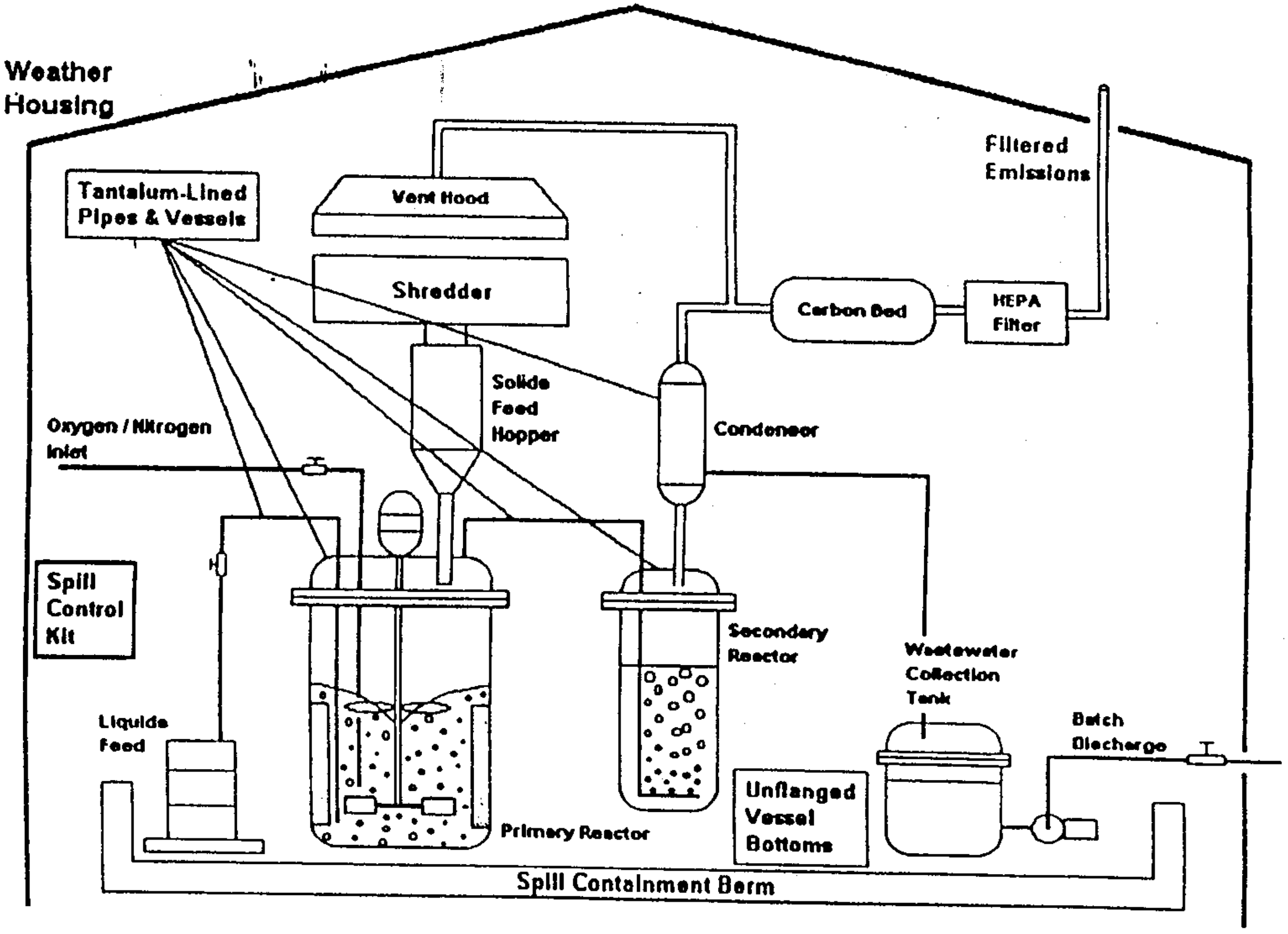
7.0 APPENDICES

7.1	Appendix A	Delphi DETOX(sm) System Schematic
7.2	Appendix B	Delphi Design Hazard and Operability Review
7.3	Appendix C	Preoperational Process Hazards Review Criteria
7.4	Appendix D	"WHAT-IF" Examination Record

APPENDIX A

DELPHI DETOX(sm) UNIT
SYSTEM SCHEMATIC

DETOX^{SM} System Schematic



APPENDIX B

Delphi Research
Design Hazard and Operability Review

COMPANY: Delphi Research, Inc. -- Proprietary Information
PROJECT: METC Wet Oxidation Project
SYSTEM NUMBER: N/A
SYSTEM NAME: GENERAL NOTES
P&ID VARIOUS
COLOR N/A

DESIGN HAZARD AND OPERABILITY REVIEW

REVISION 3
9-Aug-95

PAGE ____ OF ____

ITEM NO	RECOMMENDED ACTION	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND. NUMBER	DATE IMPLEMENTED
1	CONFIRM THE FLANGE RATING OF JACKETED PIPE SPECS TJ1 AND TJ2 - THE PIPING SERVICE INDEX SHOWS 150# WITH A MAX T&P OF 450 DEGF AND 300 PSIG.	YES	8/23/94	G001	4/20/95
2	CHANGE ONCE THROUGH COOLERS TO CHILLED OIL / WATER SYSTEM TO REDUCE PROCESS / CITY WATER USAGE.	YES	8/23/94	G002	4/20/95
3	CONFIRM THAT ALL ELECTRICAL EQUIPMENT CONFORMS TO AREA CLASSIFICATION CL I, DIV 2, GR D CONFIRM THAT GROUP C IS NOT NEEDED.	NO - STAY W/ C&D			
4	IN ORDER TO RESOLVE ALL AIR / RCRA / WATER / NEPA ISSUES - BASIC HEAT/MATERIAL BALANCE AND EMISSIONS CALCULATIONS SHOULD BE SENT TO DOE EARLY IN PHASE III	YES	8/23/94	G004	BY DRI
5	RESOLVE FIRE PROTECTION REQUIREMENTS.	YES	8/23/94	G005	DEFER TO SRS PHA
6	DESIGN SHOULD INCLUDE GROUNDING ALL EQUIPMENT AND PIPING	YES	8/23/94	G006	JAT - DETAIL DESIGN
7	DETERMINE WHAT TYPE OF INTERLOCK SYSTEM WILL BE USED (HARDWIRED, PLC, ETC)	YES - GO WITH PLC	8/23/94	G007	4/20/95
8	ADD KNOCK-OUT POT FOR ALL PSV OUTLETS.	YES	8/23/94	G008	4/20/95
9	CONSIDER DOE RADCON REGULATIONS DURING DESIGN.	YES	8/23/94	G009	NONE REQ'D PER WSSRAP
10	INVESTIGATE SITE-SPECIFIC RADIOACTIVE EXPOSURE REQUIREMENTS FOR WELDON SPRINGS.	YES	8/23/94	G010	NONE REQ'D PER WSSRAP
11	ADD SAMPLE POINTS ON DISCHARGE OF ALL PROCESS PUMPS	YES	8/23/94	G011	4/20/95
12	RESOLVE WHETHER UNIT WILL BE INSIDE OR OUTSIDE ~~AT WELDON SPRINGS~~ - IMPACT ON DESIGN COULD BE SIGNIFICANT CURRENT DESIGN BASIS IS THAT THE UNIT WILL BE INSIDE	YES - OUTDOOR DESIGN	8/23/94	G012	JAT - DETAIL DESIGN
13	PROVIDE LOCAL HAND-OFF-AUTO (HOA) SWITCHES FOR ALL MOTORS ~~ON PROCESS EQUIPMENT~~.	YES	8/23/94	G013	8/2/95
14	DESIGN TO PROVIDE BREAKOUT FLANGES AROUND ALL VESSELS WITH REMOVABLE HEADS.	YES	8/23/94	G014	JAT - DETAIL

COMPANY Delphi Research, Inc. -- Proprietary Information
PROJECT. METC Wet Oxidation Project
SYSTEM NUMBER. N/A
SYSTEM NAME: GENERAL NOTES
P&ID: VARIOUS
COLOR: N/A

DESIGN HAZARD AND OPERABILITY REVIEW

REVISION 3
9-Aug-95

PAGE ____ OF ____

ITEM NO	RECOMMENDED ACTION	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND. NUMBER	DATE IMPLEMENTED
					DESIGN
15	INVESTIGATE LIGHTNING PROTECTION / SUPPRESSION REQUIRMENTS.	YES	8/23/94	G015	DEFER TO SRS PHA
16	TRACE AND INSULATE WATER AND LIQUID ORGANIC SYSTEMS TO 40 DEG F FOR FREEZE PROTECTION	YES	8/23/94	G016	4/20/95
17	TRACE AND INSULATE DETOX SYSTEMS TO 50 DEG C FOR FREEZE PROTECTION.	YES	8/23/94	G017	4/20/95
18	ADD SAFETY SHOWERS AND EYEWASH STATIONS TO EACH OPERATING LEVEL.	YES	8/23/94	G018	4/20/95

COMPANY Delphi Research, Inc. -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 1
SYSTEM NAME LIQUID ORGANIC FEED FROM DRUM TO REACTOR
P&ID- D-2000 # D-2001
COLOR BLUE

DESIGN HAZARD AND OPERABILITY REVIEW

REVISION [illegible]
9 Aug 95
PAGE ___ OF ___

ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/ SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND. NUMBER	DATE IMPLEMENTED
1.1	NO. Flow		PUMP FAILURE. VALVES CLOSED. PSV OPEN. DRUM EMPTY. PLUGGED STRAINER.	REACTOR UPSET LOSS OF PRODUCTION VAPOR LOCK. LINE RUPTURE	RUN LIGHT ON PUMP. PSV ON PUMP DISCHARGE	/					
			RUPTURED PIPE OR DRUM.	PERSONNEL EXPOSURE FIRE HAZARD ENVIRONMENTAL EXPOSURE	PSV ON PUMP. PSV ON DRUM	CONFIRM SKID WILL BE IN DIKED AREA		YES	8/23/94	C019	4/20/95 BY DRI
			DRUM CHANGEOUT.	SEE ABOVE		ADD STANDBY DRUM STATION FOR USE DURING DRUM CHANGEOUT		NO		---	
						SOP TO SAFELY CHANGEOUT DRUM		YES	8/23/94	P001	SOP
	MORE Flow		CONTROLS FAILURE. OPERATOR ERROR. PUMP MALFUNCTION	HIGH REACTION RATE INCOMPLETE RXN CARRYOVER OF ORGANICS TO OVHD SYSTEM INCREASE REACTOR LEVEL. INCREASE TEMPERATURE IN RX HIGH VOC IN REACTOR OVHD HIGH RXN PRESSURE	TEMP AND PRESSURE CONTROL ON RX WEIGH CELLS ON RX CARBON BED ON VENT LINE O2 AND CO2 ANALYZERS ON VENT LINE						
	LESS Flow		SEE NO FLOW ABOVE								
	REVERSE Flow		LINE RUPTURE FAILED CHECK VALVE AND PUMP	ENVIRONMENTAL AND PERSONNEL EXPOSURE DETOX TO DRUM. DAMAGE TO PUMP ENVIRONMENTAL & PERSONNEL EXPOSURE	CHECK VALVE ON RX FEED LINE EMERGENCY SHUTOFF VALVES AT REACTOR PIPE DESIGNED TO HANDLE MAX PUMP PRESSURE	ADD ANTI-SIPHON HOLE TO DIP TUBE.		YES	8/23/94	C020	4/20/95
						ADD TEMP, PRESSURE, OR CONDUCTIVITY MEASUREMENT TO FEED LINE.		YES - TEMPERATURE	8/23/94	C021	4/20/95
						INTERLOCK SHUTOFF VALVES TO OPEN/CLOSE WHEN PUMP IS ON/OFF		YES	8/23/94	I001	INTERLOCK
						ADD MANUAL SHUTOFF VALVE AT REACTOR		NO			
	REVERSE Flow		DURING STARTUP BACKFLOW OF DETOX	SEE REVERSE FLOW ABOVE		SOP TO CONFIRM PUMP PRESSURED BEFORE OPENING SHUTDOWN VALVES		NO	--	--	
1.2	MORE Temp		SEE REVERSE FLOW ABOVE								
			DEADHEADED PUMP	PUMP DAMAGE - CONSIDERED UNLIKELY							
			HIGH AMBIENT TEMP	NO HAZARDS							
	LESS Temp		LOW AMBIENT TEMP	FREEZE LINE - SEE NO FLOW ABOVE		DESIGN TO ALLOW SPACE FOR FIELD TRACING AT A LATER TIME		NO	--	--	
						FIELD TRACE TO 40 DEGF		YES	8/23/94	C016	4/20/95
1.3	MORE Press		SEE REVERSE FLOW ABOVE.								
	LESS Press		SEE MORE / NO FLOW ABOVE								
	REVERSE Press	DRUM	NO N2 FLOW TO DRUM.	COLLAPSE DRUM		ADD VACUUM RELIEF PROTECTION ON DRUM		YES	8/23/94	C022	4/20/95
						INTERLOCK PUMP ON LOW N2 PRESSURE		NO	--	---	
		PIPING	PLUGGED PUMP SUCTION. CLOSED VALVE ON PUMP SUCTION	CAVITATE PUMP - SEE NO FLOW ABOVE	PIPING WILL HANDLE VACUUM WITHOUT COLLAPSING						
1.4	NO Level	DRUM	DRUM EMPTY	PUMP N2 TO REACTOR POTENTIAL BACKFLOW FROM REACTOR LOSS OF FEED TO REACTOR		ADD LEVEL MEASUREMENT TO DRUM		NO			
						ADD SCALES WITH MASS FLOW CALC BLOCK - DRUM TO BE VERTICAL WITH DIP TUBE AND FOOT VALVE		YES	8/23/94	C023	4/20/95
						ADD FLOWMETER OR SWITCH TO FEED LINE		YES - ADD FLOW SWITCH	8/23/94	C024	4/20/95
						SOP TO COVER HOW EMPTY DRUMS AND HEELS ARE TO BE HANDLED		YES	8/23/94	P002	SOP
						ADD FLOW CALCULATION BLOCK TO PUMP SPEED		YES	8/23/94	C025	4/20/95
						ADD FEED SURGE DRUM		NO	-	--	
						PLACE DRUM STAION AT EDGE OF MODULE SO AN OFF-MODULE DAY TANK CAN BE ADDED LATER		YES	8/23/94	C026	4/20/95
	MORE Level	DRUM	SEE REVERSE FLOW ABOVE								
	LESS Level	DRUM	SEE NO LEVEL ABOVE								
1.5	AS WELL AS Comp	WATER	WATER IN DRUM	UNIT UPSET		DESIGN REACTOR OVERHEAD SYSTEM FOR 100% WATER FEED		OK - NORM FLOW EXCEEDS FEED RATE	8/23/94	C027	4/20/95
		SLUDGE	SLUDGE IN DRUM	PLUGGED STRAINER POSSIBLE PUMP DAMAGE	SEE NO FLOW ABOVE	IF FEED SURGE DRUM ADDED PROVIDE SIDE FEED DRAW AND SAMPLE POINTS		NO	--	---	
		DISSOLVED METALS	DISSOLVED RCRA METALS IN DRUM (Hg, Ba, Pb, Cr, etc.)	NO HAZARDS IDENTIFIED							
	OTHER THAN Comp	WATER	IMMISCIBLE WATER LAYER IN	SUDDEN CHANGE OF FEED FROM ORGANIC	TEMPERATURE AND PRESSURE CONTROL	ADD FLOWMETER TO FEED LINE OR FLOW		YES - ADD FLOW SWITCH	8/23/94	C024	4/20/95

COMPANY	Delphi Research, Inc — Proprietary Information
PROJECT	METC Wet Oxidation Project
SYSTEM NUMBER	I
SYSTEM NAME	LIQUID ORGANIC FEED FROM DRUM TO REACTOR
P&ID	D-2000 // D-2001
COLOR	BLUE

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			DRUM.	TO WATER (OR VICE VERSA) TEMP DROPS IN REACTOR	ON REACTOR	SWITCH					
						ADD SAMPLE PORT ON LIQUID FEED LINE		YES - ON PUMP DISCHARGE	8/23/94	C011	4/20/95
		FLUOROCARBON GREASE	IMPROPER FEED SCREENING OPERATOR ERROR.	THIS MATERIAL WILL NOT BE OXIDIZED IN T REACTOR - FILL UP REACTOR, UNIT UPSET.	LOAD CELLS ON REACTOR O2 & CO2 ANALYZERS ON VENT STREAM	SOP TO COVER SAFE CLEANOUT OF EQUIPMENT		YES	8/23/94	P005	SOP
16	MORE Viscosity		SEE ABOVE.								
	LESS Viscosity		SEE ABOVE								
17	IGNITION SOURCE		SPARKS FROM MOTOR(S).		ALL MOTORS SPECIFIED FOR FOR CLASS I DIV2 GROUP D						
			STATIC SPARKS FROM DRUMS	POSSIBLE FIRE		ALL EQUIPMENT AND PIPING TO BE GROUNDED		YES	8/23/94	C006	(AT) [illegible]
						USE NON SPARKING DRUM CONNECTIONS IN DESIGN		YES	8/23/94	C028	4/20/95

COMPANY Delphi Research, Inc. -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 2
SYSTEM NAME SOLID ORGANIC FEED FROM DRUM TO REACTOR
P&ID D-2000 # D-2001 # D-2004
COLOR GREEN

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ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND NUMBER	DATE IMPLEMENTED
2.1	NO Flow	SOLID FEED	SHREDDER OR PUMP PLUGGED PLUGGED PIPING. VALVE CLOSED. LOSS OF WATER TO V-001. HOPPER BRIDGED OVER. CONTROLS FAILURE.	LOSS OF PRODUCTION REACTOR UPSET	LINE IS COOLED AT R-101 TO REDUCE POTENTIAL OF PLASTICS PLUGGING	ADD FLUSH OR ROD OUT PORTS TO FEED LINE		YES - ADD ROD OUT PORTS	8/31/94	C029	4/20/95
						ADD SOFTWARE BLOCK TO RUN P-002 ON MANUAL WHILE FEEDING LIQUID ORGANICS. THIS IS NEEDED TO ADD IRON OXIDE TO REACTOR.		YES	8/31/94	C030	4/20/95
				DEADHEAD PUMP	FLOW METER ON FEED LINE	ADD RELIEF OR TORQUE LIMITER TO PUMP - SET AT 125 PSIG DISCHARGE PRESSURE		YES	8/31/94	C031	4/20/95
			RUPTURED LINE	POSSIBLE VAPOR RELEASE FROM R-101	EMERGENCY SHUTOFF VALVES AT R-101 PRESSURE TRANSMITTER ON R-101	ADD AREA HCl MONITOR		YES	8/31/94	C012	4/20/95
						ADD PRESSURE, TEMP, CONDUCTIVITY MEASUREMENT TO FEED LINE		YES - TEMPERATURE	8/31/94	C013	4/20/95
						ADD SAMPLING TO FEED LINE TO CONFIRM PARTICLE SIZES		YES - ON PUMP DISCHARGE	8/31/94	C011	4/20/95
						CONFIRM LINE SIZING FOR 1/2" SOLIDS		YES - (ALL LINE SIZES ARE PRELIMINARY)	8/31/94	C034	8/2/95
						ADD ISOLATION VALVE BETWEEN PUMP AND HOPPER FOR MAINTENANCE		YES - CONSULT WITH VENDORS FOR OPTIONS. MAY USE IRIS OR FLANGIATE TYPE VALVE	8/31/94	C035	8/2/95
						ADD MANUAL VALVE AT R-101 FEED LINE FOR MAINTENANCE		NO	--	--	
		VENT BLOWER	BLOWER FAILURE CARBON BED PLUGGED. VALVE CLOSED HEPA FILTER PLUGGED	PERSONNEL & ENVIRONMENTAL EXPOSURE CONTAMINATE EQUIPMENT WITH DUSTS AN FUMES	LOCAL PRESSURE INDICATOR AT FAN HIGH PRESSURE SWITCH ON FAN SUCTION RUN INDICATION ON FAN PRESSURE INDICATION ON HEPA FILTER INLET	ADD PRESSURE/DP TRANSMITTERS ACROSS CARBON BED AND HEPA FILTER		YES - LOCAL	8/31/94	C016	4/20/95
						CONFIRM HEPA FILTER HAS DOP TEST PORTS		YES	8/31/94	C017	8/2/95
	MORE Flow	SOLIDS FEED	OPERATOR ERROR. CONTROLS FAILURE. PUMP MALFUNCTION	HIGH REACTION RATE INCOMPLETE RXN CARRYOVER OF ORGANICS TO OVHD SYSTEM INCREASE REACTOR LEVEL INCREASE TEMPERATURE IN RX HIGH VOC IN REACTOR OVHD HIGH RXN PRESSURE	FLOWMETER ON FEED LINE WITH HIGH ALARM CO2 & O2 SENSORS ON VENT REACTOR TEMP & PRESS INDICATORS ON R-101						
		VENT SYSTEM	DAMPER OPEN	POSSIBLY TRIP FAN	PRESSURE INDICATORS AT FAN FAN RUN LIGHT	SOP TO VERIFY ADEQUATE VENT VELOCITY		YES	8/31/94	F004	SOP
						ADD ADDITIONAL CARBON BED IN SERIES		NOT ON THIS SYSTEM	--	--	
						ADD ROTOMETERS ON VENT INLET LINES		YES	8/31/94	C018	4/20/95
						MANIFOLD AE-404 TO ANALYZE OUTLET FROM EITHER CARBON BED		YES	8/31/94	C019	4/20/95
	LESS Flow		SEE NO FLOW ABOVE								
	REVERSE Flow	SOLIDS FEED	BACKFLOW THROUGH P-001. PUMP OFF.	HCl BACKUP IN SYSTEM CORROSION PERSONNEL & ENVIRONMENTAL EXPOSURE	DOUBLE BLOCK VALVES AT R-101. VENT SYSTEM IN PLACE						
		VENT SYSTEM	NO CAUSES IDENTIFIED								
2.2	MORE Temp	SOLIDS FEED	HEAT CONDUCTION FROM REACTOR.	PLUGGED LINE	COOLER ON FEED LINE FLOWMETER ON FEED LINE	SEE ABOVE					
			DEADHEADED PUMP P-002.	PLUGGED PUMP PUMP DAMAGE	FLOW INDICATOR ON FEED LINE	ADD INTERLOCK TO TURN OFF P-002 ON LOW FLOW		NO	--	--	
						INTERLOCK SHUTOFF VALVES TO OPEN/CLOSE WHEN PUMP IS ON/OFF, AND VICE VERSA		YES	8/31/94	I002	INTERLOCK
						ADD LIMIT SWITCHES TO CONFIRM OPEN ON TWO SHUTOFF VALVES		NO		--	
		VENT SYSTEM	FIRE IN SHREDDER	ENVIRONMENTAL & PERSONNEL EXPOSURE		ADD CONTROLS TO TURN WATER SPRAY ON/OFF WHEN SHREDDER IS ON/OFF		YES	8/31/94	I005	4/20/95
	LESS Temp	SOLIDS FEED	LOW AMBIENT TEMPERATURE	FROZEN WATER LINES		DESIGN TO ALLOW SPACE FOR HEAT TRACING AT A LATER TIME		NO	--	--	
						HEAT TRACE TO 40 DEG F		YES	8/31/94	C016	4/20/95
		VENT SYSTEM	NO CAUSES IDENTIFIED								
2.3	MORE Press	SOLID FEED	SEE NO FLOW ABOVE								
		VENT SYSTEM	PLUGGED CARBON BED AND/OR HEPA FILTER.	LOSS OF VENTILATION PERSONNEL & ENVIRONMENTAL EXPOSURE		SOP OR INTERLOCK TO SHUT DOWN SHREDDER ON HIGH DP ACROSS HEPA FILTER		YES - SOP	8/31/94	F005	SOP
						INSTALL SECOND CARBON BED TO OPERATE AS LEAD LAG - WILL ALLOW BYPASSING A PLUGGED BED		NOT ON THIS SYSTEM	--	--	
	LESS Press	SOLID FEED	SEE NO FLOW ABOVE								

COMPANY Delphi Research, Inc. -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 2
SYSTEM NAME SOLID ORGANIC FEED FROM DRUM TO REACTOR
P&ID D-2000 # D-2001 # D-2004
COLOR GREEN

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		VENT SYSTEM	SEE NO FLOW & MORE FLOW ABOVE			/					
	REVERSE Press	SOLID FEED	NO CAUSES IDENTIFIED								
		VENT SYSTEM	CLOSED DAMPERS		PLASTIC PIPE CAN HANDLE VACUUM						
14	NO Level	V-001	CONTROLS MALFUNCTION OPERATOR ERROR.	SEE NO FLOW ABOVE		CONFIRM P-003 CAN RUN DRY		YES	8/23/94	[illegible]	[illegible]
	MORE Level	V-001	WATER VALVE LEFT OPEN FAILED INSTRUMENTATION OPERATOR ERROR PUMP NOT RUNNING	BACK UP WATER IN SHREDDER		ADD HIGH HIGH LEVEL SWITCH TO V-001		YES	8/23/94	[illegible]	[illegible]
						ADD OVERFLOW LINE FROM V-001 TO SAFE LOCATION		NO			
15	MORE Comp	SOLID FEED		NO HAZARDS IDENTIFIED							
	AS WELL AS. Comp	VENT SYSTEM	WATER VAPORS IN VENT STREAM	POSSIBLY PLUG HEPA FILTER		ADD HEATER UPSTREAM OF HEPA FILTER TO PREVENT WATER PLUGGING		NO			
						ALLOW SPACE FOR FUTURE HEATER		YES	8/23/94	[illegible]	[illegible]
						ADD LOW POINT DRAIN AND SIGHT GLASS TO T-401		YES	8/23/94	[illegible]	[illegible]
16	NO Agitation		MOTOR STOPS. IMPELLER FALLS OFF.	SOLIDS SETTLING OR SEPARATING IN HOPPER UNIT UPSET SEE NO FLOW ABOVE		ADD INTERLOCK TO CONFIRM THAT AGITATOR IS RUNNING BEFORE STARTING P-003 OR M-001		YES	8/23/94	[illegible]	INTERLOCK.
	MORE Agitation		NO CAUSES IDENTIFIED								
	REVERSE Agitation			NO HAZARDS IDENTIFIED							
17	IGNITION SOURCE		SPARKS FROM MOTOR(S).		ALL MOTORS SPECIFIED FOR FOR CLASS I DIV2 GROUP C & D						
18	HEAT EXCHANGER LEAKS	COOLING WATER TO SOLIDS FEED LINE COOLER	RUPTURED PROCESS PIPE.	POSSIBLE PIPE PLUGGAGE LOSS OF COOLING SEE NO FLOW ABOVE	SEE NO FLOW ABOVE.						
19	EROSION/CORROSION		ABRASIVE SOLIDS IN FEED	PUMP WEAR / DAMAGE	REPLACABLE LINER & ROTOR						
				SHREDDER DAMAGE	SHREDDER DESIGNED FOR HARSH SERVICE AND HAS REPLACABLE PARTS						
				AGITATOR DAMAGE - SEE NO AGITATION ABOVE							
				PIPING DAMAGE	PIPING CAN BE REPLACED						

COMPANY Delphi Research, Inc. – Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 3
SYSTEM NAME QUENCH WATER FROM TANK TO REACTOR
P&ID D-3000 # D-3001
COLOR PINK

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3.1	NO Flow WHEN WE WANT IT		INSUFFICIENT N2 PRESSURE BLOCK VALVE CLOSED. CONTROLS FAILURE LINE RUPTURE TANK EMPTY DRAIN VALVE OPEN	UNCONTROLLED REACTION OVERPRESSURE REACTOR TEMPERATURE RISE IN R-101	LOW LOW SWITCH ON V-002 PRESSURE TRANSMITTER ON V-002 PRESSURE REGULATOR ON N2 PT ON R-101 OVERHEAD BULL PLUG IN DRAIN VALVE	ADD REDUNDANT QUENCH LINE FOR R-101 WITH BLOCK VALVE. REDUNDANT VALVES COULD BE AUTO OR CHAINED.		YES - AUTOMATIC	8/23/94	C044	4/20/95
						CONSIDER PRESSURING WATER IN V-002 FROM R-101 PRESSURE		YES - INVESTIGATE	8/23/94	C045	4/20/95
	MORE Flow WHEN WE WANT IT		HIGHER N2 PRESSURE HIGHER AMBIENT TEMP. LOW REACTOR PRESSURE.	OVERLOAD REACTOR OVERHEAD SYSTEM	PRESSURE CONTROL VALVE ON R-101	JACOBS TO DESIGN OVERHEAD SYSTEM TO HANDLE ADDITIONAL VAPOR LOADING DURING QUENCH		YES	8/23/94	C046	KEITH BY 1 MAY
						ADD INTERLOCK TO BYPASS R-102 WHILE QUENCHING TO REDUCE CHANCES OF ENTRAINING DETOX SOLUTION		YES	8/2/95	1205	
				OVERPRESSURE QUENCH TANK.	PT ON V-002 W/ LOW ALARM LOCAL PI ON V-002. PSV ON V-002						
	MORE Flow WHEN WE DON'T WANT IT		OPERATOR ERROR. CONTROL VALVE FAILURE.	REACTOR UPSET. LOSS OF LEVEL IN V-002		REPLACE LEVEL SWITCHES ON V-002 WITH LEVEL TRANSMITTER AND LOCAL INDICATOR		YES	8/23/94	C047	4/20/95
						ADD INTERMEDIATE LEVEL SWITCH		NO	---	---	
	LESS Flow		SEE NO FLOW ABOVE								
	REVERSE Flow	QUENCH WATER	LOW N2 PRESSURE AND VALVE OPENS UNDERSIZED N2 REGULATOR ON V-002	NO COOLING FOR R-101 HOT CORROSIVE VAPORS IN V-002	PT ON V-002	ADD CHECK VALVE IN QUENCH LINE		N/A NEEDED SINCE REACTOR PRESSURE WILL BE USED TO ADD QUENCH WATER	-	---	
						CONFIRM SIZE OF V-002 N2 REGULATOR		NOT NEEDED SINCE REACTOR PRESSURE WILL BE USED TO ADD QUENCH WATER	--	—	
			RELIEF VALVE ON V-002 OPEN AND LOW LEVEL IN V-002 DURING QUENCH	POSSIBLE HCl RELEASE - CONSIDERED UNLIKELY.		ADD AREA HCl MONITOR		YES	8/23/94	C033	4/20/95
		NITROGEN	LOSS OF N2 PRESSURE LINE RUPTURE	LOSS OF QUENCH TO R-101. VENT N2 INTO ROOM POTENTIAL HCl RELEASE	PT ON V-002 N2 PIPING DESIGNED FOR HIGHER PRESSURE THAN NORMAL OPERATING	ADD AREA HCl MONITOR		YES	8/23/94	C032	4/20/95
		PROCESS H2O	OPERATOR ERROR CHECK VALVE FAILURE AND	POSSIBLE LINE RUPTURE OVERPRESSURE PROCESS	DOUBLE BLOCK AND CHECK BLOCK VALVES NORMALLY	CONSIDER STANDBY N2 ON V-002 - OPERATE V-002 AT LOW PRESSURE		N/A NEEDED SINCE REACTOR PRESSURE WILL BE USED TO ADD QUENCH WATER	---	---	
			VALVES OPEN ATTEMPTING TO FILL V-002 UNDER PRESSURE	H2O SYSTEM	CLOSED	ADD BLEED VALVE AT CHECK VALVE TO CREATE A DOUBLE BLOCK AND BLEED		YES	8/23/94	C048	4/20/95
	MORE Flow	NITROGEN	LEAKING PSV - LEAKS N2 TO ATM	LOSS OF NITROGEN PRESSURE.	PRESSURE TRANSMITTER AND LOCAL INDICATOR ON QUENCH TANK. PI ON V-001	REPLACE PSV WITH RUPTURE DISK - IT SHOULD BE RATED FOR USE WITH WATER		YES - SET AT SET PRESSURE OF REACTOR RUPTURE DISK	8/23/94	C049	4/20/95
3.2	MORE Temp	QUENCH H2O	BACKFLOW FROM REACTOR	SEE REVERSE FLOW ABOVE							
			FIRE	OVERPRESSURE V-002	PSV ON V-002						
	LESS Temp	QUENCH H2O AND PROCESS WATER	LOW AMBIENT TEMPERATURE.	LINE OR TANK FREEZES LOSS OF QUENCH WATER		DESIGN TO ALLOW SPACE FOR FIELD TRACING AT A LATER TIME		NO	---	—	
						FIELD TRACE TO 40 DEG F		YES	8/23/94	C016	4/20/95
3.3	MORE Press	V-002	INCREASE IN TANK AFTER FILLING - BY CONDUCTIVE HEAT FROM RX OR FROM HIGHER AMBIENT TEMPERATURE.	THERMAL EXPANSION OF N2 IN HEADSPACE - POSSIBLY OVERPRESSURE TANK	PSV ON V-002 PT ON V-002	SET PSV SET PRESSURE TO 300 PSIG NOTE THAT OPERATING PRESSURE IS 250 PSIG		NO		---	
	LESS Press		SEE ABOVE								
	REVERSE Press	V-002	TANK COOLS AFTER FILLING UNDERSIZED REGULATOR DURING QUENCH LOSS OF N2 DURING QUENCH	FULL VACUUM IN TANK	V-002 IS RATED FOR FULL VACUUM						
3.4	NO Level		SEE NO FLOW ABOVE								
	MORE Level		OPERATOR ERROR LEVEL SWITCH FAILURE	OVERFLOW TANK - SPILL WATER	HIGH LEVEL ALARM	SOP TO COVER PROPER FILLING PROCEDURE		YES	8/23/94	P006	SOP
						REPLACE LEVEL SWITCHES ON V-002 WITH LEVEL TRANSMITTER AND LOCAL INDICATOR		YES	8/23/94	C047	4/20/95

COMPANY Delphi Research, Inc -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 3
SYSTEM NAME QUENCH WATER FROM TANK TO REACTOR
P&ID D-2000 # D-2001
COLOR PINK

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ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/ SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND NUMBER	DATE IMPLEMENTED
	LESS Level		SEE NO FLOW ABOVE								
15	OTHER THAN Comp	HCl	SEE REVERSE FLOW ABOVE.								
	OTHER THAN Comp	NITROGEN	NO LEVEL IN QUENCH TANK. VALVE FAILURE. OPERATOR ERROR. INSTRUMENT FAILURE.	POSSIBLY OVERLOAD REACTOR OVERHEAD VAPOR SYSTEM	RUPTURE DISK ON REACTOR	CONFIRM SIZING OF RX OVHD SYSTEM CAN HANDLE FULL FLOW FROM N2 REGULATOR ON QUENCH TANK.		N/A NEEDED SINCE REACTOR PRESSURE WILL BE USED TO ADD QUENCH WATER	--	--	
	AS WELL AS Comp	BIOLOGICAL GROWTH	BUGS IN WATER	ORGANICS IN QUENCH WATER. POSSIBLE PLUG LINES POSSIBLE CORROSION IN TANK AND PIPING	SAMPLES CAN BE PULLED FROM DRAIN VALVE.	LINE V-002 WITH CORROSION RESISTANT LINING		YES - SPRAY ON TEFLON	8/23/94	C030	4/20/95
16	VISCOSITY			NO ISSUES IDENTIFIED							
17	CORROSION		LOW pH PROCESS WATER. HCL BACKFLOW FROM RX BIOLOGICAL GROWTH LOSS OF NITROGEN PAD ON TANK IN THE PRESENCE OF WATER VAPOR.	CLOG VALVE OR PIPING COMPROMISE MECHANICAL INTEGRITY OF V-002		LINE V-002 WITH CORROSION RESISTANT LINING		YES - SPRAY ON TEFLON	8/23/94	C030	4/20/95
18	INTERLOCKS AND CONTROLS					ADD INTERLOCK TO SHUT DOWN FEEDS AND OXYGEN WHEN QUENCH TANK LEVEL DROPS BELOW ITS HIGH LEVEL SETPOINT		YES	8/23/94	I001	INTERLOCK
						ADD LOCAL QUENCH ACTIVATION BUTTONS ON EACH OPERATING LEVEL		YES	8/23/94	C031	4/20/95

COMPANY Delphi Research, Inc. -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 4
SYSTEM NAME R-101 REACTOR AND OVERHEAD SYSTEM
P&ID D-2001 // D-2001A // D-2003 // D-2004
COLOR ORANGE

DESIGN HAZARD AND OPERABILITY REVIEW

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ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/ SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND NUMBER	DATE IMPLEMENTED
4.1	NO/LESS Flow	REACTOR OVERHEAD	NO REACTION PLUGGED OVERHEAD LINE OR EXCHANGER CLOSED PRESSURE CONTROL VALVE OR VALVE AT R-101. PLUGGED SPARGER IN R-102 PLUGGED CARBON BED.	HIGH PRESSURE IN RX REVERSE FLOW INTO OTHER LINES (SEE APPROPRIATE SYSTEMS)	RUPTURE DISK ON REACTORS. PRESSURE CONTROL ON REACTORS PRESSURE INDICATOR ON REACTORS FLOW METER ON OVERHEAD LINE RELIEF VALVE ON OVERHEAD RECEIVER RELIEF VALVE ON RECYCLE COMPRESSOR POSITION INDICATORS ON XVS AT R-102	SOP TO REQUIRE PERIODIC INSPECTION OF PIPING AND EQUIPMENT		YES	8/23/94	I507	SOP
						PRESSURE CONTROL VALVE TO BE A VEE-BALL TYPE VALVE		YES	8/23/94	C053	4/20/95
						CONSIDER ADDING A BACKUP PRESSURE CONTROL VALVE		YES - ADD BACKUP VALVE WITH MANUAL SOFTWARE SWITCH TO DIRECT CONTROLLER OUTPUT TO EITHER VALVE.	8/23/94	C053	4/20/95
						ADD SOP TO ENSURE PROPER VALVE LINE-UP AT R-102			8/2/95	P301	SOP
						ADD POSITION INDICATOR ON CONTROL SYSTEM FOR PRESSURE CONTROL VALVE		YES	8/23/94	C054	4/20/95
		OXYGEN	CONTROLS FAILURE VALVE CLOSED LOSS OF SUPPLY PLUGGED LINE	NO / SLOW REACTION UPSET REACTOR CONTROLS	O2 FLOW METER O2 MONITOR ON VENT LINE						
			RUPTURED LINE	OXYGEN RELEASE. POTENTIAL FIRE.	O2 FLOW METER PIPING DESIGNED TO HANDLE TAP REDUNDANT SHUTOFF VALVES AT REACTOR						
		NITROGEN (WHEN NEEDED)	CONTROLS FAILURE VALVE CLOSED. LOSS OF SUPPLY PLUGGED LINE	INABILITY TO BLANKET REACTOR WITH NITROGEN POSSIBLE INCREASE IN OXYGEN OR FLAMMABLE MIXTURE IN VENT	POSITION INDICATOR ON VALVE. O2 ANALYZER	ADD LOCAL PI TO N2 LINE AT BATTERY LIMIT BLOCK VALVE			8/2/95	G302	8/2/95
			RUPTURED LINE	NITROGEN RELEASE - NO HAZARDS SINCE UNIT WILL BE IN OUTSIDE OR IN WELL VENTILATED AREA	PIPING SYSTEM DESIGNED TO HANDLE TEMPERATURE AND PRESSURE						
		RECYCLE GAS	CLOSED VALVES EQUIPMENT FAILURE. PLUGGED LINE OR CHECK VALVE CONTROL FAILURE. LOW REACTOR WEIGHT	INCREASED REACTOR LEVEL REACTOR UPSET NO HAZARDS	RUN LIGHT ON COMPRESSOR FLOW INDICATOR ON RECYCLE STREAM						
			RUPTURED LINE	RELEASE OF GASES POTENTIAL PERSONNEL EXPOSURE POTENTIAL ENVIRONMENTAL IMPACT	LINE RATED FOR TAP FLOW INDICATOR ON RECYCLE STREAM PSV ON COMPRESSOR DISCHARGE CHECK VALVE TO PREVENT REACTOR VAPOR RELEASE						
	MORE Flow	REACTOR OVERHEAD	CONTROL FAILURE. OPERATOR ERROR. HIGH REACTOR PRESSURE PRESSURE CONTROL VALVE OPEN HIGH ORGANIC FEED RATE. HIGH OXYGEN FEED RATE INCREASED ORGANIC CONTENT IN FEED LOSS OF REACTOR COOLING LOSS OF REFLUX CONDENSING HIGH NITROGEN FLOW	INCREASED REACTOR PRESSURE. INCREASED EXCHANGER LOADING POSSIBLE EMISSIONS POSSIBLY OVERPRESSURE V-201, V-301, F-301, AND V-303 DUE TO INCREASED PRESSURE DROP THROUGH CARBON BEDS. ENTRAIN DETOX SOLUTION FROM R-102 INTO OVERHEAD SYSTEM POSSIBLY DAMAGE CARBON BEDS - BLOW CARBON OUT OF BEDS	PRESSURE CONTROL VALVE ON REACTORS PRESSURE INDICATOR ON REACTORS RUPTURE DISK ON REACTORS PSVS ON V-202, V-301, F-201, AND V-303 FLOW METER ON VAPOR LINE WITH HIGH ALARM PRESSURE INDICATION UPSTREAM OF CARBON BEDS WITH HIGH ALARM	INVESTIGATE FAILURE MODE OF REACTOR PRESSURE CONTROL VALVE		YES - FAIL CLOSED	8/23/94	C053	4/20/95
						USE BOTTLED INSTRUMENT AIR FOR CRITICAL INSTRUMENTS		NO	---	--	
						ADD AN ACCUMULATOR TO INSTRUMENT AIR LINE TO ADD SURGE CAPACITY FOR SHUTDOWN. ALSO ADD PRESSURE TRANSMITTER WITH LOW ALARM		YES	8/23/94	C056	4/20/95
						CONTROL SYSTEM SHOULD BE ON UPS		YES	8/23/94	C017	JAT - DTL DSN
						CONTROL SYSTEM NARRATIVE SHOULD IDENTIFY CRITICAL INSTRUMENTS TO ALSO BE ON UPS		YES	8/23/94	C018	JAT - DTL DSN
						JACOBS TO CONFIRM THAT ENTRAINMENT VELOCITY IN R-102 WELL EXCEEDS EXPECTED VENT FLOWS		YES	8/2/95	G303	JAT - DTL DSN
						ADD INTERLOCK TO BYPASS R-102 ON HIGH FLOW IN REACTOR OVERHEAD SYSTEM		YES	8/2/95	I304	INTERLOCK
						JACOBS TO ENSURE THAT OPERATING VELOCITY THROUGH CARBON BEDS DOES NOT EXCEED 75% MAX RECOMMENDED VELOCITY		YES	8/2/95	G308	JAT - DTL DSN
			QUENCH WATER ADDED TO REACTOR	SEE QUENCH WATER SYSTEM							
			LINE RUPTURE (UPSTREAM OF PRESSURE CONTROL VALVE)	RELEASE OF REACTION VAPORS AND ENTRAINED LIQUIDS PERSONNEL EXPOSURE ENVIRONMENTAL RELEASE	PRESSURE INDICATIONS ON REACTOR AND VENT LINE LINES RATED FOR TAP	ADD REACTOR ISOLATION VALVE INTERLOCKED ON LOW REACTOR PRESSURE		NO		--	
						ADD AN AREA HCl MONITOR		YES	8/23/94	C032	4/20/95
						CONSIDER JACKETED PIPE TO PROVIDE SECONDARY CONTAINMENT WITH HCl MONITOR ON ANNULUS		NO		-	
						ADD INTERLOCKS TO SHUT OFF FEEDS		YES	8/23/94	I306	INTERLOCKS

COMPANY Delphi Research, Inc – Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 4
SYSTEM NAME R-101 REACTOR AND OVERHEAD SYSTEM
PAID D-2001 // D-2001A // D-2003 // D-2004
COLOR ORANGE

DESIGN HAZARD AND OPERABILITY REVIEW

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ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/ SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND. NUMBER	DATE IMPLEMENTED
						AND OXYGEN AND TO ACTIVATE QUENCH ON LOW (OR HIGH) REACTOR PRESSURE OR AREA HCL SENSOR					
						ADD AN AUDIBLE ALARM IN THE AREA ON SUDDEN LOSS (OR GAIN) OF REACTOR PRESSURE OR AREA HCL SENSOR		YES	8/31/94	C059	4/20/95
						PROVIDE PLEXIGLASS SPRAY PROTECTION SHIELD AROUND HIGH RISK FAILURE POINTS		NO	---	---	
						ADD FLANGE COVERS TO PROTECT AGAINST LEAKS		YES	8/31/94	C060	JAT - BY 31MAY
						SOPS TO ADDRESS PROPER PERSONAL PROTECTIVE EQUIPMENT WHILE IN UNIT		YES	8/31/94	P008	SOP
						ADD SPRAY CURTAIN TO KNOCK DOWN HCL - CONSIDER SIZING OF SECONDARY CONTAINMENT VESSEL		NO			
						ADD EYEWASH STATIONS AND SAFETY SHOWERS THROUGHOUT UNIT		YES	8/31/94	C018	4/20/95
	REVERSE Flow	RX OVERHEAD	REACTOR DOWN + RX PRESSURE CONTROL VALVE OPEN + OPERATOR ERROR V-303 AND INTO REACTOR	CONSIDERED UNLIKELY - NO SERIOUS CONSEQUENCES							
		OXYGEN AND NITROGEN	CHECK VALVE FAILS PLUS LOW SUPPLY PRESSURE PLUS VALVES OPEN - CONSIDERED UNLIKELY	BACK DETOX SOLUTION INTO LINE - CONSIDERED UNLIKELY.		CHANGE UPSTREAM SHUTOFF VALVE AND PIPING BETWEEN VALVES TO SS1 SPEC		YES	8/31/94	C061	4/20/95
						ADD TEMPERATURE, PRESSURE OR CONDUCTIVITY MEASUREMENT TO FEED LINE		YES - TEMPERATURE	8/31/94	C062	4/20/95
		RECYCLE GAS	EQUIPMENT FAILURE OPERATOR ERROR CONTROL FAILURE	POSSIBLE HOT HCL VAPORS IN RECYCLE PIPING, COMPRESSOR, AND CARBON BEDS	CHECK VALVE IN PIPING INTERNAL CHECK VALVES IN COMPRESSOR FLOW METER ON RECYCLE LINE						
4.3	MORE Temp	REACTOR OVERHEAD	LOSS OF COOLING TO REACTORS LOSS OF COOLING ON REFLUX CONDENSER LOSS OF COOLING ON OVERHEAD CONDENSER INCREASED ORGANIC FEED RATE INCREASE IN HEAT OF COMBUSTION OF FEEDSTOCK LOSS OF PRESSURE CONTROL (VALVE CLOSES)	OVERPRESSURE REACTORS OVERLOAD OVERHEAD SYSTEM - POSSIBLE EMISSIONS POSSIBLY DAMAGE RECYCLE COMPRESSOR DUE TO HIGH TEMPERATURE OR HOT HCL GASES	RUPTURE DISK ON REACTOR TEMPERATURE INDICATION ON REACTOR FEED AND OXYGEN FLOW CONTROLS PRESSURE CONTROL ON REACTOR TEMPERATURE INDICATION DOWNSTREAM OF E-301 SOLUTION COOLER E-201 CAN BE USED TO COOL REACTION DESIGN TEMPERATURE OF RECYCLE COMPRESSOR SET AT 450 DEGF	ADD INTERLOCK TO SHUT OFF FEEDS, MAXIMIZE COOLING TO REACTORS AND OVERHEAD EXCHANGERS, STOP OXYGEN FLOW, AND STOP REACTOR AGITATOR ON HIGH TEMPERATURE SWITCH TSHH 141		YES	8/31/94	I007	INTERLOCK
						ADD INTERLOCK TO TURN COMPRESSOR OFF ON HIGH OVERHEAD TEMPERATURE OR FLOW		YES	8/31/94	I006	INTERLOCK
			INTERNAL FIRE - EXCESS OXYGEN IN THE PRESENCE OF ORGANICS	OVERPRESSURE REACTOR POSSIBLE FLAME FRONT EQUIPMENT DAMAGE INJURY POTENTIAL POTENTIAL ENVIRONMENTAL RELEASE	OXYGEN FLOW CONTROL ON ANALYSIS OF REACTOR VENT STREAM OXYGEN ANALYZER ON VENT SYSTEM WITH HIGH ALARM NITROGEN PURGE TO REACTOR	ADD RESTRICTION ORIFICE IN OXYGEN LINE TO REDUCE POTENTIAL OF EXCESS OXYGEN ADDITION		YES	8/31/94	C063	4/20/95
						ADD INTERLOCKS TO SHUTDOWN OXYGEN ON HIGH REACTOR TEMPERATURE, QUENCH ACTIVATION, LOSS OF AGITATION, HIGH HIGH OXYGEN CONTENT IN REACTOR VENT, HIGH OXYGEN FLOW RATE, AND HIGH OR LOW REACTOR PRESSURE		YES	8/31/94	I008	INTERLOCK
			FIRE	OVERPRESSURE REACTOR	4" INSULATION RUPTURE DISK						
				DAMAGE TO EQUIPMENT, INSTRUMENTATION, AND PIPING	PSVS ON ALL EQUIPMENT	DETERMINE FIRE PROTECTION / SUPPRESSION REQUIREMENTS		YES	8/31/94	C065	DEFER TO PID
			JACKET STARTUP LINES IMPROPERLY VALVED	LOSS OF COOLING TO REACTOR - POSSIBLY ADD HEAT TO REACTOR	TEMPERATURE SENSOR ON REACTOR WITH HIGH AND LOW ALARMS	REPLACE VALVING ON STARTUP LINES WITH THREE-WAY VALVES		NO	---	---	
			EXCESS ORGANIC FEED IN COLD REACTOR AT STARTUP	DRAMATIC INCREASE IN REACTION RATE DURING HEATUP HIGH CO2 GENERATION POSSIBLY OVERPRESSURE REACTOR OVERLOAD OVERHEAD SYSTEM	PRESURE CONTROL ON REACTOR LOAD CELLS ON REACTOR RUPTURE DISK ON REACTOR TEMPERATURE MEASUREMENT ON REACTOR	SOP TO REQUIRE CHECKING INITIAL CONDITIONS OF REACTOR AND TO START UP REACTOR SLOWLY		YES	8/31/94	P009	SOP
						ADD INTERLOCK TO PREVENT FEEDING ORGANICS UNTIL REACTOR IS UP TO TEMPERATURE		YES	8/31/94	I009	INTERLOCK
				BOIL OFF ORGANICS DURING HEATUP POSSIBLE	VOC ANALYSIS ON VENT LINE	ADD SAMPLE / DRAIN VALVE BETWEEN MANUAL AND AUTOMATIC VALVES ON		YES	8/31/94	C064	4/20/95

COMPANY Delphi Research, Inc. -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 4
SYSTEM NAME R-101 REACTOR AND OVERHEAD SYSTEM
P&ID D-2001 // D-2001A // D-2003 // D-2004
COLOR ORANGE

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				EMISSIONS - ORGANICS IN OVERHEAD CONDENSATE		V-301 OUTLET TO ALLOW DRUMMING OFF TANK FOR RETURN TO REACTOR					
		OXYGEN	SEE REVERSE FLOW ABOVE.								
			GREASE IN LINE FROM FABRICATION.			SOP TO CLEAN LINE, INSTRUMENTS, FITTINGS FOR OXYGEN SERVICE AT COMISSIONING AND AFTER MAINTENANCE		YES	8/23/94	P010	SOP
	LESS Temp		LOW AMBIENT TEMPERATURE. EXCESSIVE REACTOR COOLING EXCESS WATER IN FEED. INSUFFICIENT OXYGEN LOSS OF AGITATION COLD FEED	INCOMPLETE REACTION FREEZING DETOX COULD SEIZE AGITATOR NO HAZARDS IDENTIFIED		ADD INTERLOCK TO SHUT DOWN AGITATOR ON REACTOR TEMP LESS THAN 70 DEG C		YES	8/23/94	I010	INTERLOCK
				FREEZE WATER CONDENSED BY E-301		INSULATE PIPING BETWEEN E-301 AND V-301		NO	--	--	
						DESIGN TO ALLOW SPACE FOR FIELD TRACING AT A LATER TIME		NO	--	--	
						FIELD TRACE TO 40 DEG C		YES	8/23/94	G016	4/20/95
			MOISTURE AROUND RECYCLE FREEZES.	POSSIBLE PLUGGED LINE OR DAMAGED COMPRESSOR	FREE DRAIN LINES FROM RECYCLE COMPRESSOR.	FREE DRAIN LINES TO CARBON BEDS; TAKE RECYCLE COMPRESSOR SUCTION FROM TOP OF LINE		YES	[illegible]	G107	[illegible]
			WATER QUENCH ACTIVATED	SEE QUENCH WATER SYSTEM							
4.3	MORE Press		SEE FLOW AND TEMPERATURE SECTIONS ABOVE.								
		R-101 JACKET	HOT OIL THERMAL EXPANSION.	DAMAGE TO JACKET	RELIEF VALVE ON REACTOR JACKET						
		OXYGEN / NITROGEN	SEE MORE / LESS FLOW ABOVE								
	LESS Press		SEE FLOW AND TEMPERATURE SECTIONS ABOVE, PLUS:								
			LEAKING AGITATOR SEAL.	PERSONNEL EXPOSURE ENVIRONMENTAL RELEASE POTENTIAL TO EQUIPMENT AND INSTRUMENTATION		ADD AREA HCl MONITOR		YES	8/23/94	G013	4/20/95
		OXYGEN / NITROGEN	SEE LESS FLOW ABOVE								
	REVERSE Press		REACTORS ARE BLOCKED IN HOT AND THEN COOLS	COLLAPSE EQUIPMENT AND PIPING	REACTORS HAVE VACUUM PROTECTION TO 10 " WC VACUUM	CONFIRM THAT R-101 AND E-101 (T/S) ARE RATED FOR FULL VACUUM		NO			
						CONFIRM THAT RUPTURE DISK CAN HANDLE VACUUM		YES	8/23/94	G066	IAT - DTL DSN
						SPAN PRESSURE TRANSMITTERS TO INDICATE PARTIAL VACUUM (-1 PSIG)		YES	8/23/94	G067	IAT - DTL DSN
						SOP TO REQUIRE OPENING PRESSURE CONTROL VALVE DURING HOT SHUTDOWN		YES	8/23/94	P011	SOP
						CONFIRM TANTALUM LINED PIPE CAN HANDLE FV.		YES	8/23/94	G068	IAT - DTL DSN
4.4	NO/LESS Level		TANK RUPTURE RUPTURED LINES CONNECTED TO REACTORS	SEE MORE FLOW (CAUSE = RUPTURE LINE) ABOVE		MEASURE ELECTRIC POTENTIAL BETWEEN REACTOR SHELL AND ONE OF TANTALUM HP TUBES TO DETECT A BREACH OF THE TANTALUM LINER		YES	8/23/94	G069	4/20/95
						IMPLEMENT A BACK UP SYSTEM TO DETECT LINER BREACH ON BOTH REACTORS BY EITHER USING (1) AIR SWEEP WITH HCl SENSOR, OR (2) PRESSURE MEASUREMENT BETWEEN SHELL AND LINER		YES	[illegible]	G109	IAT TO RECOMMEND
						ADD CORROSION COUPONS TO REACTOR		YES	8/23/94	G078	IAT - DTL DSN
			BOIL OFF TOO MUCH WATER OPERATOR OR INSTRUMENTATION ERROR. REMOVED TOO MUCH DETOX SOLUTION TO FILTRATION AREA	SYSTEM UPSET INCREASE SPECIFIC GRAVITY AND VISCOSITY OF SOLUTION THROUGH EVAPORATION POSSIBLY DAMAGE TO AGITATOR DUE TO LOW LEVEL OR HIGH VISCOSITY	LOAD CELLS ON REACTORS	SOP TO REQUIRE PERIODIC SAMPLING AND TESTING OF DETOX SOLUTION		YES	8/23/94	P012	SOP
	MORE Level		LOSS OF REACTION EXCESSIVE REFLUX. EXCESSIVE WATER IN FEED	OVERFILL REACTORS INTO OVERHEAD SYSTEM. FILL FREE SPACE IN REACTOR REQUIRED TO ACCOMODATE QUENCH WATER	LOAD CELLS ON REACTORS. ABILITY TO CONTROL REFLUX RATE BY VARYING T&P ON THE OUTLET OF E-101. LEVEL INDICATION ON REACTOR OVERHEAD RECEIVER	ADD LOW POINT DRAIN & SIGHT GLASS TO BOTTOM OF T-401 AND ELIMINATE V-401		YES	8/23/94	G033	4/20/95
4.5	MORE Comp	WATER IN FEED	SEE MORE LEVEL ABOVE								
		ORGANIC IN	SEE MORE FLOW ABOVE								

COMPANY. Delphi Research, Inc. -- Proprietary Information
PROJECT. METC Wet Oxidation Project
SYSTEM NUMBER 4
SYSTEM NAME R-101 REACTOR AND OVERHEAD SYSTEM
P&ID D-1001 // D-1001A // D-1003 // D-1004
COLOR. ORANGE

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		FEED									
		FERRIC CHLORIDE	BOIL OFF TOO MUCH WATER	INCREASE SOLUTION VISCOSITY POSSIBLY SOLIDIFY SOLUTION DAMAGE OR TRIP OUT AGITATOR	LOAD CELLS ON REACTOR EXCESS CONDENSATE WILL BE NOTICED IN V-301	SOP TO REQUIRE PERIODIC SAMPLING AND TESTING OF DETOX SOLUTION		YES	8/23/94	P012	SOP
		GREASE IN OXYGEN LINE	SEE MORE TEMPERATURE ABOVE								
		FERRIC PHOSPHATE	INCOMPLETE OR IMPROPER FILTRATION WHILE RUNNING TDP	REDUCED FERRIC IONS DECREASED REACTION RATE POSSIBLY PLUG SOLUTION LINES OR REACTOR	IT WILL BE EVIDENT THAT AN INSUFFICIENT AMOUNT OF PHOSPHATE SALTS WILL BE REMOVED BY INSPECTING AND WEIGHING THE FILTER CAKES WEIGH CELLS ON REACTOR	SOP TO REQUIRE PERIODIC SAMPLING AND TESTING OF DETOX SOLUTION		YES	8/23/94	P013	SOP
		CARBON BED	ORGANIC CARRYOVER INTO VENT SYSTEM	PERSONNEL / ENVIRONMENTAL EXPOSURE	CARBON BED T-401 VOC MONITOR ON VENT	ADD SECOND CARBON BED IN LEAD/LAG CONFIGURATION		YES - NAME THEM T-401A/B	8/23/94	C076	4/20/95
	LESS Comp	DETOX SOLUTION	SEE LOW LEVEL ABOVE.	NO HAZARDS IDENTIFIED	O2 AND CO2 ANALYZERS ON VENT SYSTEM						
		OXYGEN	SLOW REGENERATION OF IRON IONS	SLOW REACTION - NO HAZARDS							
		ORGANICS	SEE NO/LESS FLOW, SYSTEM 1.	SLOW REACTION - NO HAZARDS							
	AS WELL AS Comp	RADIONUCLIDES	RADIONUCLIDES IN FEED			DETERMINE DOE REQUIREMENTS, IF ANY		NO SPECIAL REQMTS PER DOE			
		TOXIC METALS	TOXIC METALS IN FEED	POTENTIALLY GET METALS IN FILTER CAKE - PERSONNEL EXPOSURE		SOPS TO REQUIRE PROPER PPE WHILE HANDLING FILTER CAKES		YES	8/23/94	P013	SOP
						SOP TO PERIODICALLY SAMPLE DETOX SOLUTION		YES	8/23/94	P013	SOP
	PART OF Comp		SEE LESS COMPONENTS ABOVE								
	OTHER THAN Comp	SOLID CHUNKS GREATER THAN ONE HALF INCH IN REACTOR	SHREDDER MALFUNCTION AGGLOMERATION OF SOLIDS	PLUG SOLUTION DIP TUBE		ADD SCREEN AT BOTTOM OF DIP TUBE TO PREVENT PLUGGING BY LARGE CHUNKS		YES - TEFLON CAGE	8/23/94	C071	4/20/95
	OTHER THAN Comp	SLUDGES	SEE ORGANIC FEED SYSTEMS								
		OXYGEN	NO RESAONABLE CAUSES IDENTIFIED								
4.5	VISCOSITY		SEE ABOVE								
4.6	IGNITION SOURCE		SPARKS FROM MOTOR(S)		ALL MOTORS SPECIFIED FOR FOR CLASS I DIV2 GROUP C&D.	DETERMINE IF TANTALUM CAN SPARK IF HIT BY ANY METAL		YES	8/23/94	C076	KH - 13MAY
4.7	SHUTDOWN					SOP TO PROVIDE SAFE REMOVAL OF REACTOR HEADS		YES	8/23/94	P014	SOP
4.8	JACKET LEAK	R-102 CLAMP-ON JACKET	JACKET FAILURE.	PERSONNEL & ENVIRONMENTAL EXPOSURE							
4.9	LOSS OF POWER		LOSS OF POWER AGITATOR STOPS. FEED STOPS O2 STOPS COOLING OIL STOPS	HOT GASSES TO OVERHEAD		CONSIDER POWER FAILURE FOR REACTOR PRESSURE CONTROL VALVE FAILURE MODE		YES - SEE ABOVE	8/23/94	C035	4/20/95
						ADD INTERLOCK TO SHUTOFF O2 ON LOSS OF AGITATOR		YES	4/20/95	C035	INTERLOCK
						ESTIMATE IMPACT OF POWER FAILURE IN TERMS OF ENVIRONMENTAL RELEASE		YES - WILL BE ADDRESSED IN ENVIRONMENTAL REVIEW	8/23/94	C039	DEFER TO PHR
4.10	SERVICE FAILURE	CHILLER	MALFUNCTION OF EQUIPMENT OPERATOR ERROR LOSS OF POWER	PERSONNEL & ENVIRONMENTAL EXPOSURE	TEMP INDICATORS ALONG VENT LINE HCl ANALYZER IN VENT LINE RUN LIGHT ON CHILLER	ADD INTERLOCK TO SHUTDOWN UNIT ON LOSS OF CHILLER, HIGH HIGH TEMPERATURE IN VENT, OR HIGH HIGH HCl IN VENT		YES	8/23/94	H11	INTERLOCK
						CHILLER DESIGN TO INCLUDE RUN LIGHT IN CONTROL SYSTEM		YES	8/23/94	C080	4/20/95
4.11	EROSION	REACTOR	SOLIDS IN REACTOR SOLUTION	EARLY AGITATOR, LINER, BAFFLE, AND/OR D TUBE FAILURE POTENTIAL DETOX RELEASE	STRAINER ON SUCTION OF P-001	ADD LIQUID ORGANIC SURGE DRUM WITH SIDE DRAW		NO	---	---	
						ADD SCREENS, TRAPS, OR BASKETS TO CATCH INSOLUBLE CHUNKS TO PREVENT SCRATCHING REACTOR LINER		NO - LEARN FROM DEMONSTRATION UNIT	---	---	
		VENT LINES	NONE								
4.12	THERMAL EXPANSION	HOT OIL EXCHANGER AND JACKETS	BLOCKED IN COOLING OIL	RUPTURED EXCHANGER	THERMAL RELIEF ON R-101 JACKET	ADD THERMAL RELIEF VALVES ON ALL HOT OIL HEAT EXCHANGERS		YES	8/23/94	C081	4/20/95
4.13	TUBE RUPTURE	E-301	HARDWARE FAILURE	HOT OIL INTO PROCESS UNIT UPSET	TEMPERATURE INDICATOR DOWNSTREAM OF E-301 [illegible] DOWNSTREAM OF E-301						
4.14	STATIC ELECTRICITY		FALLING LIQUIDS	POSSIBLE FIRE - CONSIDERED UNLIKELY DUE TO HIGH HUMIDITY AND HIGH CONDUCTIVITY							

COMPANY Delphi Research, Inc. - Proprietary Information
PROJECT MEIC Wet Oxidation Project
SYSTEM NUMBER 4
SYSTEM NAME R-101 REACTOR AND OVERHEAD SYSTEM
P&ID D-2001 # D-2001A # D-2003 # D-2004
COLOR ORANGE

DESIGN HAZARD AND OPERABILITY REVIEW

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				IN THE REACTOR							

COMPANY Delphi Research, Inc. -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 5
SYSTEM NAME SOLIDS HANDLING SYSTEM
P&ID D-2001 // D-2001A // D-2002 // D-2002A
COLOR YELLOW

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3.1	NO/LESS Flow	DETOX SOLN FROM REACTOR	VALVES CLOSED PLUGGED DIP TUBE PLUGGED LINE PLUGGED FILTER PLUGGED COOLER SCALING IN E-101. LOW PRESSURE IN R-101 IMPROPER VALVE LINEUP. LOSS/DAMAGED DIPTUBE.	DAMAGE TO A 101 DUE TO BUILDUP OF SOLIDS IN R-101 INCREASED EROSION IN RX REDUCED COOLING IN E-101 PLUGGED SAMPLE POINT REACTOR UPSET POSSIBLE FLOODING OF R-101	FLOW METER ON DETOX LINE WITH LOW ALARM TEMPERATURE MEASUREMENT ON DETOX LINE PRESSURE MEASUREMENT ON F-201 & REACTORS LAB STUDIES SHOW THAT SOLIDS SETTLE SLOWLY TANTALUM IS HIGHLY RESISTANT TO FOULING WEIGH CELLS ON REACTORS	ADD SCREEN AT BOTTOM OF DIP TUBE TO PREVENT PLUGGING BY LARGE CHUNKS		YES - TEFLON CAGE	8/23/94	G073	4/20/95
						CONFIRM LINE SIZE ADEQUATE FOR SOLIDS		YES	8/23/94	G034	IAT-DTL DSN
						MINIMIZE POCKETS IN LINE TO FILTER		YES	8/23/94	G074	4/20/95
						INVESTIGATE SOLUBILITY OF FePO4		YES - BY DELPHI	8/23/94	G139	DRI.
						CONSIDER CRYSTALLIZATION OF FePO4 IN F-201 DESIGN.		NO PROBLEM PER DRI			
						CONSIDER FILTERING AT REDUCED R-101 TEMP		NO ACTION NOW - INVESTIGATE AFTER STARTUP	...	...	
						FINALIZE DETOX SAMPLING SYSTEM		YES	8/23/94	G070	IAT - DTL DSN
						CHANGE FILTER VENT VALVE TO MODULATING V-PORT BALL TO ALLOW SLOW DEPRESSURIZATION		YES	8/23/94	G033	4/20/95
						ADD TEMPERATURE INDICATION ON F-201		YES	8/23/94	G078	4/20/95
						FREE DRAIN FILTER VENT LINE TO V-202		YES	8/23/94	G079	4/20/95
				DETOX FREEZES IN LINE		HEAT TRACE LINES TO PREVENT FREEZING		YES - TO 50 DEGC	8/23/94	G017	4/20/95
						ADD WATER & ACID WASHES THROUGH DETOX LINE		YES	8/23/94	G080	4/20/95
			FILTER OUTLET CLOSED	OVERPRESSURE FILTER	PSV'S ON F-201 & V-202 IS SET BELOW RUPTURE DISKS ON REACTORS F-201 AND V-202 DESIGNED FOR TAP						
			RUPTURED LINE	PERSONNEL / ENVIRONMENTAL EXPOSURE	JACKETED PIPE TO FILTER	JOIN JACKETS WITH TUBING AND ADD HCl OR PRESSURE SENSOR		YES - PRESSURE	8/23/94	G081	IAT - DTL DSN
		DETOX SOLN RETURN	PLUGGED FILTER. P-202A/B FAILS. V-202 EMPTY 3 WAY VALVE FAILS OR WRONG LINEUP. CONTROLS FAILURE.	BUILDUP OF SOLIDS IN R-101. PUMP DAMAGE. POSSIBLE INCREASED CORROSION AT PUMP. OVERPRESSURE V-202	LEVEL SWITCH ON V-202 LEVEL INDICATION ON FILTER POSITION INDICATOR ON 3 WAY VALVE PRESSURE INDICATION ON P-202 PRESSURE INDICATION ON FILTER ZSC ON FILTER VENT VALVE PRESSURE INDICATOR ON NITROGEN LINE PSV ON V-202 SET LOWER THAN RUPTURE DISKS ON REACTORS SOLUTION RETURN PUMP HAS INSTALLED SPARE						
			HIGH R-101 PRESSURE LACK OF OR LOW N2 PRESSURE CLOSED VALVE FILTER VENT VALVE OPEN	DETOX FREEZES IN LINE OR V-202	ACID WASH LINE INSTALLED TO FLUSH DETOX LINE TO FILTER	SOP TO SEND FIRST ACID WASH TO SURGE TANK		YES	8/23/94	P016	SOP
						HEAT TRACE ALL DETOX PIPING AND EQUIPMENT		YES - TO 50 DEGC	8/23/94	G017	4/20/95
			DRUM DISCHARGE VALVE OPEN AGITATOR SEAL LEAK	ENVIRONMENTAL & PERSONNEL EXPOSURE		REVIEW VENDOR DESIGN FOR FILTER INTEGRITY		YES	8/23/94	G082	IAT - DTL DESIGN
						ADD AREA HCl MONITOR		YES	8/23/94	G083	4/20/95
			LINE RUPTURE.	ENVIRONMENTAL & PERSONNEL EXPOSURE		JACKET DETOX RETURN PIPING		YES	8/23/94	G084	4/20/95
		ACETIC ACID	PUMP FAILURE NO ACID IN TANK(S)	IMPROPER FILTER CAKE WASHING	PRESSURE INDICATION ON P-201 LEVEL SWITCH ON V-201 AND V-203 ZSC ON VALVE TO FILTER FROM ACID TANKS	IAT TO COORDINATE WITH FILTER VENDOR TO PROVIDE LEVEL TRANSMITTER		YES	8/2/95	G310	IAT - DTL DSN
			LINE RUPTURE	ENVIRONMENTAL & PERSONNEL EXPOSURE. - NO MAJOR HAZARD	EQUIPMENT & PIPING DESIGNED FOR MAX OPERATING PRESSURES						
	MORE Flow	SYSTEM	FCV OPEN HIGH PRESSURE IN R-101. HIGH FLOW THROUGH P-202 CONTROL FAILURE	INSUFFICIENT SOLUTION COOLING POTENTIAL CORROSION IN SYSTEM. OVERFILL OR EMPTY V-202 DROP LEVEL IN REACTORS - UNIT UPSET OVERFILL FILTER - SEE MORE LEVEL BELOW	TEMPERATURE INDICATION ON DETOX LINE FLOW INDICATION ON DETOX LINE HIGH HIGH LEVEL ON V-202 LOAD CELLS ON REACTORS PRESSURE INDICATION ON FILTER	ADD INTERLOCK TO CLOSE SOLUTION SHUTOFF VALVE ON 101 TEMP OR HIGH REACTOR PRESSURE FOR E-101		YES - MAY NEED TO SET 101 TEMPERATURE HIGH IF E-101 IS USED TO COOL REACTOR	8/23/94	I013	INTERLOCK
						ADD INTERLOCK TO CLOSE SURGE VESSEL VENT VALVE ON HH LEVEL		YES	8/23/94	I013	INTERLOCK
						ADD INTERLOCK TO CLOSE ACID, N2, AND SOLIDS DISCHARGE VALVES TO FILTER ON HIGH FILTER PRESSURE		YES	8/23/94	I014	INTERLOCK
			LINE RUPTURE	SEE LESS FLOW ABOVE							
	REVERSE Flow	DETOX PIPING	NO REASONABLE CAUSES								

COMPANY Delphi Research, Inc -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 5
SYSTEM NAME SOLIDS HANDLING SYSTEM
P&ID D-2001 // D-2001A // D-2002 // D-2002A
COLOR YELLOW

DESIGN HAZARD AND OPERABILITY REVIEW

REVISION 1
9-Aug-95
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ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/ SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND. NUMBER	DATE IMPLEMENTED
			IDENTIFIED								
		N2 AND ACETIC ACID	FAILURE TO DEPRESSURIZE FILTER. IMPROPER VALVE LINE-UP.	DETOX FLOW INTO N2 OR ACID LINES.	CHECK VALVE ON P-201 DISCHARGE	ADD CHECK VALVE IN N2 LINE		YES	8/31/94	C085	4/20/95
						ADD INTERLOCK TO CLOSE VALVES ON FILTER PRESSURE ABOVE 45 PSIG		YES	8/31/94	I014	INTERLOCK
5.2	MORE Temp	DETOX LINES	LOSS OF COOLING. HIGH REACTOR TEMP SEE MORE FLOW ABOVE.	INCREASED CORROSION. SEE MORE FLOW ABOVE.							
		SYSTEM	FIRE	PERSONNEL / ENVIRONMENTAL EXPOSURE EQUIPMENT DAMAGE	PSV ON EQUIPMENT	ADD PSV ON SHELL SIDE OF COOLER		YES	8/31/94	C086	4/20/95
						ADD RELIEF PROTECTION ON TUBE SIDE FOR ALL HOT OIL SHELL & TUBE EXCHANGERS		YES	8/31/94	C087	8/2/95
	LESS Temp	DETOX LINE	SEE NO/LESS FLOW ABOVE								
		ACETIC ACID	LOW AMBIENT TEMP.	FREEZE LINES AND EQUIPMENT		DESIGN TO ALLOW SPACE FOR HEAT TRACING AT A LATER TIME		NO	...	...	
						FIELD TRACE TO 40 DEG F		YES	8/31/94	C016	4/20/95
5.3	MORE Press		SEE MORE/LESS FLOW ABOVE.	OVERPRESSURE EQUIPMENT	RELIEF VALVES F-201 & V-202 SET ABOVE MAX PUMP DISCHARGE PRESSURE.	CONFIRM THAT OSHA DOES NOT LIMIT PRESSURE OF NITROGEN USED IN PRESSURE FILTERING		DETERMINED THERE WAS NO PROBLEM	8/31/94	C088	8/2/95
						DELETE DIAPHRAGM SEAL ON PRESSURE INDICATOR PI-208		YES	8/31/94	C089	4/20/95
	LESS Press			NO NEW HAZARDS IDENTIFIED.							
	REVERSE Press	DETOX SYSTEM	BLOCKED IN E-201, F-201 AND V-202 WHILE LIQUID FULL AND COOLED DOWN. PUMP OUT OF V-202 WHILE TANK BLOCKED IN	REDUCED PRESSURE IN EQUIPMENT		RATE E-201 (T/S), F-201, AND V-202 FOR FULL VACUUM OR ADD VACUUM PROTECTION		YES	8/31/94	C090	IAT - DTL DSN
		V-202	PUMPING OUT OF V-202 - CHECK VALVE ON VENT LINE WON'T ALLOW AIR TO DISPLACE LIQUID	FULL VACUUM ON TANK.		REMOVE CHECK VALVE ON V-202 VENT LINE #205. NOTE THAT THIS LINE NUMBER IS INCORRECTLY LABELLED #025 ON D-2004		YES	8/31/94	C091	4/20/95
						RATE V-202 FOR FULL VACUUM - SEE C090					
		ACETIC ACID	NO NEW CAUSES IDENTIFIED								
5.4	NO/LESS Level	F-201		NONE IDENTIFIED							
		V-202	OPERATOR ERROR. INSTRUMENT FAILURE	NO HAZARDS IDENTIFIED							
		ACETIC ACID	OPERATOR ERROR INSTRUMENT FAILURE	UNABLE TO WASH FILTER CAKE. NO HAZARDS IDENTIFIED							
	MORE Level	FILTER CAKE	OPERATOR ERROR EQUIPMENT FAILURE	PLUGGED FILTER. SEE NO/LESS FLOW ABOVE							
		FILTER		NO NEW HAZARDS IDENTIFIED							
		SOLIDS DRUM	OPERATOR ERROR.	OVERFILL DRUM - DRUM TOO HEAVY TO HANDLE		ADD WEIGH SCALE WITH READOUT IN CONTROL ROOM FOR DRUM WEIGHT INDICATION		NO		...	
						ALLOW FORKLIFT ACCESS TO MOVE 55 GALLON DRUMS OR USE SMALLER DRUMS		YES - INVESTIGATE	8/31/94	C092	IAT - DTL DESIGN
		V-202		NO NEW HAZARDS							
		V-301	OPERATOR ERROR	HOUSEKEEPING PROBLEM NO HAZARDS IDENTIFIED							
		V-201	OPERATOR ERROR	ACID / DETOX IN VENT LINE TO CARBON BED	HIGH LEVEL SWITCH IN V-201 TANK IS SIZED TO HANDLE FILTER CAPACITY	ADD LSHH ON V-201		YES	8/1/95	C211	8/2/95
5.5	MORE Comp	SOLIDS IN DETOX LINE.	OPERATOR ERROR	LOSS OF IRON IN R-101. LARGER FILTER CAKES / INEFFICIENT FILTRATION REACTOR UPSETS LINE PLUGGAGE NO MAJOR HAZARDS IDENTIFIED							
	AS WELL AS Comp	INERT SOLIDS IN DETOX.	RESIDUAL SOLIDS IN REACTOR	PLUG DIP TUBE, LINES, E-201, OR FILTER.		ADD SCREEN AT BOTTOM OF DIP TUBE TO PREVENT PLUGGING BY LARGE CHUNKS		YES - FILTER CAGE	8/31/94	C013	4/20/95
		REACTOR VAPORS TO FILTER	CORROSION - LOSS OF DIP TUBE	UNIT UPSET. NO LIQUID FLOW. LOSS OF COOLING TO REACTOR	FLOW INDICATION ON DETOX LINE. LEVEL INDICATION ON FILTER. TEMPERATURE AND PRESSURE INDICATION ON REACTOR.						
	MORE Comp	ACETIC ACID	OPERATOR ERROR- HIGHER ACID CONCENTRATION.	COULD GET SOME FeOH DISSOLVING INTO ACID INCREASED ORGANIC FLOW TO REACTORS		SOP TO PRESCRIBE HOW ACID IS TO BE MADE.		YES	8/1/95	C212	SOP
5.6	VISCOSITY		SEE TEMP ABOVE								
5.7	HEATING/COOLING		SEE TEMP ABOVE								
5.8	SERVICE FAILURE		NO NEW HAZARDS IDENTIFIED								
5.9	HEAT EXCHANGER	E-201	TUBE LEAK	PERSONNEL & ENVIRONMENTAL	CONDUCTIVITY ANALYZER ON OIL	ADD ISOLATION VALVES ON PUMP SUCTION		YES	8/31/94	[illegible]	4/20/95

COMPANY Delphi Research, Inc -- Proprietary Information
PROJECT MTTC Wet Oxidation Project
SYSTEM NUMBER 5
SYSTEM NAME SOLIDS HANDLING SYSTEM
P&ID D-2001 // D-2001A // D-2002 // D-2002A
COLOR YELLOW

DESIGN HAZARD AND OPERABILITY REVIEW

REVISION [illegible]
[illegible] Aug 9[illegible]

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ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/ SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMENDATION NUMBER	DATE IMPLEMENTED
	LEAKS			EXPOSURE. CONTAMINATION OF OIL SYS REACTION OF DETOX WITH OIL. CONDUCTIVITY MEASUREMENT ON OIL SYSTEM.	SYSTEM.	OF EACH HOT OIL LOOP TO FACILITATE SPEEDY ISOLATION					
		V-202 REFLUX CONDENSER	PIPE RUPTURE	CHILLED OIL IN SOLUTION SURGE DRUM HIGH LEVEL IN V-202.	HIGH HIGH LEVEL INDICATION ON V-202	INTERLOCK VENT VALVE TO CLOSE ON HH LEVEL ON V-202.		YES	8/23/94	K013	INTERLOCK
5.10	POWER FAILURE		SEE LESS TEMPERATURE NO NEW HAZARDS IDENTIFIED								
5.11	DETOX SAMPLE STATION					FORMALIZE DESIGN OF DETOX SAMPLING SYSTEM. SELECT SAMPLE BOMBS IF POSSIBLE, WHICH WON'T ALLOW PULLING A SAMPLE UNLESS THE BOMB IS IN PLACE.		YES	8/23/94	G020	IAT - DET DSN
5.12	LOSS OF COOLING	V-202 VENT CONDENSER	CLOSED VALVE CHILLER PACKAGE FAILURE	INCREASED HCl TO CARBON BED NO SERIOUS HAZARDS	HCl ANALYZER ON VENT LINE						

COMPANY Delphi Research, Inc -- Proprietary Information
PROJECT MLTC Wet Oxidation Project
SYSTEM NUMBER 6
SYSTEM NAME CONDENSATE SYSTEM
P&ID D-3001
COLOR PURPLE

DESIGN HAZARD AND OPERABILITY REVIEW

REVISION 3
9-Aug 95
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ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/ SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMENDATION NUMBER	DATE IMPLEMENTED
6.1	NO / LESS Flow	WATER TO V-301	VALVE CLOSED PLUGGED LINE V-301 FULL VENT ON V-301 BLOCKED OPERATOR ERROR NO LEVEL IN V-303 CONTROLS FAILURE	V-303 OVERFILLS UNIT UPSET OVERPRESSURE R-101 CARBON BED FILLS WITH WATER POTENTIAL WATER DAMAGE TO ANALYZERS	LEVEL TRANSMITTER ON V-301 AND V-303 WITH ALARMS LSHH ON V-301 ZIC ON DRAIN VALVE FROM V-303 PSV ON V-303 & RUPTURE DISK ON R-101	ADD OVERFLOW LINE FROM V-303 TO V-301		NO			
						ADD SEPARATE LSHH ON V-301		YES	8/23/94	G098	4/20/95
						ADD PT ON V-301 WITH HIGH ALARM		YES	8/23/94	G099	4/20/95
			LINE RUPTURE	PERSONNEL / ENVIRONMENTAL EXPOSURE	PIPING DESIGN TO HANDLE PRESSURE						
		CAUSTIC TO E-304	VALVE CLOSED DRUM EMPTY. PUMP FAILURE. CONTROLS FAILURE HIGH PRESSURE IN V-301	NO PH ADJUSTMENT UNIT UPSET NO HAZARDS		SOP TO REQUIRE CHECKING DRUM LEVEL		YES	8/23/94	2018	SOP
						ADD SAMPLE PORT ON P-302 TO VERIFY FLOW		YES - ON ALL PROCESS PUMPS	8/23/94	G100	4/20/95
						CONFIRM SIZE OF P-302		YES	8/23/94	G101	8/2/95
						CONFIRM 35 GALLON DRUM IS A WORKABLE SIZE FOR THIS SERVICE		YES	8/23/94	G102	IAT - DTL DSN
			LINE RUPTURE.	PERSONNEL / ENVIRONMENTAL EXPOSURE EQUIPMENT DAMAGE	PSV ON V-301 AND P-302 PIPING DESIGNED TO HANDLE PRESSURE RUN LIGHT ON P-302	PROVIDE FLANGE COVERS FOR CAUSTIC SERVICE		YES	8/2/95	G113	IAT - DTL DSN
						LOCATE SAFETY STATION CLOSE TO P-302		YES	8/2/95	G214	IAT - DTL DSN
		NEUTRALIZATION LOOP	3 WAY IN WRONG POSITION PUMP FAILS VALVES CLOSED BLOCKED LINE	SEND LOW PH MATERIAL TO WWT UNABLE TO NEUTRALIZE CONDENSATE	RUN LIGHT ON P-301 TEMPERATURE INDICATORS ON ON TANK AN LOOP LEVEL INDICATION ON V-301 PRESSURE INDICATION ON P-301 DISCHARGE.	ADD INTERLOCK TO PREVENT DIVERTING TO WWT UNLESS PH IN TANK IS IN ACCEPTABLE RANGE		YES	8/2/95	1215	INTERLOCK
						ADD ROTAMETER OR BULLSEYE TO CIRCULATION LOOP		YES	8/2/95	1216	INTERLOCK
			LINE RUPTURE	PERSONNEL / ENVIRONMENTAL EXPOSURE POTENTIAL EQUIPMENT DAMAGE	PIPING DESIGNED TO HANDLE PRESSURE PSVS ON P-301 AND V-301						
		TO WWT	PUMP FAILS VALVES CLOSED TANK EMPTY VALVE IN WRONG POSITION	UNIT UPSET - NO HAZARDS							
			LINE RUPTURE	PERSONNEL / ENVIRONMENTAL EXPOSURE	PIPING DESIGNED TO HANDLE PRESSURE.						
		N2 TO V-301	VALVE CLOSED LOSS OF NITROGEN PRESSURE PLUGGED LINE	CAN'T PURGE ORGANICS FROM WATER	FLOW AND PRESSURE INDICATION ON NITROGEN LINE FIC ON NITROGEN BLOCK VALVE	ADD VOC ANALYZER ON VENT LINE FROM V-301		YES	8/23/94	G095	4/20/95
			LINE RUPTURE	VENT TO ATM DRAIN WATER FROM V-301	LEVEL TRANSMITTER ON V-301 FLOW INDICATOR ON N2 LINE PIPING DESIGNED TO HANDLE PRESSURE						
	MORE Flow	WATER TO V-301 FROM V-303	CONDENSE EXCESS WATER IN E-301 XV BETWEEN TANKS OPEN OR LEAKS	HIGH LEVEL IN V-301 OR V-303	LEVEL TRANSMITTERS AND HIGH LEVEL ALARMS ON V-301 AND V-303 MANUAL VALVE ON V-303	SET CV OF VALVE TO DELIVER REASONABLE FLOW FROM V-303 TO V-301		YES	8/2/95	1217	IAT - DTL DSN
		CAUSTIC TO E-304	CONTROL FAILURE. OPERATOR ERROR. NO CONDENSATE FLOW.	EXOTHERM IN V-301	PH METER ON DISCHARGE OF E-304 TEMPERATURE INDICATION ON LINE AND IN V-301						
		NITROGEN	PCV FAILS	POSSIBLY OVERPRESSURE V-301.	VENT TO CARBON BED IS CSO PSV ON V-301 WILL BE SIZED FOR REGULATOR FAILURE	CONFIRM MAX NITROGEN FLOW RATE DOES NOT EXCEED ENTRAINMENT VELOCITY FOR V-301		YES	8/2/95	1218	IAT - DTL DSN
	REVERSE Flow	V-301 TO V-303	CONSIDERED UNLIKELY.			ADD INTERLOCK TO PREVENT STARTING P-302 UNLESS XV-309 IS CLOSED		NO	-	-	
						ADD INTERLOCK TO SHUTDOWN P-302 ON HIGH LEVEL IN V-301		YES	8/23/94	1016	INTERLOCK
		P-301 TO P-302	RUNNING P-301 WHILE P-302 IS OFF	CONTAMINATE CAUSTIC EXOTHERM IN CAUSTIC LINE AND DRUM	INTERNAL CHECK VALVE(S) IN P-302	ADD CHECK VALVE IN LINE		YES	8/2/95	1219	8/2/95
		NITROGEN	SEE LINE RUPTURE ABOVE								
6.2	MORE Temp	SYSTEM	LOSS OF COOLING IN E-301 OR E-304	INCREASED TEMPERATURE AND PRESSURE IN V-301 POSSIBLE BOILING UPON ADDITION OF CAUSTIC POSSIBLY SEND HOT WATER TO WWT	TEMPERATURE INDICATION AND HIGH HIGH TEMPERATURE SWITCH ON E-301 AND E-304 DISCHARGE TEMPERATURE INDICATION ON V-301 WITH HIGH ALARM	ADD INTERLOCK TO SHUTDOWN UNIT ON TSHH 302 TRIP		YES	8/23/94	1017	INTERLOCK
						ADD INTERLOCK TO PREVENT DIVERTING TO WWT UNLESS EXIT TEMPERATURE OF E-304 IS IN ACCEPTABLE RANGE		YES	8/2/95	1220	INTERLOCK
						ADD INTERLOCK TO STOP CAUSTIC FLOW ON HIGH HIGH TEMPERATURE ON E-304 EXIT		YES	8/2/95	1221	INTERLOCK
			EXTERNAL FIRE	EQUIPMENT DAMAGE	PSVS ON ALL VESSELS	RESOLVE FIRE PROTECTION REQUIREMENTS- SEE SYSTEM #6		YES	8/23/94	G105	DEFER TO SRS PHA
		CAUSTIC TO E-304	HIGH AMBIENT TEMPERATURE	NONE IDENTIFIED							

COMPANY Delphi Research, Inc -- Proprietary Information
PROJECT METC Wet Oxidation Project
SYSTEM NUMBER 6
SYSTEM NAME CONDENSATE SYSTEM
PAID [illegible]
COLOR PURPLE

DESIGN HAZARD AND OPERABILITY REVIEW

REVISION [illegible]
9 Aug 93
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ITEM NO	GUIDE WORD VARIABLE DEVIATION	SUB-VARIABLE	CAUSES	CONSEQUENCES	EXISTING SYSTEMS/ SAFEGUARD	QUESTION OR ACTION RECOMMENDED	ACTION BY	INCORPORATE INTO DESIGN?	DATE APPROVED	RECOMMEND NUMBER	DATE IMPLEMENTED
	LESS Temp	SYSTEM	LOW AMBIENT TEMP.	FREEZE LINE- SEE NO FLOW ABOVE		DESIGN TO ALLOW SPACE FOR FIELD TRACING AT A LATER TIME		NO			
		CAUSTIC	LOW AMBIENT TEMP	FREEZE LINE- SEE NO FLOW ABOVE		HEAT TRACE TO 40 DEG F HEAT TRACE TO 35 DEG F		YES YES	6/23/94 8/2/95	G016 G211	4/20/95 8/2/95
6.3	MORE Flow	V-301	SEE MORE PRESSURE SYSTEM #4 SEE MORE FLOW ABOVE								
		V-301	VENT VALVE CLOSED	OVERPRESSURE VESSEL	VENT VALVE CSO PSV ON V-301						
		NEUTRALIZATION LOOP	SEE LESS FLOW ABOVE								
	LESS Pres	SYSTEM	NO NEW HAZARDS IDENTIFIED								
	REVERSE Flow	E-301 / V-301	CLOSE PRESSURE CONTROL VALVE - REACTOR COOLS	PULL VACUUM ON OVERHEAD SYSTEM	VACUUM PROTECTION ON REACTORS E-301 RATED FOR -10" WC	RATE V-301 FOR 10" WC VACUUM SERVICE		YES	8/2/95	G117	8/2/95
		V-301	BLOCK VALVES CLOSED WHILE PUMPING OUT	COLLAPSE TANK	BLOCK VALVE CSO						
			PLUGGED LINE OR CARBON BED DURING PUMPOUT	CONSIDERED UNLIKELY		SOP TO INSPECT VENT LINES PERIODICALLY		YES	6/23/94	1019	SOP
		CAUSTIC DRUM	VENT BUNG HOLE CLOSED DURING PUMPOUT	COLLAPSED DRUM PERSONNEL / ENVIRONMENTAL EXPOSURE		SOP TO INSURE TOP BUNG HOLE OPEN PLACE DRUM IN UPRIGHT POSITION TO MINIMIZE HANDLING - ADD DIPTUBE WITH FOOT VALVE		YES - [illegible] YES	6/23/94 6/23/94	1020 G116	SOP 4/20/95
6.4	NO/LESS Level	V-303	VALVE BETWEEN V-301 & V-303 LEFT OPEN NO CONDENSATE	HIGH PRESSURE VAPORS TO V-301	LEVEL TRANSMITTER IN V-303 LEVEL TRANSMITTER IN V-301 PRESSURE INDICATION AT CARBON BEDS	ADD INTERLOCK TO LIMIT WHEN BLOCK VALVE CAN BE OPEN SIZE PSV ON V-301 TO HANDLE MAX VAPOR FLOW		YES YES	8/2/95 8/2/95	1224 1225	INTERLOCK INTERLOCK
		V-301	TANK EMPTY	DAMAGE PH SENSOR		PLACE PH SENSOR BELOW LOWER LEVEL TAP		YES	6/23/94	G118	IAT - DTL DSN
		CAUSTIC DRUM	DRUM EMPTY - SEE NO FLOW ABOVE								
	MORE Level	V-303	UNIT UPSET OPERATOR ERROR NOTE THAT UNDER NORMAL OPERATIONS RUNNING TBP, V-303 SHOULD FILL UP EVERY HOUR	SEE MORE PRESSURE, SYSTEM 4		DOUBLE VOLUME OF V-303, BUT SET THE HIGH ALARM AROUND 50% FULL TO ALLOW SURGE CAPACITY DURING UNIT UPSET		YES	6/23/94	G119	IAT - DTL DSN
		V-301	CONTROLS FAILURE OPERATOR ERROR LEAKING VALVE	SEE ABOVE							
6.5	MORE Comp	WATER TO V-301	HIGH ORGANICS IN WATER.	INCREASED EMISSIONS	CARBON BED VOC ANALYZER ON VENT LINE N2 SPARGE LINE ON V-301 TEMPERATURE INDICATION DOWNSTREAM OF E-301						
	OTHER THAN Comp	CAUSTIC DRUM	WRONG DRUM HOOKUP	CONTAMINATION OF NEUTRALIZATION SYSTEM UNIT UPSET		SOP TO CONFIRM CAUSTIC IN DRUM BEFORE HOOKUP SOP TO CONFIRM PH IS RISING WHEN ADDING NaOH		YES YES	6/23/94 6/23/94	1501 1502	SOP SOP
		CONDENSATE TO WWT	OVER NEUTRALIZE UNDER NEUTRALIZE	HIGH PH LOW PH UPSET WASTEWATER SYSTEM	pH METER ON V-301 pH METER ON LINE TO WWT	ADD LOCAL PH INDICATION ON V-301 ADD REDUNDANT PH METER CONSIDER 25% CAUSTIC SUPPLY SOURCE CONSIDER VARIABLE STROKE NEUTRALIZATION PUMP OR COMPUTER CONTROLLED NEUTRALIZATION PACKAGE		YES NO - PULL SAMPLES NO - [illegible] YES - [illegible]	6/23/94 6/23/94	G114 G126	4/20/95 8/2/95
6.6	MORE / LESS Viscosity		SEE TEMPERATURE AND LEVEL SECTIONS ABOVE								
6.7	COOLING/HEATING	E-304	SEE MORE / LESS TEMP ABOVE								
6.8	REACTION	V-301	SEE ABOVE								
6.9	AGITATION	V-301	NO MIXING	NO HAZARDS - MIXING ACCOMPLISHED IN NEUTRALIZATION LOOP.							
6.10	IGNITION SOURCE		SPARKS FROM MOTOR(S)		ALL MOTORS SPECIFIED FOR FOR CLASS I DIV2 GROUP C & D						
			STATIC SPARKS FROM DRUMS	POSSIBLE FIRE		ALL EQUIPMENT AND PIPING TO BE GROUNDED USE NON SPARKING DRUM CONNECTORS IN DESIGN		YES YES	6/23/94 6/23/94	G006 G028	IAT - DTL DSN 4/20/95
6.11	HEAT EXCH LEAKS	E-304	CORROSION SPLIT TUBE SHELL RUPTURE	CONTAMINATION OF OIL OR PROCESS - CONSIDERED UNLIKELY.	EXCHANGER RATED FOR MAX TEMP & PRESSURE LEVEL INDICATION ON V-301	ADD AREA HCl MONITOR ADD CONDUCTIVITY ANALYZER ON OIL SYSTEM		YES NO - UNLIKELY	6/23/94	G013	4/20/95
6.12	THERMAL EXPANSION	E-304				DESIGN TO INCLUDE THERMAL RELIEF		YES	6/23/94	G038	4/20/95

APPENDIX C

PREOPERATIONAL
PROCESS HAZARDS REVIEW
ACCIDENT CRITERIA

Category	Criteria
A. Personnel Safety:	
1. Bodily Injury	Fatality
2. Radiation Exposure	
a On plant	5 rem (external plus committed effective dose equivalent).
b Off plant	500 mrem (All pathways). Atmospheric release: 25 mrem/year to individual. Liquid release: 4 mrem/year to individual.
3. Toxic Material	
a Onsite	Immediately Dangerous to Life or Health (IDLH).
b Offsite	Emergency Action Limit.
B. Loss of Equipment or Facilities	$1,000,000 (includes clean up costs).
C. Nuclear Safety	Uncontrolled Nuclear Criticality
D. Loss of Production	Six Months or more

APPENDIX D

PREOPERATIONAL
PROCESS HAZARDS REVIEW
"WHAT IF" EXAMINATION RECORD

PREOPERATIONAL PROCESS HAZARDS REVIEW
"WHAT-IF" EXAMINATION RECORD

HAZARD	WHAT IF ___?	CONSEQUENCES	MAY EXCEED WHAT CRITERIA?	EXISTING PROTECTION	ACTION ITEMS OR RECOMMENDATIONS
Chemical Splash	Low flow or low pressure on skid safety showers	Personal injury (burns)	None	• 3 safety showers • building safety showers	• Ensure sufficient flow and pressure available and tested
Steam	Steam leak/hot Pipes	Personnel injury	None	• Insulated piping • Procedures • Limited steam use	• Ensure remote shut-off available • WSRC to provide steam trap and drain
Lightening	Lightening strikes equipment	Personnel Injury Equipment damage	None		• Have technical experts determine need for lightening suppression • Check outside components (Heat-x, Gas supplies, etc.)
Fire	Fire starts anywhere in building	Personnel injury	None	Fire extinguishers Emergency response	• Perform FHA and respond accordingly
Grounding	Insufficient grounding causing potential between skid and building	Personnel injury Equipment damage	None		• WSRC responsible for adequate grounding
No Communication (TNX-Delphi)	Situation arises requiring interaction with EP or evacuation/shelter/ accountability	Personnel injury	None	PA system	• Require Delphi operators to attend TNX Ops Mtgs. • Brief Delphi operators of TNX projects in 673-T (vice-versa) • Training • Radios • Phones • More PA speakers

Dark	Loss of electrical power - cannot find egress	Personnel injury	None		• Ensure battery egress lighting • Operators carry lights
No communication (skid-trailer)	Situation arises requiring fast action between trailer and skid	Personnel injury Equipment damage	None		• PA system interface • Consider radios
Hazardous chemical	Chemical spill causes vapor or gas harmful to personnel	Personnel injury	None	• EP Procedures • Training	• WSRC provide spill kit • Evaluate building ventilation requirements
Hazardous Chemical	Chemicals mix to form non-evaluated chemical	Personnel injury	A1	• Chemical inventory evaluated	• (A) Ensure list evaluated prior to new chemical additions to inventory

Appendix G – Fire Hazards Analysis

F-FHA-T-00003
Revision 0

Fire Hazards Analysis
for
Building 673-T Delphi DETOX^{sm} Process, CR-95-011 (U)

Does Not Contain
Unclassified Controlled Nuclear Information (UCNI)

Prepared By:

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Senior Engineer - WSRC TNX Operations Section

Fire Protection Program Department Review:

Khoi V. Vo

Khoi V. Vo
Senior Fire Protection Engineer - WSRC Fire Protection Program Department

APPROVALS:

DLBooth

D. L. Booth
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M. A. Fachada
Principal Investigator - Delphi CRADA

UCNI Reviewing Official

7/22/97

EXECUTIVE SUMMARY

This document is a Fire Hazards Analysis for Building 673-T, but is limited in scope to the impact of the new Delphi DETOX^{sm} Process.

The analysis reviewed the construction features, combustible loading, and process hazards to determine the potential fire scenario, and the building and equipment values and damage estimates to determine the resulting fire loss. The evaluation determined that the maximum possible fire loss is less than $10 million, and therefore automatic sprinklers are not required by DOE Order.

The electrical classification of the building was reviewed and a section of the building high bay area will now require Class 1, Division 2 electrical equipment.

Deficiencies were noted in this review and are listed in section 4.2 of this FHA.

Items affecting this analysis and yet to be installed are listed in section 4.3 of this FHA.

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REVISION SUMMARY

Revision Number	Revision Description
A	Initial draft issued for review.
0	Document issued for approval.

1.0 INTRODUCTION

Building 673-T is a pilot process facility used for Defense Waste Process Facility Canister studies and the Delphi Research, Inc. DETOX℠ process. This building is not defined as "vital" to the DOE Savannah River mission.

The objective of this Fire Hazards Analysis (FHA) is to evaluate the impact of the Delphi DETOX℠ installation on Building 673-T. It also determines the maximum possible fire loss (MFPL) for the building.

This evaluation is based on current national codes and standards. This document treats portions of the building modified by installation of Delphi DETOX℠ equipment as "new" as defined by the national codes and standards. Other portions of the building are treated as "existing".

This FHA is based on the current process and proposed modifications defined in available design documents. Any process or design modifications made after the issue of this document will require additional review and, if required by the review, revision to this document.

1.1 Limitations

The scope of this FHA is limited to the major items required to support the Delphi DETOX℠ process. To the extent practical, the content required for a standard FHA was included, but any analysis outside the scope limitations was not carried out which is consistent with a graded approach for commercial-type operations at the Savannah River Site. The following limitations apply to the scope of this document.

- The impact of the Delphi DETOX℠ installation on the existing facility.
- The current MPFL values for the building.
- The electrical classification of the building and Delphi DETOX℠ process area.

The analysis of the modifications is based on the available design documents. The proposed changes are compared to the existing facility to determine the impact of these changes. The new designs are also compared to the requirements found in the codes and standards regarding fire protection.

1.2 Assumptions

This FHA assumes that all currently installed fire protection equipment is fully operational. A detailed evaluation of existing systems and equipment was not made. Facility equipment is inspected and tested on periodic basis by the facility and SRS Digital Controls & Systems personnel to confirm this assumption. This FHA also assumes that fire protection equipment installed on the Delphi DETOX℠ modules is operational and will be inspected or tested in compliance with applicable codes and standards.

This FHA assumes that the following NFPA Codes will be used in applicable operations for the Delphi DETOX℠ process.

- NFPA 10, Portable Fire Extinguishers
- NFPA 13, Installation of Sprinkler Systems
- NFPA 30, Flammable and Combustible Liquids Code
- NFPA 50, Bulk Oxygen Systems at Consumer Sites
- NFPA 55, Compressed and Liquefied Gases in Portable Cylinders
- NFPA 70, National Electric Code
- NFPA 101®, Safety to Life from Fire in Buildings and Structures
- NFPA 111, Stored Electrical Energy Emergency and Standby Power Systems
- NFPA 780, Installation of Lightning Protection Systems

1.3 Fire Severity, Calculations, and Fire Modeling Bases

No special fire modeling methods were used in this analysis. Fire spread was determined by relative severity based on combustible loading in various areas. Walls were assumed to inhibit, but not prevent, the spread of fire.

Replacement values for the existing building were determined from the Investment and Replacement Value and Plant and Equipment Replacement Costs data base. The estimated project cost for equipment and construction determined the value added to the building. The replacement cost estimated for high value equipment was used to allocate the total cost of the facility to certain areas. This allocation of cost formed the basis for determining the loss estimates from different fire scenarios. Table 1 lists the estimates used in the determination.

Table 1 - Replacement Cost Estimates

	Replacement Cost of Building	Replacement Cost of Contents	Total Replacement Cost
Existing Facility	$1,321,002	N/A	$1,321,002
Delphi DETOX℠ Process	N/A	$4,500,000	$4,500,000
Delphi Laboratory Trailer	$25,000	$225,000	$250,000
Oil Heating / Chiller Unit	N/A	$250,000	$250,000
Effluent Water Tanks	N/A	$25,000	$25,000

2.0 FACILITY DESCRIPTION, CLASSIFICATION, AND PROCESSES

This section provides an overview of the existing facility and the proposed changes by the installation of the Delphi DETOX℠ process. This section does not include analyses of fire protection features.

2.1 General Description and Location

2.1.1 Building 673-T

2.1.1.1 General Information

Building 673-T is a single story structure measuring 62 feet x 81 feet x 50 feet high. The pitched roof and exterior walls are constructed of corrugated sheet metal supported by a steel frame on a concrete pad. There is no access to the roof. The facility is centrally located in the TNX area as shown in Attachment A.

The office area contains typical amounts of Class A materials such as office furniture, computers, printers, paper, and file cabinets. There are no flammable chemicals, gases, or open flames used in this area. The combustible loading of this area is "Ordinary" based on the Hazard Categories as listed in Table A1-2 of the WSRC FHA guideline. The facility does not contain an automatic suppression system. The office room is the only area monitored by a smoke detection system.

2.1.1.2 Construction Classification

The facility meets the requirements of Type II-N as outlined in Table 17-A of the Uniform Building Code. This is equivalent to Type II(000) in NFPA 220.

2.1.2 Delphi Laboratory Trailer

2.1.2.1 General Information

The Delphi Laboratory Trailer is a single story structure measuring 12 feet x 24 feet x 8 feet high. The pitched roof and exterior walls are constructed of wood supported by a wood frame approximately 3 feet above grade. There is no access to the roof. The structure is located north of Building 673-T.

The office area contains typical amounts of Class A materials such as office furniture, computers, printers, paper, and file cabinets. There are no flammable gases or open flames used in this area. Approximately 4 ml of a methanol-based chemical will be stored in a flammable liquid storage cabinet for infrequent use in calibration of laboratory equipment. The combustible loading of this area is "Ordinary" based on the Hazard Categories as listed in Table A1-2 of the WSRC FHA guideline. The facility does not contain an automatic suppression system or a detection system.

2.1.2.2 Construction Classification

The facility meets the requirements of Type V-N as outlined in Table 17-A of the Uniform Building Code. This is equivalent to Type V(000) in NFPA 220.

2.1.3 Delphi DETOX^{sm} Oil Heating / Chiller Unit

2.1.3.1 General Information

The Delphi DETOX^{sm} Oil Heating / Chiller Unit (module #3) is a single story structure measuring 18 feet x 9 feet x 11 feet high. The unit has no exterior walls or roof. The equipment is constructed of metal supported by a metal frame on a concrete pad. The structure is located west of Building 673-T.

Located on the top of module #3 is the heat exchanger unit measuring 25 feet x 8 feet x 10 feet high. This metal contains a cooling coil body with two fan units passing air upward across the coils.

The oil chiller is a single level one piece unit mounted to the concrete slab north of module #3. The unit measures 22 feet x 7 feet x 5 feet high and is constructed of metal.

Module #3 contains no combustible materials. There are no flammable chemicals, gases, or open flames used in this area; however, Dow Chemicals SYLTHERM® 800 oil, a non-conducting fluid with the potential to build-up a static electric charge, is used in the process. Additionally, during portions of operation, the SYLTHERM® 800 oil (flashpoint - 320° F) is heated to 500° F. The current design does not provide for bonding or grounding of pipes containing SYLTHERM® 800 oil above its flashpoint as required by NFPA 30.

The combustible loading of this area is low based on the Hazard Categories as listed in Table A1-2 of the WSRC FHA guideline. The facility does not contain an automatic suppression system or a detection system.

2.1.3.2 Construction Classification

The facility meets the requirements of Type II-N as outlined in Table 17-A of the Uniform Building Code. This is equivalent to Type II(000) in NFPA 220.

2.1.4 Occupancy and Hazard Classification

The hazard class designation in WSRC-TR-94-0392, TNX Area Hazards Assessment, is Non-Nuclear, Low Hazard for the entire building.

These facilities are classified as a special purpose industrial occupancy as defined in NFPA 101®, Chapter 28.

The fire hazard class, as defined in NFPA 13, is Ordinary Hazard, Group 1.

The area within 5 feet around the liquid transfer line to the reactor (including the space directly under this 5 foot area) is classified as Class 1, Division 2, Groups C & D electrical for a hazardous area as defined by NFPA 70, Article 500. The area 25 feet horizontally from this transfer line at or below 3 feet from the floor is also classified as Class 1, Division 2, Group D electrical for a hazardous area as defined by NFPA 70, Article 500.

At each of the oil lines that can contain SYLTHERM® 800 oil at temperatures above its flashpoint, the following areas near potential leak points (flanges, valves, pumps, threaded connections, etc.) are classified as Class 1, Division 2 electrical for hazardous areas as defined by NFPA 70, Article 500.

- Inside Building 673-T

  The area within 5 feet around the above oil lines (including the space directly under this 5 foot area). The area 25 feet horizontally from these oil lines at or below 3 feet from the floor.

- Outdoors

  The area within 3 feet around the above oil lines (including the space directly under this 3 foot area). The area 10 feet horizontally from these oil lines at or below 18 inches above grade.

2.2 Facility Operations and Processes

Building 673-T is a multi-use facility for both DWPF containerization equipment development and the Delphi DETOX℠ Process. It houses a process area, an office area, and an abandoned telecommunications room located in the northeast corner. The process area is used for cutting non-radioactive DWPF glass canisters and the operation of the Delphi DETOX℠ process.

A portable laboratory / office trailer is located approximately 10 feet north of Building 673-T. This facility will be used to analyze samples from the Delphi DETOX℠ process.

A separate Dow Chemicals SYLTHERM® 800 oil heating / chiller unit is installed approximately 35 feet west of Building 673-T. This unit is designed to heat the SYLTHERM® 800 oil to 500° F and pumps it to the heating coils in the Delphi DETOX℠ reactor to facilitate the initial reaction. Once the reaction begins, the unit will provide chilled oil to the reactor cooling coils to remove unwanted heat from the process.

These facilities do not contain nuclear process systems, critical or high value equipment, or safety class components. They are normally occupied during daily business hours. When the Delphi DETOX℠ process is in operation, these facilities will be occupied 24 hours per day, five days a week. The maximum number of occupants for Building 673-T during normal operations is estimated to be 10 people.

The maximum number of occupants for the laboratory trailer is estimated to be 3 people.

2.3 Facility Information

2.3.1 Utilities and Services

Substations located south of the building supply primary power to the building. The power is supplied to the Building 673-T Motor Control Center (MCC) located in the southwest corner of the building, which contains both primary power panels and standby power panels. Power to the Delphi DETOX℠ process is supplied from substation 652-22T breaker 3B directly to the Delphi DETOX℠ process MCC building located in the north bay area.

Power to the laboratory trailer is supplied through the Delphi DETOX℠ process MCC building which contains a Uninterruptible Power Supply (UPS) system for backup power for the Delphi DETOX℠ process.

2.3.2 Special Conditions or Hazards

As given in a fax from Delphi Research, Inc., dated 4/22/97, normal "dry" feed runs of the Delphi DETOX℠ process will consist of ordinary combustible items (plastics, rubber, etc.) placed in a 55 gallon drum with a closed, 500 milliliter vial containing 350 milliliters of surrogate materials used to simulate contaminants (approximately 41 grams Naphthalene, 105 grams Chlorobenzene, and 105 grams Dichlorobenzene). The drum is physically transferred to the third level of module #1 and dumped into a grinder hopper with a hood vented to the process off gas system. Upon startup of the grinder (which breaks the vial and mixes the components), a water spray covers this surrogate feed mixture. The mixture is then transferred to the reactor vessel for processing.

During normal "wet" feed runs, a 55 gallon drum of pre-mixed acetone / toluene solution is connected to the closed wet feed transfer system and the solution pumped directly to the reactor vessel for processing.

As part of the feed stock solution, mineral oil is added at ambient temperature through the wet feed transfer system to the reactor.

Per the current design, 2 pressurized gas cylinders will be located northwest module #2 on the wall of the high bay area. These cylinders will contain non-flammable gas used for instrument calibration.

During processing and neutralization activities, heated caustic solution is added at 140° F downstream of the overhead distillate tank to stabilize pH in the system.

During processing, trace amounts of VOC's will be released into the DETOX℠ vent system. However, these amounts will be well below hazard levels and system monitors will be installed requiring process shut down should high VOC levels be encountered.

As part of the process, a 6000 gallon liquid oxygen tank will be installed outdoors and east of the building. Per the current design, oxygen will be supplied to the Delphi DETOX℠ reactor at 3300 SCFH using 1-1/2" schedule 80 stainless steel pipe. This oxygen supply will be used to regenerate the iron oxidant inside the reactor vessel.

As a supplemental safety system, 100 gallons of water secured in an overhead tank is designed to automatically quench the reactor vessel in the event that a high temperature is attained, thereby stopping the reaction process when the limit signal is received by the control valve on the quench tank drain line. This quench system also contains a manual backup. In addition, a rupture disk is installed on the reactor vessel should excessive pressure build up.

Backup power for the unit is provided with batteries in a UPS configuration. Hydrogen generation calculations have been performed showing adequate ventilation in the MCC to maintain hydrogen concentrations below lower explosive limits.

3.0 FIRE PROTECTION ANALYSIS

This section describes the facility's existing fire protection features and systems. The scope of this FHA does not include a detailed design review.

3.1 Fire Area Definition

Because this facility contains no passive fire protection features, the entire building is considered one fire area. The portable laboratory module located north of the building and the Delphi DETOX℠ oil heater / chiller unit (module #3) are installed at appropriate separation distances from the facility and are therefore considered separate fire areas.

3.2 Fire Area Analysis

3.2.1 Building 673-T

3.2.1.1 Fixed Automatic Systems

There is no automatic suppression installed in this facility.

3.2.1.2 Manual Systems

Portable dry chemical fire extinguishers are located throughout the building proper in accordance with NFPA 10. Facility personnel inspect these extinguishers monthly and the SRS fire department inspects them annually.

Portable dry chemical fire extinguishers will be located throughout the Delphi DETOX℠ modules in accordance with NFPA 10. According to the current revision of the Delphi Safety Manual, Delphi personnel shall inspect these extinguishers monthly and the SRS fire department will inspect them annually.

3.2.1.3 Detection and Alarm Systems

Smoke detection is installed in the facility office area, covering approximately 400 ft^2. There are 2 pull stations located at the north and east exits from the facility.

3.2.1.4 Passive Protection and Features

There are no passive fire protection features installed within this facility.

3.2.1.5 Smoke and Heat Ventilation

There are no smoke and heat ventilation features installed within this facility.

3.2.2 Delphi Laboratory Trailer

3.2.2.1 Fixed Automatic Systems

There is no automatic suppression installed in this facility.

3.2.2.2 Manual Systems

Portable dry chemical fire extinguishers will be located throughout the building in accordance with NFPA 10. According to the current revision of the Delphi Safety Manual, Delphi personnel shall inspect these extinguishers monthly in accordance with NFPA 10 and the SRS fire department will be notified to inspect them annually.

3.2.2.3 Detection and Alarm Systems

There is no smoke detection system or manual pull station installed in this facility as personnel will be warned by the fire itself due to the small size of the building.

3.2.2.4 Passive Protection and Features

There are no passive fire protection features installed within this facility.

3.2.2.5 Smoke and Heat Ventilation

There are no smoke and heat ventilation features installed within this facility.

3.3 Water Supply and Manual Fire Attack

3.3.1 Water Supply Analysis

Hydrant #3007 is located approximately 45 feet southeast of the facility. Hydrant #3008 is located approximately 130 feet northeast of the facility. Each of these hydrants is supplied by the 8 inch area compound loop distribution system. Water is supplied to the system via a diesel fire pump rated for 1000 GPM at 100 psi. Flow tests on November 20, 1996 show that the pump can provide 1500 GPM at 65 psi. The fire water supply is tested weekly by facility personnel and is properly maintained.

3.3.2 Manual Attack and Emergency Response

The F-Area Fire Station provides the primary response to a fire alarm at TNX, with the L-Area Fire Station as the secondary response. The estimated response times from these areas are approximately 18 minutes. The first unit will respond with a single pumper truck with a 1250 GPM pump and a 1000 gallon tank. Other support vehicles respond according to the type of alarm.

The Building 673-T facility custodian is trained as a fire warden. During operations, the Delphi DETOX℠ unit will be continuously manned.

3.4 Life Safety Analysis

This section deals with life safety features and components in the facility. It includes only the significant features observed.

3.4.1 Building 673-T

3.4.1.1 Occupancy Classification and Occupant Loading

Building 673-T is classified as a Special Purpose Industrial Occupancy. In accordance with NFPA 101®, the calculated occupant load is 50 people, which exceeds the estimated maximum occupant load of 10 people.

3.4.1.2 Stairs and Vertical Exits

Access to the upper levels of Delphi module # 2 and the north section of Delphi module #1 is accomplished through stairs located on the north edge of the unit. Most of these stairs meet the minimum NFPA 101® requirements. However, the stairs from the second level of module # 2 to the third level of module # 1 have deficiencies as noted in section 4.2.

According to the design sketch in the 4/17/97 memorandum from Delphi Research, Inc. to WSRC, access the south and west section of levels 2 and 3 on Delphi DETOX℠ module #1 is to be accomplished through one platform of temporary scaffolding with a ladder installed.

Since the means of egress on the north side of these platforms cannot be accessed from the south portion of the upper levels, the scaffolding for the south platform areas shall comply with NFPA 101®.

3.4.1.3 Door Widths, Horizontal Exits, Aisles, and Travel Distances

A. General Building

Building 673-T has 3 exits leading directly to the outside from all general areas of the building, providing an egress capacity of approximately 510 people using an average door width of 34 inches. Other than the east exit from the office area, there are no enclosed corridors. A containment wall with a 36 inch door has been placed over the north opening from the high bay area. On the building floor, the maximum common path of travel is less than 50 feet. Travel distance from the most remote point to the nearest exit is less than 200 feet.

All existing exit access routes and exits meet or exceed the minimum width of 28 inches.

B. Unit Modules

The common path of travel from the third level of module #1 to the bottom of the existing exit stairs is 83 feet, which is greater than the 50 feet allowed by NFPA 101®. Travel distance for the north exit from the module is less than 200 feet. Exit access routes and exits for the module meet or exceed the minimum width of 36 inches.

3.4.1.4 Exit Signs and Emergency Lighting

Each existing building exit has an internally illuminated sign as required by NFPA 101®.

Battery powered emergency lights are situated throughout the building floor area in accordance with NFPA 101®.

The current design utilizes the Delphi UPS to power modules #1 and #2 lights as emergency lighting. However, it is indeterminate as to whether or not NFPA 111 requirements were met in the design and installation of the UPS. In addition, the UPS will only supply power for 1 hour, not the required 90 minutes per NFPA 101®.

3.4.1.5 Occupant Notification

A public address system is installed throughout the building and personnel confirm that it can be heard in all areas.

The fire alarm has one buzzer in the process area located on the north high bay wall.

3.4.2 Delphi Laboratory Trailer

3.4.2.1 Occupancy Classification and Occupant Loading

The Delphi Laboratory Trailer is classified as a Special Purpose Industrial Occupancy. In accordance with NFPA 101®, the occupant load shall be the maximum number of persons to occupy the area under any probable conditions (10 people).

3.4.2.2 Stairs and Vertical Exits

Landings and stairs have been be provided for each exit from the building.

3.4.2.3 Door Widths, Horizontal Exits, Aisles, and Travel Distances

The laboratory has 2 exits leading directly to the outside. There are no enclosed corridors. The maximum common path of travel is less than 50 feet. Travel distance from the most remote point to the nearest exit is less than 200 feet. All exit access routes and exits meet or exceed the minimum width of 36 inches.

3.4.2.4 Exit Signs and Emergency Lighting

Because of the small size of the trailer and the means of egress are obvious to personnel, exit signs are not required by NFPA 101®.

The current design utilizes the Delphi UPS to power trailers lights as emergency lighting. However, it is indeterminate as to whether or not NFPA 111 requirements were met in the design and installation of the UPS. In addition, the UPS will only supply power for 1 hour, not the required 90 minutes per NFPA 101®.

3.4.2.5 Occupant Notification

In accordance with WSRC-PH-96-36, Pre-Operational Process Hazards Review for Delphi DETOX℠ Unit, Recommendation DD-R10, Delphi personnel shall be provided with radios and telephones. In addition, a speaker connected to the area PA system has been installed in the laboratory trailer.

3.5 Fire Exposure Analysis

This section only deals with the exposures to Building 673-T from new installations within the Delphi DETOX℠. These exposures involve the installation of the new Delphi Laboratory Trailer north of the facility, the Delphi DETOX℠ Oil Heating / Chiller Unit and the new oxygen tank installed east of the facility.

Per DOE-STD-1088-95, Fire Protection for Relocatable Structures, section 6.1, the required separation distance between this structure and Building 673-T of 10 feet has been met.

The Oil Heating / Chiller Unit is located 35 feet west of building 673-T. Dow Chemicals SYLTHERM® 800 oil is heated in this process to 500° F, which is well above the oil's flashpoint of >320° F. However, because of the unit's construction and low fire hazard, it presents no exposure to Building 673-T.

The oxygen tank will be installed approximately 10 feet from the outside east wall of Building 673-T, which is greater than the minimum required by NFPA 50. Therefore, this tank is not considered an exposure to Building 673-T.

3.6 Natural Phenomena Hazards Fire Impacts

Per the Delphi DETOX℠ Process Hazards Review, the only natural phenomena considered a threat to this facility is a lightning strike. To mitigate this potential difficulty, lightning protection shall be installed on Building 673-T. This lightning protection shall meet the requirements of NFPA 780, Installation of Lightning Protection Systems.

3.7 Review of Existing Fire Protection Findings

There are no open fire protection findings for this facility.

4.0 ANALYSIS RESULTS AND CONCLUSIONS

4.1 Summary of Analysis Results

The facility as designed has several deficiencies that must be resolved prior to operation of the DETOX℠ unit. Included are several means of egress issues involving stair configuration and common path of travel lengths. In addition, several applicable fire protection items yet to be installed have been identified.

4.2 Tabulation of Deficiencies

The items listed below were noted as deficient in this analysis. These items shall be resolved prior to operation of the Delphi DETOX℠ process.

4.2.1 Delphi DETOX℠ Unit Modules #1 and #2

- The common path of travel from the third level (north) of module #1 to the ground is 83 feet, exceeding the maximum of 50 feet allowed by NFPA 101® for special purpose industrial occupancies.

- The stairs between the second level of module #2 and the third level of module #1 do not meet the minimum requirements of NFPA 101® as follows.

Item	Requirement	Actual
Tread Depth	≥ 10"	9"
Handrail Height	34" to 38"	30"

- No complete design meeting NFPA 101® requirements exists for egress from the south platforms.

- The current design utilizes the Delphi UPS for emergency lighting; however, it is indeterminate as to whether or not NFPA 111 requirements were met in the design and installation this system, nor does the UPS provide 90 minutes of emergency lighting as required by NFPA 101®.

4.2.2 Delphi Portable Laboratory Trailer

- The current design utilizes the Delphi UPS for emergency lighting; however, it is indeterminate as to whether or not NFPA 111 requirements were met in the design and installation this system, nor does the UPS provide 90 minutes of emergency lighting as required by NFPA 101®.

4.2.3 Delphi DETOX℠ Oil Heating / Chiller Unit

- TNX Operations equipment in the electrical hazardous area around potential hot oil line leak points is not designed to Class 1, Division 2 requirements.

- Current design does not provide for bonding or grounding of pipes containing SYLTHERM® 800 oil above its flashpoint as required by NFPA 30.

4.3 Installation Items to be Completed

NOTE: Completion of the following items shall be considered to resolve the deficiencies listed in section 4.2.

4.3.1 Building 673-T

- Install lightning protection for the building in accordance with NFPA 780.

- Install portable dry chemical fire extinguishers throughout the Delphi DETOX℠ modules in accordance with NFPA 10.

- Issue a technical position paper regarding the module #1 third level stairs.

- Install scaffolding or other equipment complying with the requirements of NFPA 101® for access and egress from the south areas of module #1.

- Install a second means of egress complying with the requirements of NFPA 101® from the north areas of modules #1 and #2.

- Either verify that the Delphi UPS system is designed, installed, and will be maintained to NFPA 111 and will supply 90 minutes of emergency lighting as required in NFPA 101®, or install battery powered emergency lighting throughout the modules as given in NFPA 101®.

4.3.2 Delphi Portable Laboratory Trailer

- Install portable dry chemical fire extinguishers inside the trailer in accordance with NFPA 10.
- Install telephone lines to the trailer.
- Either verify that the Delphi UPS system is designed, installed, and will be maintained to NFPA 111 and will supply 90 minutes of emergency lighting as required in NFPA 101®, or install battery powered emergency lighting in the laboratory trailer as given in NFPA 101®.

4.3.3 Delphi DETOX℠ Oil Heating / Chiller Unit

- Install a wall break at the transformer south of the oil heating / chiller unit to separate this equipment from potential leaks at the piping flanges of lines containing SYLTHERM 800® above its flashpoint.
- Design and install bonding or grounding for pipes containing SYLTHERM® 800 oil above its flashpoint.

4.3.4 Delphi DETOX℠ Oxygen Tank

- Install the oxygen tank system per NFPA 50.

4.4 Conclusions

The Delphi DETOX℠ unit operation involves significant fire protection issues due to the presence of both flammable and combustible liquids in the process. However, these items can be mitigated through proper design and rigorous fire protection practices in this facility. Once appropriate design and practices are outlined and implemented by the operating agency, this system can be allowed to begin operations.

5.0 REFERENCES

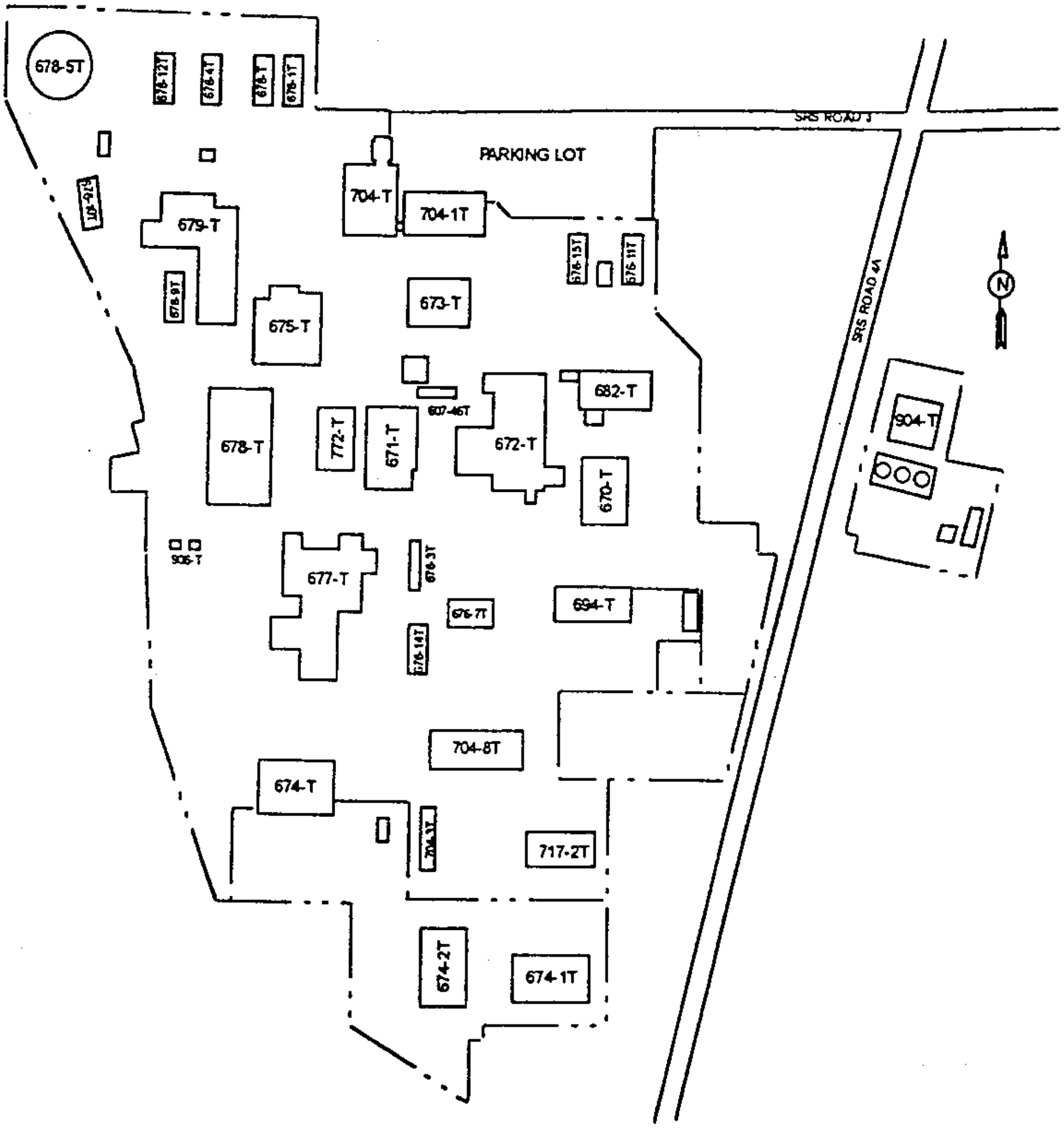
- WSRC-PH-96-36, Delphi DETOX℠ Process Hazards Review
- NFPA 10, Portable Fire Extinguishers
- NFPA 13, Installation of Sprinkler Systems
- NFPA 30, Flammable and Combustible Liquids Code
- NFPA 50, Bulk Oxygen Systems at Consumer Sites
- NFPA 55, Compressed and Liquefied Gases in Portable Cylinders
- NFPA 70, National Electric Code
- NFPA 101®, Safety to Life from Fire in Buildings and Structures
- NFPA 111, Stored Electrical Energy Emergency and Standby Power Systems
- NFPA 780, Installation of Lightning Protection Systems
- F-FHA-G-00001, Guideline for the Preparation of Fire Hazards Analyses for the Savannah River Site

6.0 APPENDICES

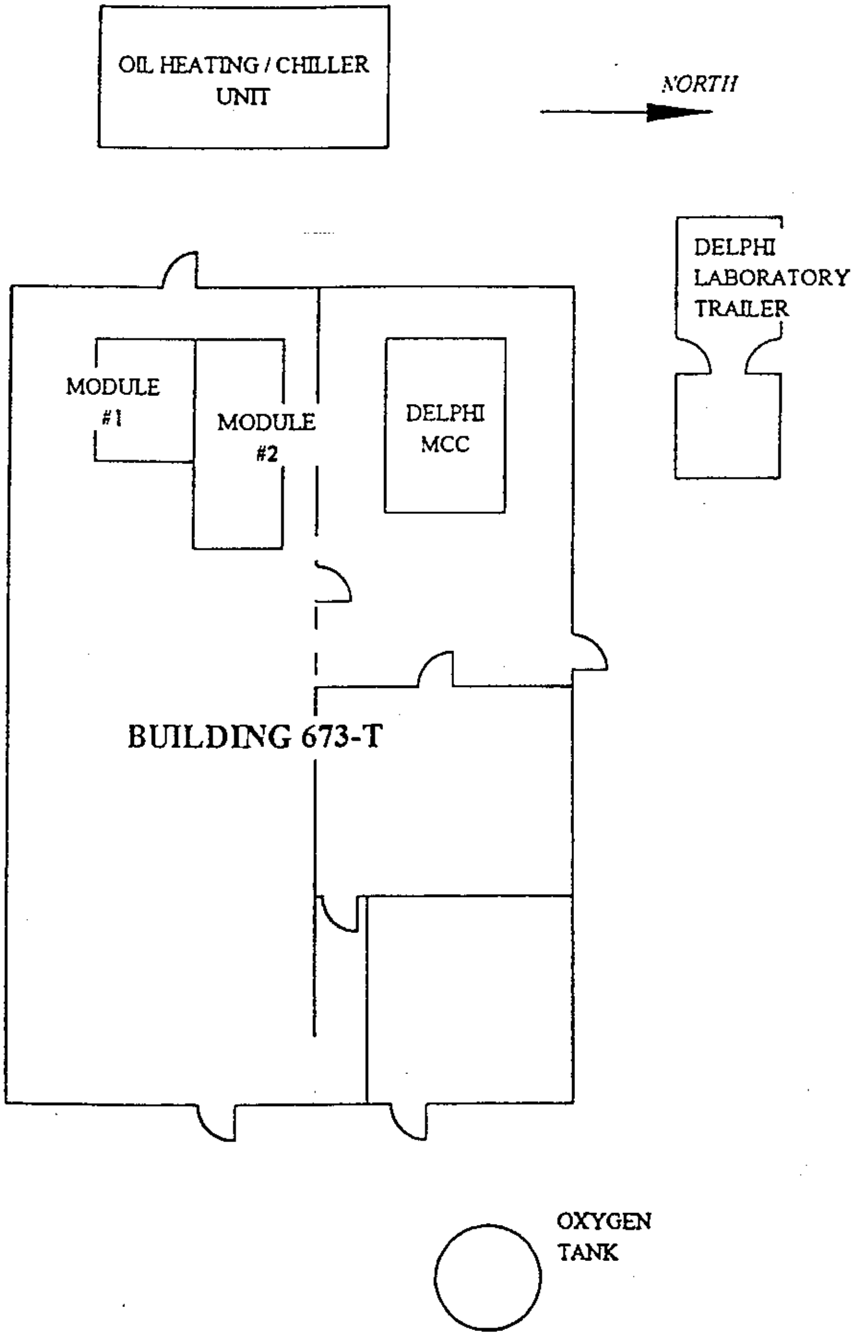
- Appendix A, TNX Area Map

- Appendix B, Delphi DETOX℠ Modules Layout

APPENDIX A
TNX Area Map



APPENDIX B
Delphi DETOX^{sm} Modules Layout



Appendix H – Validation Plan Objectives

1.0 Introduction and Objectives of Validation Program

Government and industry are actively seeking and supporting the development of new and innovative technologies for the management, prevention, and treatment of hazardous and toxic wastes. Interest in implementing new approaches and technologies is continually increasing due to an expanding understanding of the long- and short-term hazards associated with such wastes, the liabilities and costs of managing and treating the wastes, and public demand for improved protection of health and the environment.

One important step leading to the implementation of new and innovative waste treatment technologies is the demonstration of such technologies to generate reliable performance and applicability information. These demonstrations are typically conducted in the field with actual or surrogate wastes and are closely monitored to ensure the validity and reliability of data obtained.

Demonstrations of a new and innovative technology will be conducted at two Department of Energy (DOE) facilities in 1997 and 1998. The technology to be demonstrated, DETOX^{SM}, is a catalyzed wet oxidation technology which offers a non-combustion alternative for the oxidation of organic wastes as well as a method for extracting and accumulating toxic and radioactive metals from wastes. The technology, developed and patented by Delphi Research, Inc., will be demonstrated to assess its performance in treating a range of wastes and waste matrices representative of those found within the DOE complex.

All activities related to the demonstrations are being performed under an Industry Programs technology development contract managed by the DOE Federal Energy Technology Center (FETC) for the Environmental Management (EM) Office of Science and Technology Demonstration. Demonstration site needs are being supported by the Mixed Waste Focus Area (MWFA) and DOE EM site organizations.

During the performance of the DETOX^{SM} demonstrations, a third party validation will be conducted by Arthur D. Little, Inc. The objective and purpose of this validation are described in the box below.

A plan for the validation of the DETOX^{SM} technology is presented in this document. The Validation Plan includes a description of the approach for technology validation as well as background information relating to technology development, the technology demonstrations, and the roles of the various stakeholders in the implementation of the technology.

This Plan is intended to complement rather than duplicate existing documentation prepared by Delphi Research, Inc., including the Demonstration Test Plan (October 22, 1996) and the Sampling and Analysis Plan (October 25, 1996 and January 16, 1997-revised). Specific details regarding the technology, conduct of the demonstration, and coordination of stakeholders are found in those plans.

Validation Program Objective

An independent engineering, technical, permitting, and safety assessment of the integrated DETOX^{SM} process will be performed to confirm various technology characteristics relating to performance (including system integrity, reliability, and maintainability) environmental compliance, safety, and cost. The results of the validation may be used to assist in the removal of barriers to technology implementation and to facilitate acceptance of the technology by potential users and state and federal regulatory agencies.

Appendix I – Milestone Schedule Plan

U. S. Dep. . of Energy

Milestone Schedule ( ) Plan (x) Status Report

1. Title
Development of a Novel Wet Oxidation Process

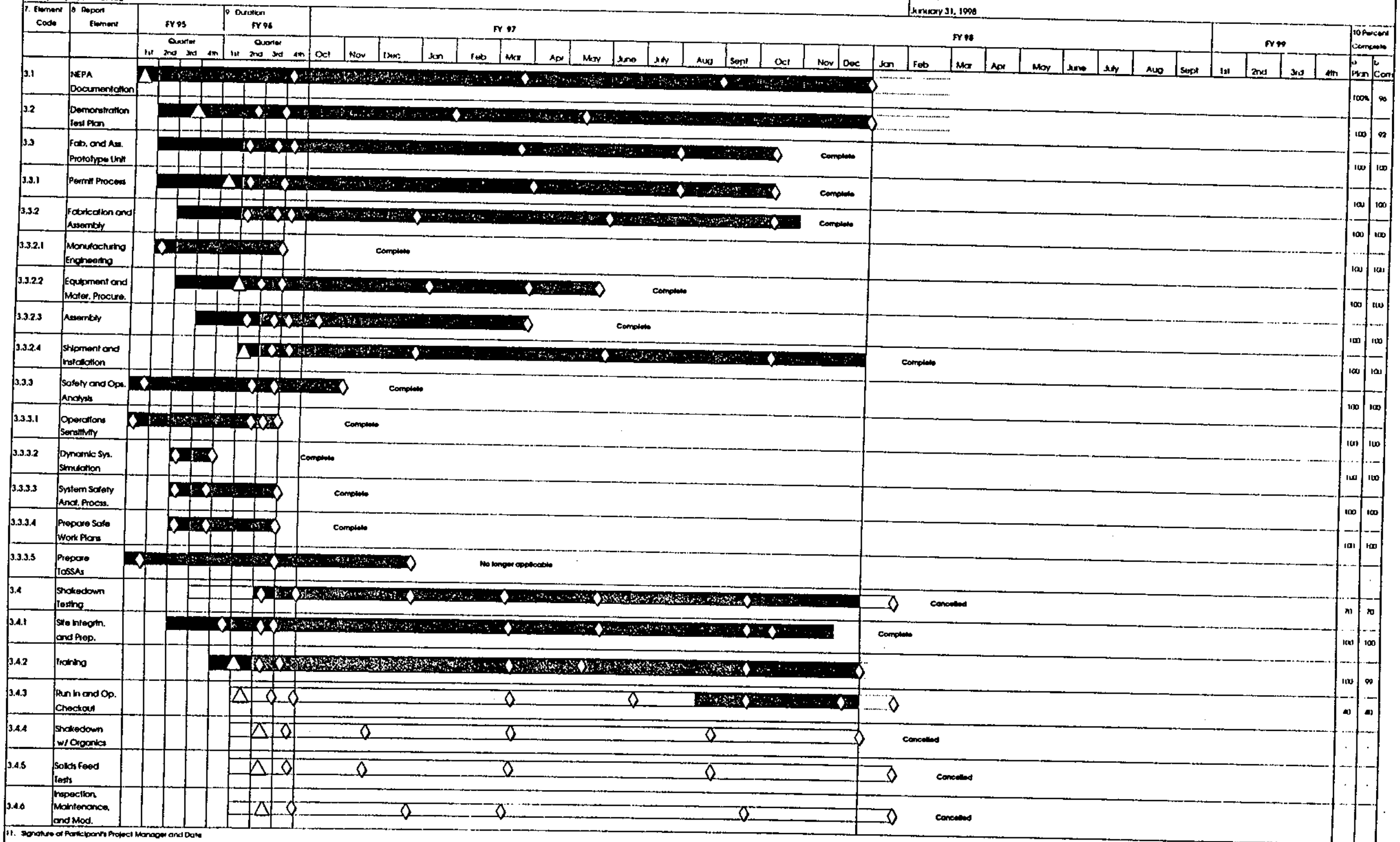
2. Reporting Period
December, 1997

3. Identification Number
DE-AC21-92MC29107

4. Participant Name and Address
Delphi Research, Inc.
701 Haines Avenue NW
Albuquerque, NM 87102

5. Start Date
March 13, 1995

6. Completion Date
January 31, 1998



11. Signature of Participant's Project Manager and Date

Date: January 12, 1998

Schedule bar dark shading reflects progress versus schedule as defined in April 7, 1995 Milestone Schedule Plan

Lighter color in bars shows time elapsed since original milestone dates given in April 7, 95 Milestone Schedule Plan