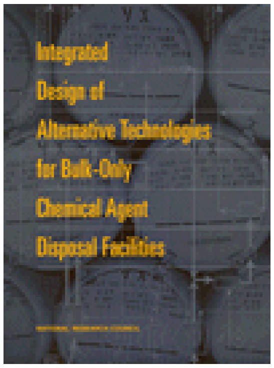

Integrated Design of Alternative Technologies for Bulk-Only Chemical Agent Disposal Facilities

Committee on Review and Evaluation of the Army Chemical Stockpile Disposal Program, Board on Army Science and Technology, National Research Council

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Committee on Review and Evaluation of the
Army Chemical Stockpile Disposal Program
Board on Army Science and Technology
Commission on Engineering and Technical Systems
National Research Council

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Preface

The United States has maintained a stockpile of chemical warfare agents and munitions for more than half a century. In 1985, Public Law 99-145 mandated an "expedited" effort to dispose of M55 rockets containing unitary chemical warfare agents because of their potential for self-ignition. This program soon expanded into the Army Chemical Stockpile Disposal Program (CSDP), whose mission is to eliminate the entire stockpile of unitary chemical weapons. The CSDP developed the baseline incineration system for that purpose. Since 1987, the National Research Council (NRC), through the Committee on Review and Evaluation of the Army Chemical Stockpile Disposal Program (Stockpile Committee), has provided technical and scientific advice and counsel to the Army's disposal program. In 1992, after setting several intermediate goals and dates, Congress enacted Public Law 102-484, which directed the Army to dispose of the entire stockpile of unitary chemical warfare agents and munitions by December 31, 2004. In 1994, the committee endorsed the baseline incineration system as an adequate technology for destroying the stockpile.

In the 1970s, the Army commissioned studies of different disposal technologies and tested several of them. In 1982, the Army selected incineration as the method it would use for the disposal of agents and associated propellants and explosives and the thermal decontamination of metal parts. In 1984, the NRC Committee on Demilitarizing Chemical Munitions and Agents reviewed a range of disposal technologies and endorsed the Army's selection of incineration. Since then, in response to public concerns about incineration and the evolution of other technologies, the NRC has also carried out several evaluations of alternative disposal technologies (*Alternative Technologies for the Destruction of Chemical Agents and Munitions* [1993], *Review and Evaluation of Alternative Chemical Disposal Technologies* [1996], and *Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons* [1999]). These reports recommended the development of chemical detoxification technologies for use at the two stockpile sites (Aberdeen, Maryland, and Newport, Indiana) where only bulk chemical agent (i.e., no energetically configured munitions) is stored.

The Army's selected nonincineration technologies for the Aberdeen, Maryland, and Newport, Indiana, stockpile sites both use chemical hydrolysis (reaction with alkaline water at elevated temperatures) to destroy chemical agent. The hydrolysis process is followed by either biodegradation (for treatment of mustard hydrolysis products at Aberdeen) or supercritical water oxidation (for destruction of VX hydrolysis products at Newport) to comply with requirements of the Chemical Weapons Convention and with U.S. environmental regulations. The development of these processes required extensive laboratory, bench-scale, and pilot-scale testing of individual processing steps to support engineering designs for the complete, integrated processes.

The Stockpile Committee has been evaluating the engineering process designs for integrating the individual processing steps for each site in parallel with the finalization of these designs by the Army and its contractors. In this report, the committee began with the design documents used by the Army as a basis for selecting final design contractors. Additional test data and design modifications suggested by the final design contractors were also reviewed as they became available through documentation and briefings. The committee's objective is not to provide a detailed, comprehensive design review of the processes, but to assess the integration of processing steps, analyze major design changes, and identify overarching issues that should be addressed to enhance safety and efficacy.

The committee greatly appreciates the support and assistance of NRC staff members, Donald L. Siebenaler, Harrison T. Pannella, William E. Campbell, Daniel E.J. Talmage Jr., and Carol R. Arenberg, in the production of this report.

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Charles E. Kolb, *vice chair*
Committee on Review and Evaluation
of the Army Chemical Stockpile
Disposal Program

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This report has been reviewed by individuals chosen for their diverse perspectives and technical expertise, in accordance with procedures approved by the National Research Council's Report Review Committee. The purpose of this independent review is to provide candid and critical comments that will assist the authors and the NRC in making the published report as sound as possible and to ensure that the report meets institutional standards for objectivity, evidence, and responsiveness to the study charge. The contents of the review comments and draft manuscript remain confidential to protect the integrity of the deliberative process. We wish to thank the following individuals for their participation in the review of this report:

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While the individuals listed above have provided many constructive comments and suggestions, responsibility for the final content of this report rests solely with the authoring committee and the NRC.

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Acronyms

ABCDF	Aberdeen Chemical Agent Disposal Facility
ACWA	Assembled Chemical Weapons Assessment
ADP	acquisition design package
CWC	Chemical Weapons Convention
EST	engineering-scale test
GC	gas chromatography
HD	distilled mustard
HE	hazard evaluation
HPLC	high-performance liquid chromatography
HRA	health risk assessment
HVAC	heating, ventilation, and air conditioning
ITMS	ion-trap mass spectrometry
MOC	material(s) of construction
MS	mass spectroscopy
MSD	mass selective detection
MSZ	magnesia-stabilized zirconia
NECDF	Newport Chemical Agent Disposal Facility
NMR	nuclear magnetic resonance
NRC	National Research Council
PMCD	Program Manager for Chemical Demilitarization
ppb	parts per billion
QRA	quantitative risk assessment
RCRA	Resource Conservation and Recovery Act
SBR	sequencing biological reactor
SCWO	supercritical water oxidation
SWEC	Stone and Webster Engineering Company
TDG	thiodiglycol
VOC	volatile organic compound
VX	a nerve agent
YSZ	yttria-stabilized zirconia

Executive Summary

The U.S. Army is pilot testing chemical hydrolysis as a method for destroying the chemical agents stockpiled at Aberdeen, Maryland (HD mustard agent), and Newport, Indiana (VX nerve agent). The chemical agents at both locations, which are stored only in bulk ton containers, will be hydrolyzed (using aqueous sodium hydroxide for VX and water for HD) at slightly below the boiling temperature of the solution. The resulting hydrolysate at Aberdeen, which will contain thiodiglycol as the primary reaction product, will be treated by activated sludge biodegradation in sequencing batch reactors to oxidize organic constituents prior to discharge to an on-site federally owned wastewater treatment facility. The hydrolysate at Newport, which will contain a thiol amine and methyl phosphonic acid as the major reaction products, is not readily amenable to treatment by biodegradation. Therefore, organic constituents will be treated using supercritical water oxidation (SCWO).

In keeping with the statement of task for this study, the Committee on Review and Evaluation of the Army Chemical Stockpile Disposal Program (Stockpile Committee) gathered and reviewed information on the preliminary process design for the Aberdeen Chemical Agent Disposal Facility (ABCDF) and Newport Chemical Agent Disposal Facility (NECDF). The report focuses on the overarching issues in the process designs integrating individual processing steps, including potential alternative configurations and process safety and reliability.

The committee reviewed the acquisition design packages (ADPs) for the ABCDF and NECDF prepared by Stone and Webster Engineering Company for the U.S. Army. An ADP is a 60-percent design (approximately) that is used as a basis for competitive bids for completion of the design, construction, commissioning, and operation of each chemical agent disposal facility. The committee also reviewed additional test data and design modifications suggested by the selected systems contractors as they became available. The committee did not attempt to provide a detailed, comprehensive review of the process designs. Rather, this report evaluates major design changes, assesses the integration of processing steps, and identifies overarching issues that should be addressed to enhance the safety and efficacy of the processes. The committee also identifies additional development and testing that may be necessary.

A review of the proposed process designs for the ABCDF and NECDF indicated that the reliability of the integrated SCWO process step at the NECDF is the only significant obstacle in terms of design and development. Although the ability of SCWO to destroy organic constituents in the hydrolysate from VX has been demonstrated, the adequacy of the materials of construction for the SCWO reactor and downstream components and their reliability have not been adequately demonstrated. An engineering-scale test to demonstrate the fully integrated SCWO processing step and address these issues is scheduled for the spring of 2000.

The committee believes that the off-site treatment of hydrolysates may have significant cost and schedule benefits for the Chemical Stockpile Disposal Program and could be a contingency plan if difficulties arise during start-up and pilot testing of the on-site hydrolysate (postneutralization) process steps. Off-site treatment of the hydrolysates would require that suitable treatment and disposal facilities be identified and that public acceptance be obtained. Identification and public acceptance of off-site treatment and disposal facilities for hydrolysates may be challenging. However, at the Tooele Chemical Agent Disposal Facility (Tooele, Utah), aqueous waste streams that do not contain agent are currently being disposed of off site. The committee believes that the Army should maintain both on-site and off-site treatment and disposal options where possible. The development and verification of alternatives for the management of nonprocess wastes will also be important to avoid the on-site accumulation of these materials and delays during facility closure.

The committee also discusses: evaluations that should be completed; improvements in monitoring that would maximize worker safety; improvements in process efficiency through design changes; and more rapid process monitoring.

RECOMMENDATIONS

Recommendation 2-1. The alternative process for the removal, recovery, and disposal of volatile organic compounds (VOCs) recommended by Bechtel, the contractor for the Aberdeen Chemical Agent Disposal Facility (ABCDF), using air stripping followed by vapor-phase activated carbon absorption, is an improvement over the VOC removal process described in the acquisition design package and should be incorporated into the final design of the ABCDF. The activated carbon filter system should be designed to sustain high loading and enable rapid change-out of VOC-laden filter material.

Recommendation 2-2. The Army should continue to identify and pursue regulatory approval for waste management facilities to receive off-site shipments of brine from the supercritical water oxidation processing of VX hydrolysate at the Newport Chemical Agent Disposal Facility. However, given the modest cost of the equipment and the significant costs and processing delays that could result if shipping or receiving of the effluent were not allowed or became prohibitively expensive, the capability of processing the brine on site should be maintained.

Recommendation 2-3. The Army should conduct a comparative evaluation of the designs for enclosures to store and process chemical agent at the Aberdeen and Newport facilities to determine if similar enclosures could be used for storing and processing drained agent.

Recommendation 2-4. The Army should conduct a detailed analysis of the cascade ventilation system and its performance at both the Aberdeen and Newport facilities to determine the potential for agent migration during normal operations, maintenance, and upset conditions. A formal, structured evaluation should be performed that includes (1) the use of a dynamic model of the ventilation system to determine the migration of both lethal and sublethal agent concentrations and (2) reported instances of agent migration at the Tooele and Johnston Atoll baseline incineration facilities.

Recommendation 2-5. The design teams for the Aberdeen and Newport facilities, in coordination with the Army, should develop a formal means of sharing design information and developing best practices. Personnel at each facility should be designated and held accountable for facilitating the exchange of design information and lessons learned.

Recommendation 2-6. The end points for plant closure of the Aberdeen and Newport facilities should be identified (e.g., allowable residual contamination thresholds and possible future uses of the facilities). Plans for plant design, construction, and operation should include plans for facility closure, and reviews of plant designs should include preliminary plans for closure. Public involvement in determining this aspect of plant design should be encouraged.

Recommendation 3-1a. The Army should develop criteria and a schedule for resolving design and operational issues raised in the 1998 report, *Using Supercritical Water Oxidation to Treat Hydrolysate from VX Neutralization,* that have not yet been resolved for supercritical water oxidation operation at Newport. These issues include materials of construction, fabrication methods, system plugging, pressure let-down, and the duration of successful continuous pilot-scale operations.

Recommendation 3-1b. The Army should pursue the testing of materials of construction for treating VX hydrolysate by supercritical water oxidation (SCWO) more aggressively to finalize materials selection, design, and fabrication methods for critical components, including the SCWO reactor, inlet, and pressure let-down system. This testing should clearly define mechanisms and rates of corrosion and erosion under the range of anticipated process conditions. An independent panel of experts in materials of construction should evaluate testing to date and identify further needs to ensure that the reliability of the SCWO system is adequate to meet the processing objectives.

Recommendation 3-2. For worker protection and secondary containment, the final design package for the Newport facility must include the physical hazard controls (e.g., protective barricades) common to industrial operations involving high pressure and stored energy. Systems must be designed to minimize leaks, plugging, and ruptures of the supercritical water oxidation reactor and associated plumbing and protective barriers. Secondary containment equipment will also be necessary, including safety systems for handling high-purity oxygen at high pressure, such as protection against downstream fires and explosions caused by contact between combustible materials (e.g., activated carbon) and oxygen-enriched gas streams, under normal and upset conditions.

Recommendation 4-1. The Army should evaluate off-site management of hydrolysates both for potential cost and schedule benefits and as a contingency plan in case difficulties arise during start-up and pilot testing of the on-site (postneutralization) process steps.

Recommendation 4-2. The Army should define the quantity and characteristics of anticipated nonprocess waste streams and evaluate options for the final disposition of each type of waste. On-site processes should be developed and demonstrated for treating all agent-contaminated wastes. Methods of representative sampling and characterization of wastes should be developed to verify that treated wastes are free of agent to below appropriate risk thresholds.

Recommendation 5-1. Plant ventilation air at each facility should be monitored to verify the absence of agent in processing areas downstream of the toxic cubicles. Gaseous exhausts to the atmosphere should also be monitored to verify the absence of agent. Agent monitoring at each facility should incorporate the lessons learned from agent monitoring at the other disposal facilities.

Recommendation 5-2. After the transfer of each batch of hydrolysate from the reactors to storage tanks, the hydrolysate should be sampled from the hydrolysate storage tank and analyzed before subsequent processing to verify that it is free of detectable agent.

Recommendation 5-3. The criteria for releasing VX hydrolysate to subsequent processing steps at the Newport facility should be based on the 99.9999 percent agent destruction level required by the state of Indiana and an evaluation of worker and public risks associated with releases of agent in hydrolysate equal to or less than this destruction level (i.e., 330 ppb). Assessments of worker risk should include a thorough evaluation of risk mitigation measures and additional design and operating requirements necessary to satisfy this release criterion.

Recommendation 5-4. The Army should develop and demonstrate methods of chemical analysis to confirm the destruction of VX in the hydrolysate at the Newport facility. These methods should include procedural specifications and provisions for training so that confirmation at the required detection limits can be confirmed by testing by different analysts at multiple laboratories.

Recommendation 5-5. The Army should develop and demonstrate innovative analytical techniques that require significantly less time than present techniques to verify agent destruction for both VX and HD hydrolysates at the required detection limits.

Recommendation 6-1. Quantitative risk assessments and hazard evaluations for both the Newport and Aberdeen facilities should be carried out as early as possible during facility design, and the findings from these assessments should be used in both the design and future operation of the facilities to minimize risk and maximize safety.

Recommendation 6-2. Schedule risk should be evaluated for the Aberdeen and Newport facilities in the context of program implications and public risks and appropriate contingency plans developed if needed.

1

Introduction

Congress has directed the U.S. Army to dispose of its stockpile of chemical warfare agents in accordance with the Chemical Weapons Convention (CWC). At five of nine stockpile sites, the Army is using, or intends to use, high-temperature incineration as part of a baseline system to destroy the chemical agents in stored weapons. However, for two sites where chemical agents are stored solely in bulk containers, the Army has been pursuing alternative agent-destruction technologies that are more acceptable to the public than incineration. Several alternative technologies were evaluated intensively by the National Research Council (NRC, 1996a) and by the Army for use at these sites. Consistent with NRC recommendations in these evaluations, the Army chose to demonstrate one technology, chemical hydrolysis[1] of the toxic agents, on a pilot scale (U.S. Army, 1996). The products of chemical hydrolysis (hydrolysate) will require further treatment to satisfy the requirements of the CWC and U.S. environmental regulations.

The chemical agents stored at the two bulk-only sites differ substantially (HD mustard agent at Aberdeen, Maryland, and VX nerve agent at Newport, Indiana). Therefore, different treatment process sequences were selected for use at the two sites. In both processes, the chemical bonds associated with agent toxicity will be destroyed by hydrolysis (with water for HD or aqueous sodium hydroxide for VX) (NRC, 1996a). However, in order to achieve rapid and complete hydrolysis, different reaction conditions will be used for each agent. Following hydrolysis, the processing steps for treating the hydrolysate for disposal will also differ greatly. At both sites, the stockpiled agents also contain significant concentrations of impurities, which had to be taken into account in the selection of postneutralization process steps (these impurities were examined in *Review and Evaluation of Alternative Chemical Disposal Technologies* [NRC, 1996a]). At the Aberdeen site, the HD stockpile also contains significant concentrations of volatile organic compounds (VOCs), chlorinated aliphatic hydrocarbons (which are common industrial solvents) that must be treated in the overall process.

The stockpile at Aberdeen Proving Ground, Maryland, consists of 1,625 tons of HD (mustard agent) stored in ton containers. HD, a blistering agent that produces painful, slow-healing burns on the skin, is toxic upon inhalation. The Army plans to detoxify HD by treatment with water slightly below the boiling point to produce an acidic aqueous solution of thiodiglycol (TDG). Although this solution is relatively nontoxic, TDG is regulated by the CWC because it could potentially be reconverted to mustard agent. Both to comply with the CWC and to ensure safe, environmentally responsible disposal, the HD hydrolysate will be made slightly alkaline and subjected to biological oxidation. Microorganisms will then be used to oxidize the TDG to carbon dioxide and an aqueous sulfate solution that can be safely discharged to a federally owned waste treatment facility on site. The resulting filter cake will be shipped to a commercial facility for disposal.

The Newport, Indiana, stockpile contains 1,269 tons of VX nerve agent, a relatively nonvolatile, persistent toxin that disrupts signal transmission in the central nervous system. VX can be hydrolyzed in hot aqueous sodium hydroxide solution. This process produces a strongly alkaline solution containing salts of methylphosphonic acid, its monoethyl ester, and a foul-smelling thiol amine compound. In 1996, the NRC recommended further evaluation of the Army's

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[1] The Army refers to the destruction of chemical agent via hydrolysis as chemical *neutralization*. The term is derived from the military definition of *neutralize*, to render something unusable or nonfunctional. Hydrolysis is a reaction of a target compound with water, often catalyzed by an acid or a base, in which a chemical bond is broken in the target, and the components of water, OH^- and H^+, are inserted at the site of the bond cleavage. The technical definition of neutralization is a chemical reaction between an acid and a base to form a salt and water. Chemical agents are neither acids nor bases, however, so the use of the term neutralization is somewhat confusing. Nevertheless, in the literature on chemical demilitarization in aqueous systems, the terms *neutralization* and *hydrolysis* are used interchangeably.

plans to use biological oxidation to destroy the organic components of this solution (NRC, 1996a). To date, attempts to treat VX hydrolysate by biological oxidation have not been successful. Therefore, the treatment of the hydrolysate by supercritical water oxidation (SCWO) is under development. Laboratory and bench-scale tests of the SCWO treatment have demonstrated nearly complete destruction of the organic compounds in VX hydrolysate. The combination of hydrolysis followed by SCWO would meet the regulatory requirements for the safe destruction and disposal of VX. However, more development and testing will be necessary to ensure the reliable operation of the SCWO processing step (NRC, 1998).

The Stockpile Committee's statement of task for this study was as follows:

> The NRC study will accomplish the following:
>
> - gather and review information on the 60-percent process design for the chemical agent demilitarization facilities to be constructed in Aberdeen, Maryland, and Newport, Indiana. Information to be evaluated will include the 60% process design documentation, testing, and evaluation data developed in support of the design development, relevant information contained in environmental permit applications, previous related NRC reports on chemical agent demilitarization, and briefings from the Army and its contractors.
> - focus the evaluation on overarching issues in the process design which result from integration of the individual process steps, process safety, and process reliability. This evaluation will not include verification of specific design calculations or regulatory compliance.
> - identify process design components which the Stockpile Committee believes warrant additional evaluation or action, including potential alternative configurations, options, or supporting information, prior to the Army finalizing the design for each facility.
> - present the evaluation in a report which identifies each design issue, articulates the basis for the recommended further evaluation or action, and potential alternative approaches (where possible).

In keeping with this statement of task, the committee gathered and reviewed information on the 60-percent process design[2] for the chemical agent disposal facilities to be constructed at Aberdeen and Newport. The committee focused on the overarching issues resulting from the integration of individual processing steps, including process safety, reliability, and potential alternative configurations.

An acquisition design package (ADP) is an approximately 60-percent completed design that is used as a basis for competitive bids for completion of the design, construction, and operation of each chemical agent destruction facility. The committee has reviewed the ADP designs for the Aberdeen and Newport facilities prepared by Stone and Webster Engineering Company (SWEC) (U.S. Army, 1998a, 1998b). As additional test data and design modifications suggested by the systems contractors became available, the committee reviewed them as well. No attempt was made to provide a detailed, comprehensive review of the process designs. Rather, this report reviews the major design changes, assesses the integration of processing steps, and identifies overarching issues that should be addressed. The committee also notes future development and tests that may be necessary. Chapter 2 presents the committee's evaluations, findings, and recommendations for the overall ADP designs for the disposal technologies at the Aberdeen and Newport chemical stockpile sites. Chapter 3 addresses issues related to the use of SCWO to treat VX hydrolysate at the Newport site. Chapter 4 focuses on the management of process and nonprocess wastes; Chapter 5 examines agent and nonagent monitoring; and Chapter 6 focuses on risk management.

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[2] The 60-percent design specifies the detailed design basis, primary process steps, process flow diagrams, major equipment components, monitoring requirements, facility requirements (e.g., building layouts, electrical and ventilation requirements), and preliminary operational strategies. Detailed final specifications of all process components, facilities, and operations are not complete.

2

Acquisition Design Package Processes and Modifications

Initial designs for the ADPs of the Aberdeen and Newport facilities were developed by SWEC under supervision by the U.S. Army Program Manager for Chemical Demilitarization (PMCD) (U.S. Army 1998a, 1998b). The selected contractors, Bechtel National, Inc., for the Aberdeen Chemical Agent Disposal Facility (ABCDF), and Parsons Infrastructure and Technology Group for the Newport Chemical Agent Disposal Facility (NECDF), will subsequently procure, install, and operate the facilities. After reviewing the ADPs for their respective sites, both contractors suggested modifications to the processes. The modifications were discussed with SWEC and the PMCD, who will authorize accepted variations.

ABERDEEN CHEMICAL AGENT DISPOSAL FACILITY

The basic component processes in the ADP for the ABCDF (shown in Figure 2-1), which were described in *Review and Evaluation of Alternative Chemical Disposal Technologies* (NRC, 1996a), are listed below:

- draining of HD from ton containers and removal of the residual agent by a series of hot water and steam sprays
- neutralization of the HD by hydrolysis using water in a stirred tank at a temperature just below the boiling point, followed by the addition of aqueous sodium hydroxide to neutralize acid formed during hydrolysis
- removal of VOCs from the resulting hydrolysate by vaporization in a falling film evaporator
- decomposition of condensed VOCs in an aqueous hydrogen peroxide and sodium hydroxide solution exposed to ultraviolet radiation
- removal of organic compounds remaining in the hydrolysate (after VOC removal) with a sequencing biological reactor (SBR), using an activated sludge process with added nutrients

The treated hydrolysate and sludge from the SBR flow to an on-site, federally owned treatment works for further biological and physical treatment prior to discharge. The sludge consists of excess biomass produced during the treatment of hydrolysate in the SBR. The excess biomass, which is separated, dewatered, and shipped off site for commercial disposal, is expected to be similar to sludges generated by traditional wastewater treatment processes, which are routinely disposed of in commercial facilities.

Each step of the ABCDF process is decoupled from the other process steps by storage of the product of 24 hours of operation to allow operation with either the preceding or subsequent processing step off line. The draining and decontamination of ton containers and the agent hydrolysis steps are batch processes, and reactors are sized to accommodate the agent from one ton container per batch. The VOC removal system is a continuous operation with significant turn-down capacity to accommodate changes in the rate of hydrolysate production; the VOC removal system is buffered by both upstream and downstream tankage. The biological reactors are also operated in a batch or semibatch mode. Vent gases from tankage that could potentially contain agent are treated by activated carbon before being discharged into the cascade ventilation system of the facility, which is modeled after the ventilation systems at the baseline incineration sites at Tooele, Utah, and Johnson Atoll. In this system, air flows from its highest pressure in nonagent areas to potentially agent-contaminated areas at lower pressure, then to toxic cubicles maintained at still lower pressure prior to being discharged through a series of activated carbon filters. The areas are separated by air locks. Air from areas that will not be exposed to agent (e.g., biological reactors) is exhausted through separate activated carbon filters. The facility is designed to treat six ton containers per day, or 10,000 pounds of agent per day. The plant is scheduled to be operated for nine months during pilot operations and 11 months during full operations.

Bechtel has evaluated various alternative processes for the removal, recovery, and destruction of VOCs at the

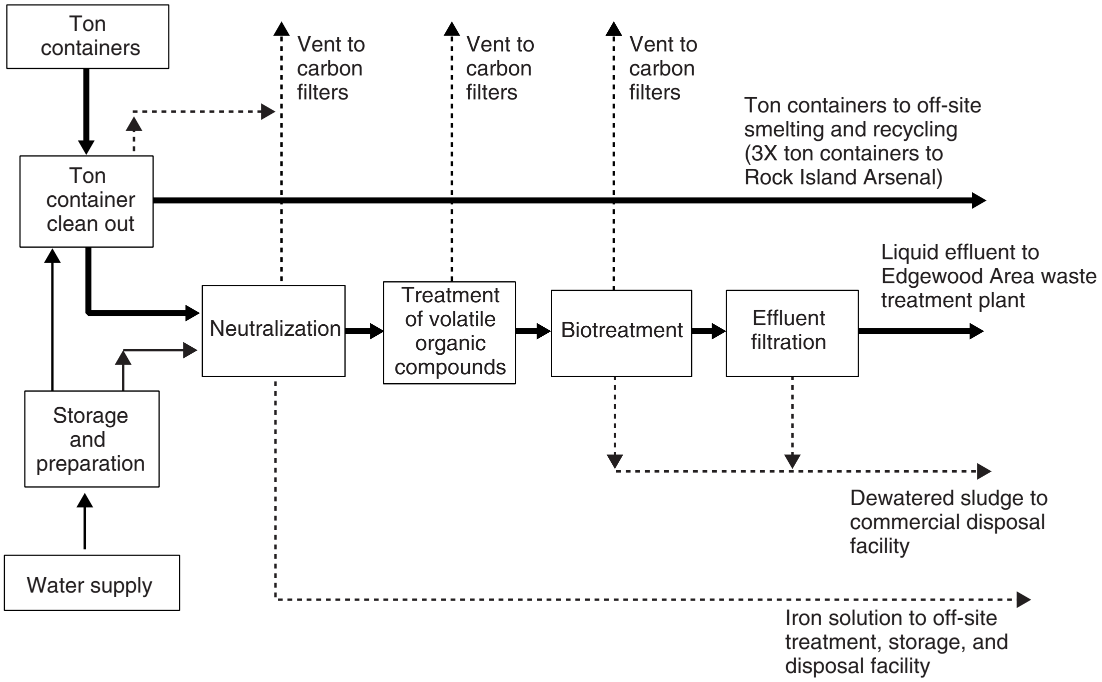

FIGURE 2-1 Basic component processes in the ADP for the ABCDF. Source: Ward, 1999.

ABCDF, including either steam or air stripping of the VOCs from the hydrolysate, followed by VOC recovery on an activated carbon sorbent. In the case of steam stripping, the VOCs are recovered from the liquid phase after condensation of the steam. In the case of air stripping, they are recovered from the gas phase. In both cases, the spent activated carbon is returned to the supplier of the carbon, and the recovered VOCs are destroyed in the process of regenerating the sorbent.

Following a comparison of the complexity, safety, and cost of these treatment processes, including a modification of the process in the ADP, Bechtel recommended that air stripping of VOCs from the hydrolysate be used (Bechtel, 1999). Bechtel's preliminary, revised design includes two packed stripper columns in parallel, followed by three carbon filters in series. The carbon filters recover both VOCs stripped from the hydrolysate and organic compounds in vapors released from the hydrolysate storage tanks. (An overall process description and material throughputs for the ABCDF can be found in Appendix A.)

The modification recommended by Bechtel eliminates the need for steam to heat an evaporator or for stripping; eliminates the need for a condenser and storage tank to provide liquid for the ultraviolet oxidation process; and eliminates the need for hydrogen peroxide to augment ultraviolet oxidation of VOCs. With the revised process, the plant will be safer, more reliable, and less costly. However, the revision does involve a significant change in the overall process flow and creates an additional process stream (i.e., the air flow from the carbon filters). Therefore, the change would require modifications of various permits, which could delay the construction and operation of the plant, although the overall schedule for completing disposal operations is not likely to be affected.

Air stripping has been used in various applications to remove VOCs from water. For example, VOCs similar to those from the ABCDF process streams have been removed from contaminated groundwater by air stripping. At the ABCDF, the stripper will have to remove approximately 99 percent of the VOCs to meet the requirements of the Resource Conservation and Recovery Act (RCRA) for discharge of the aqueous stream. Furthermore, the high entry temperature of the hydrolysate into the air stripper, and the consequent temperature change in the hydrolysate as it flows through the stripper and as the water is vaporized, are design considerations that will have to be addressed.

The quantity of activated carbon required to remove stripped VOCs from the air stream has been estimated to be 600 lb/day (Bechtel, 1999). Frequent change-outs of the activated carbon will be necessary. The design and instrumentation of the filter system should have the following capabilities:

- handling high loadings of VOCs
- indicating when fresh filter material is required
- facilitating the addition of fresh, and the removal of spent, filter material with minimum down time to plant operations

A critical consideration is identifying an off-site waste processor who is able and willing to receive the carbon filter material from the VOC treatment step. A waste processor will certainly require that VOCs be stripped from the hydrolysate only after agent destruction has been confirmed by chemical analysis. Consequently, the spent activated carbon used to recover VOCs should be agent free and can be treated like any other commercially produced spent filter material. If the spent activated carbon cannot be certified as agent free, it will have to be managed in the same manner as spent activated carbon that is known or likely to be agent contaminated.

Recommendation 2-1. The alternative process for the removal, recovery, and disposal of volatile organic compounds (VOCs) recommended by Bechtel, the contractor for Aberdeen Chemical Agent Disposal Facility (ABCDF), using air stripping followed by vapor-phase activated carbon absorption, is an improvement over the VOC removal process described in the acquisition design package and should be incorporated into the final design of the ABCDF. The activated carbon filter system should be designed to sustain high loading and enable rapid change-out of VOC-laden filter material.

NEWPORT CHEMICAL AGENT DISPOSAL FACILITY

The basic component processes in the ADP for the NECDF (shown in Figure 2-2), which were described in *Review and Evaluation of Alternative Chemical Disposal Technologies* (NRC, 1996a), are listed below:

- neutralization of VX agent by hydrolysis using an aqueous solution of sodium hydroxide in a stirred tank, at a temperature just below the boiling point
- removal of organic compounds from the resulting hydrolysate by oxidation with pure oxygen by SCWO
- evaporation of the aqueous stream from the SCWO reactor with a forced-circulation evaporator/crystallizer, followed by condensation of the vapor, crystallization of the inorganic salts from the concentrated liquid, and recovery of the solids

Prior to discharge, the condensate flows to the on-site federally owned treatment works, which includes an activated-sludge sewage treatment plant. The crystallized salts are shipped to an off-site commercial treatment, storage, and disposal facility.

Each step of the NECDF process is decoupled from the other process steps by storage of the product of 24 hours of operation to allow operation with either the preceding or subsequent processing step off line. The draining and decontamination of ton containers and the agent hydrolysis steps are batch processes, and reactors are sized to accommodate the agent from one ton container per batch. The SCWO

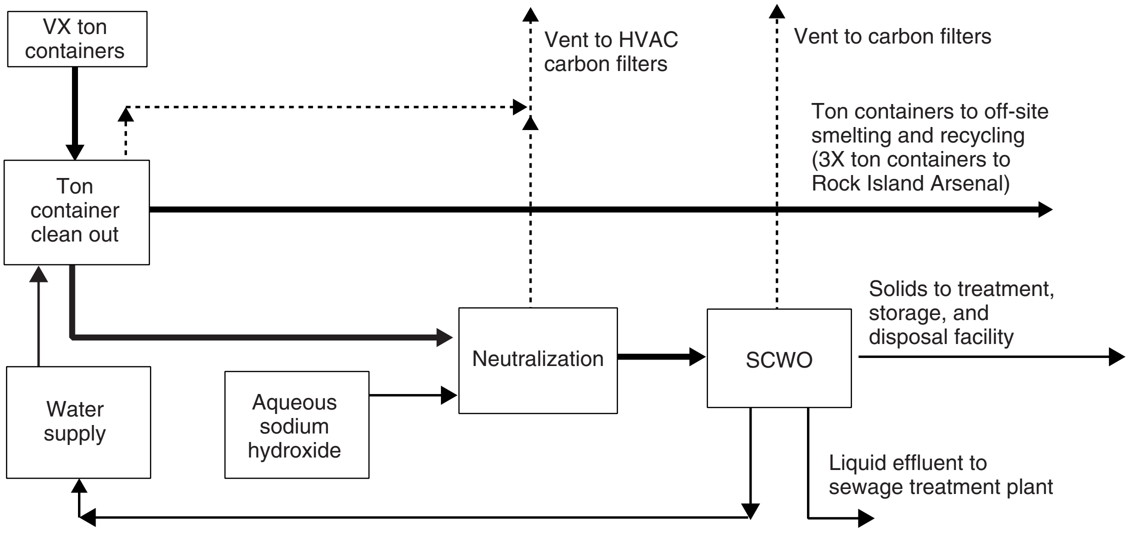

FIGURE 2-2 Basic component processes in the ADP for the NECDF. Source: Ward, 1999.

reactor process step includes two processing lines that can be operated either individually or in parallel as continuous processes, with scheduled off-line periods for rinsing the reactor. Tankage is provided both upstream and downstream of the SCWO reactor. Vent gases from tankage that could potentially contain agent are treated by activated carbon before being discharged into the cascade ventilation system of the facility, which is modeled after the ventilation systems at the baseline incineration sites at Tooele, Utah, and Johnston Atoll. In this system, air flows from its highest pressure in nonagent areas to potentially agent-contaminated areas at lower pressure, then to toxic cubicles maintained at still lower pressure prior to being discharged through a series of activated carbon filters. The areas are separated by air locks. Air from areas that will not be exposed to agent (e.g., SCWO effluent gases) is exhausted through separate activated carbon filters. The facility is designed to treat six ton containers per day, or 9,000 pounds of agent per day. The plant is scheduled to be operated for seven months during pilot operations and eight months during full operations.

The contractor, Parsons, is now considering the following alternatives for dealing with the effluent aqueous stream from the SCWO reactor:

- alternative evaporator/crystallizer designs (e.g., drum drier, spray drier, thin-film evaporator)
- off-site shipment and disposal of the unconcentrated effluent, eliminating the evaporation, crystallization, and crystal drying processing steps
- off-site shipment and disposal of a concentrated brine from the evaporator (containing primarily sodium sulfate and sodium phosphate), eliminating the crystallization and crystal drying processing steps

In the meantime, Parsons is seeking public and regulatory approval for the off-site shipment of the dilute effluent from the SCWO reactor and continuing design development of the evaporator/crystallizer included in the ADP. A number of similar evaporator/crystallizer installations have been cited as examples of such equipment (NRC, 1998). Parsons recommends eliminating the evaporator/crystallizer in the NECDF design if shipment of dilute SCWO effluent from the site is approved, which would significantly simplify overall process operations. (A process description and material throughputs for the NECDF is included in Appendix B.)

Recommendation 2-2. The Army should continue to identify and pursue regulatory approval for waste management facilities to receive off-site shipments of brine from the supercritical water oxidation processing of VX hydrolysate at the Newport Chemical Agent Disposal Facility. However, given the modest cost of the equipment and the significant costs and processing delays that could result if shipping or receiving of the effluent were not allowed or became prohibitively expensive, the capability of processing the brine on site should be maintained.

AGENT HOLDING AND PROCESSING

The Aberdeen and Newport ADPs include essentially the same arrangements for handling ton containers (i.e., agent draining, clean out, and preparation for off-site disposal). All processing of ton containers to remove agent is carried out in toxic cubicles designed to contain agent vapors, maintained under negative pressure, and ventilated through a series of activated carbon filters. The design of the toxic cubicles is essentially the same as the design of the cubicles at the Johnston Atoll and Tooele facilities. Operations in the cubicles are carried out by automated machinery operated remotely. The ton container is initially punched in two locations, and the agent is removed through a drain tube. Next, the ton container is cut in half perpendicular to the primary axis. Each half is then subjected to high-pressure hot water and steam sprays to remove residual agent. After treatment, the container halves are transferred through air locks to a holding chamber with atmospheric monitoring to verify that no detectable agent remains. If agent is detected, the container half is returned for further hot water and steam treatment until agent is no longer detected. Personnel are not present in the toxic cubicles during operations. During maintenance and decontamination operations, they must wear protective ensembles specific to the type of agent.

Holding and Processing

The chemical agent holding and processing arrangements for the ABCDF includes three identical processing lines, each of which includes the following components:

- a chemical agent holding tank and associated surge tank
- two agent neutralization reactors
- a holding tank for effluent from the clean-out of ton containers
- a holding tank for effluent from the decontamination of ton containers
- a scrubber system, including a condenser and two carbon filters in series, for the neutralization of vent gas

This equipment is housed in one cubicle for each processing line. Drained agent is stored in the same room as the neutralization reactors and other processing equipment.

The plan for holding and processing chemical agent at the NECDF calls for a chemical agent holding tank and associated surge tank, from which agent is fed into two identical neutralization processing lines, each of which includes the following components:

- a reactor for treating drained agent
- a reactor for treating effluent from the clean-out of ton containers
- a holding tank for effluent from the decontamination of ton containers

The agent holding and surge tanks are located in an enclosed toxic cubicle. Each neutralization processing line is located in a separate enclosed cubicle. No vent gas scrubber or carbon filter is located in either of the neutralization cubicles to treat vent gases.

The rationale for differences between the agent holding and processing arrangements at the Aberdeen and Newport facilities is not immediately clear. The ABCDF approach, with its multiple agent holding tanks, increases plant complexity. In addition, because the chemical agent holding and surge tanks are located in the neutralization processing cubicle, rather than in a separate enclosure, a larger area than necessary could be contaminated from an accidental spill. The use of multiple agent holding tanks also differs from currently operating baseline facilities at Tooele, Utah, and Johnston Atoll, both of which have a single agent storage tank in a separate enclosure.

Recommendation 2-3. The Army should conduct a comparative evaluation of the designs for enclosures to store and process chemical agent at the Aberdeen and Newport facilities to determine if similar enclosures could be used for storing and processing drained agent.

COMMINGLING OF PROCESS VENT GASES AND PLANT VENTILATION AIR

At both the ABCDF and NECDF, the plant ventilation air flows through a sequence of enclosed areas. First, air is drawn from the ambient air into areas where agent contamination is least likely. Then, controlled by vents, the air cascades through a sequence of plant areas where contamination is increasingly likely. Upon exiting these areas, the ventilation air passes through an activated carbon filter to remove any traces of agent and VOCs. Drawn by exhaust fans, it then flows back to the ambient air. Thus, the air pressure in the plant is lower than the ambient air pressure and is lowest where agent contamination is most likely. Under normal operating conditions, the airflow and pressure levels in the plant will prevent the migration of agent vapors from contaminated to uncontaminated areas. However, upsets can occur in the plant heating, ventilation, and air conditioning (HVAC) system as a result of the misalignment of doors and dampers, obstructions in ducts, malfunctioning air locks (used to pass objects between areas of the plant), and failures of exhaust fans. Moreover, the ABCDF and NECDF ventilation systems contain parallel flow paths, which are particularly susceptible to upsets.

Process vent gases from agent holding and surge tanks, neutralization reactors, and holding tanks for effluent from the clean-out of ton containers are all likely to contain agent vapors and VOCs, both of which can be removed either by first passing them through a carbon filter or a scrubber. The scrubbed gas can then be directed into the air ventilation duct passing into the ton container cleanout area or exhausted to the HVAC air filtration system. If process vent gases without pretreatment (as currently designed) are piped into the cascade ventilation system, hazardous process gases could potentially flow back into a nontoxic area of the plant as a result of operational error. Therefore, the direct discharge of process vent gases into ventilation air in the cubicle, even after cleaning, should be avoided.

At both the ABCDF and NECDF, the vent gases from the agent holding and surge tanks pass through an activated carbon filter. The discharge is directed to the sump of the cubicle, to prevent liquid agent from being forced from the vents of these tanks through the filter. However, based on the relative sizes of the primary agent holding tanks and the larger agent surge (overflow) tanks, the rationale for this design is unclear. If only vapors flow through the tank vents and the carbon filter, the discharged gases could be directed into the duct of the ventilation air leaving the toxic cubicle. The systems controlling the flow and level of liquid in the agent holding and surge tanks, as well as the vents, should be designed to minimize the possibility of liquid flowing into and through the carbon filter.

At the ABCDF, vent gases from the neutralization reactors and the ton container effluent tanks pass through a scrubber, two carbon filters in series, and a fan, prior to entering the HVAC ducts that exit the three toxic/neutralization cubicles. These exhaust HVAC ducts lead to the final banks of activated carbon filters and to the induced draft fans.

Vent gases from the hydrolysate storage tanks and the SBRs at the ABCDF are vented to a condenser, a knockout drum, two activated carbon filters in series, and an induced draft fan, prior to being discharged to the atmosphere through the plant stack. At the NECDF, the vents of the four hydrolysate storage tanks are directed through a reflux condenser to a common vent cubicle, then to the activated carbon filters and the induced draft fans of the HVAC system, and finally to the plant stack.

However, vent gases from the neutralization reactor for drained agent and the reactor for the effluent from the clean-out of ton containers in the two parallel neutralization cubicles at the NECDF are vented through a condenser to a single process ventilation cubicle. The vent gases then go to the final set of activated carbon filters and the induced draft fans of the HVAC system and then to the plant stack. The committee is concerned that interior plant air could be contaminated at the NECDF because this design includes no carbon filtration or other detoxification of the process tank vent gases before they flow directly into the HVAC exhaust system of the plant, which includes carbon filtration. At the Tooele and Johnston Atoll baseline incineration facilities, process vent gases (incinerator exhaust gases) are not piped into the plant cascade ventilation system at all. Instead, they are handled and treated entirely separately from the processing building's cascade ventilation system.

For the purposes of a quantitative risk assessment (QRA) and hazards evaluation (HE), a thorough analysis will be required to determine the potential upset conditions for the ventilation systems, as well as to prevent agent migration into uncontaminated areas from changes in ventilation patterns during normal or maintenance operations. The model currently used for QRAs focuses only on concentrations of lethal agent. Dynamic models of the airflows and pressures for the plant ventilation systems at the ABCDF and NECDF could be used to determine agent levels at various locations in the plants during maintenance activities, in the event of power or interlock failures, and for other upset conditions. Comparisons of predicted properties for parallel systems versus single systems would reveal the safest, lowest risk alternative.

Recommendation 2-4. The Army should conduct a detailed analysis of the cascade ventilation system and its performance at both the Aberdeen and Newport facilities to determine the potential for agent migration during normal operations, maintenance, and upset conditions. A formal, structured evaluation should be performed that includes (1) the use of a dynamic model of the ventilation system to determine the migration of both lethal and sublethal agent concentrations and (2) reported instances of agent migration at the Tooele and Johnston Atoll baseline incineration facilities.

USE OF COMMON DESIGNS

In the preceding section, the committee suggested that a common design for the agent holding and processing systems at the ABCDF and NECDF could be used, as well as common designs and equipment selections for other systems. Candidate systems for common design include ton container clean out, activated carbon filtration of ventilation air and process off-gases, and management of nonprocess waste streams (e.g., agent-contaminated activated carbon; disposal of demilitarization protective ensemble suits, hydraulic fluids, hoses, and cleaning materials). The committee believes that an investigation of using common designs and equipment could encourage the contractors completing the ADP designs for the ABCDF and NECDF to share their knowledge and experience of procurement, installation, operational, and maintenance processes. In addition, if common designs and equipment were used, lessons learned at either installation would be pertinent to the other, and spare parts might be shared.

Recommendation 2-5. The design teams for the Aberdeen and Newport facilities, in coordination with the Army, should develop a formal means of sharing design information and developing best practices. Personnel at each facility should be designated and held accountable for facilitating the exchange of design information and lessons learned.

PLANNING CLOSURE OF THE FACILITIES

In addition to the completion of the ADP designs and the construction, operation, and maintenance of the Aberdeen and Newport facilities, the contractors have accepted the responsibility for plant closure, which will require dealing with stored wastes, plant equipment, buildings, and the land on which the facilities are located. The plant closure end point involves defining the condition of the land and whatever building structures and equipment will remain on the site. Closure can be facilitated if the requirements for closure are carefully considered as part of the completed designs. The Army's closure team for the Johnston Atoll facility is now studying the process of closure and has published a draft report, *Guide to Closure Planning: Draft, Revision 0* (U.S. Army, 1999a). Many of the lessons learned from the closure of the Johnston Atoll facility should be transferable to the ABCDF and NECDF.

Elected officials, professional planners, the site workforce, local businesses, and the surrounding public all have important stakes in the closure and future uses of the site. Some of the issues the PMCD will have to address with stakeholders are the economic impact of closure, mitigating negative economic impacts, and analyzing options for future land use. Public involvement will be critical to the resolution of these issues, and local governments and residents are likely to have strong feelings and considerable expertise about economic issues and future use.

Recommendation 2-6. The end points for plant closure of the Aberdeen and Newport facilities should be identified (e.g., allowable residual contamination thresholds and possible future uses of the facilities). Plans for plant design, construction, and operation should include plans for facility closure, and reviews of plant designs should include preliminary plans for closure. Public involvement in determining this aspect of plant design should be encouraged.

3

Supercritical Water Oxidation Process for the Treatment of VX Hydrolysate

MATERIALS OF CONSTRUCTION AND OTHER ISSUES

The subject of SCWO for treating VX hydrolysate was discussed extensively in *Using Supercritical Water Oxidation to Treat Hydrolysate from VX Neutralization* (the *SCWO* report) (NRC, 1998). The treatment of VX hydrolysate is significantly different and more complex than previous treatments using SCWO. Because of the multitude of outstanding challenges with this process, the committee recommended that a pilot-scale test (i.e., an engineering-scale test, [EST]) be done to define operating characteristics and to demonstrate sustained continuous operation. A number of critical issues identified in the recommendations of the *SCWO* report have not been sufficiently addressed in the ADP (60-percent design package).

Although progress has been made on two of the most critical issues, the selection of materials of construction (MOC) and demonstration of salts/solids handling in and downstream of the SCWO reactor, but has not yet been demonstrated during the EST. A number of key issues, including demonstration of a robust pressure let-down system, worker safety (see below), and regulatory, analytical, and disposal issues associated with salts and other process effluents, have yet to be addressed. A contract has been awarded to General Atomics, Inc., for an EST to be carried out with VX hydrolysate in 2000. The results will have to be carefully evaluated to ensure that lessons learned are incorporated into the design and operation of the full-scale SCWO process. The Army should consider design contingencies or alternative hydrolysate management options in case adequate process throughput cannot be achieved with the current design.

The selection of appropriate MOCs is a key requirement for the successful treatment of VX hydrolysate by SCWO (see Finding 2 and Recommendation 2 of the *SCWO* report [NRC, 1998]). SCWO treatment of VX hydrolysate involves highly corrosive and erosive environments that change drastically as the hydrolysate passes through the reactor and its peripheral components (inlet, outlet, and pressure let-down valves). As the oxidation of VX hydrolysate proceeds, the reaction mixture undergoes dramatic changes in pH (from strongly basic initially to slightly acidic as acids are formed), temperature, salt content, and dielectric constant. Based on available data, the committee believes that the hydrolysate becomes a changing, multiphase mixture consisting of dense fluids, salt solutions, and precipitated solids.

The treatment of VX hydrolysate puts very stringent requirements on the MOCs, which must be extremely durable and reliable. Furthermore, the use of unusual MOCs may create challenges for reactor fabrication. Consequently, the importance of tests for specifying MOCs to be used in the full-scale system cannot be overstressed. To date, no demonstrated material has provided reliable operation over a sufficient period of time; however, recent results indicate that platinum and platinum alloys may be viable materials. The Army recently provided the Stockpile Committee with preliminary draft evaluations of the results of two tests: static coupon tests in small laboratory-scale autoclaves (batch testing) with hydrolysate surrogate[1] (the SWEC test) (Ballinger, 1999; Jensen et al., 1999); and General Atomics continuous coupon testing with VX hydrolysate and hydrolysate surrogate (the General Atomics MOC test; three campaigns from July 31, 1999, to October 4, 1999, totaling 500 hours) (Jensen et al., 1999).

SWEC Test Results

Screening tests were performed in a small laboratory-scale Alloy 625 autoclave (at 3,500 psi) charged with a small volume of liquid hydrolysate surrogate, a headspace of gas (oxygen or an inert gas), and coupons supported on yttria-stabilized zirconia (YSZ) buttons (for galvanic isolation from

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[1] Hydrolysate surrogate is comprised of 50.5 wt percent deionized water, 15.3 wt percent sodium salt of isethionic acid, 13.2 wt percent dimethyl methylphosphonate, 9.8 wt percent diethanolamine, 9.0 wt percent sodium hydroxide, and 2.2 wt percent isopropyl alcohol (Landry, 1999).

the autoclave). Three separate conditions were tested to simulate reaction zones in a continuous SCWO reactor:

- Zone 1: heat-up/mixing zone—surrogate, 690°F, inert gas (argon)
- Zone 2: reaction zone—surrogate, 1,200°F, oxygen
- Zone 3: cool-down/quench zone—end product surrogate, CO_2, O_2, 700°F

In addition to the Alloy 625 of the autoclave itself, the materials or material junctures examined were: nickel (Ni) 201, platinum, platinum to Hastelloy C-276 weld in Zone 3, the YSZ supports, and arc-plasma sprayed YSZ on zirconium (thermal cycling only). The nickel, YSZ supports, and YSZ coating on zirconium exhibited poor performance (high corrosion, fracture, and delamination, respectively) in Zone 1 and Zone 2. Platinum was deemed to be satisfactory in Zones 1 and 3 but exhibited corrosive attack, including pitting and the formation of a platinum-sulfur compound, at the grain boundaries in Zone 2. The platinum/Hastelloy C-276 weld was determined to be satisfactory in Zone 3.

The platinum coupons in experiments for Zone 2 resulted in only a 0.05 percent weight gain after 160 hours of exposure. However, photomicrographs indicated two forms of attack: (1) formation of a reaction product on the grain boundaries; and (2) significant pitting of the surface. Annealed samples underwent the same forms of attack (a total of three samples were studied). The reported formation of a platinum-sulfur compound from oxidized sulfur (from the sulfate product of the sulfonic acid moiety in the hydrolysate surrogate) in an oxidizing environment is somewhat surprising, although the report claims it was consistent with thermodynamic considerations (General Atomics, 1999a).

Although these preliminary results are discouraging, the chemical and physical environments in the system used for these batch tests (i.e., headspace in the batch reactor, different phases, and variable temperatures) may be very different from those in a continuous SCWO reactor. Additional fundamental work and continuous reactor testing appear to be necessary. For example, oxygen depletion resulting from the corrosion/oxidation of the wall of the autoclave could account for the presence of reduced sulfur species, including hydrogen sulfide. The Army and the contractor will need a more thorough understanding of the processes in the autoclave and of the reactivity and thermodynamics of sulfur species and platinum. Furthermore, because the presence of platinum-sulfur compounds implies platinum oxidation, with the consequent potential for solubilization, the effluent from the reactor should be analyzed for platinum.

General Atomics Test Results

The preliminary results of recent MOC tests conducted by General Atomics under continuous conditions in a SCWO reactor are more encouraging (General Atomics, 1999b). This series of three sequential MOC coupon tests used a hydrolysate surrogate and VX hydrolysate provided by the Assembled Chemical Weapons Assessment (ACWA) program. The VX hydrolysate used in the tests was higher in NaOH concentration and had higher trace amounts of chlorine and fluorine than the hydrolysate that will be produced from the NECDF stockpile. The goal of the three tests was to recommend MOCs for the SCWO reactor. Coupons made from a range of materials were exposed to representative SCWO conditions to determine corrosion rates and mechanisms. Because the amount of available VX hydrolysate was insufficient to support a test with a duration of 500 hours, one test with VX hydrolysate was run for 167 hours, and two tests with hydrolysate surrogate were run for 167 hours to expose the coupons to a total of 500 hours of operation.

The MOC tests were carried out in a 4-in diameter by 6-ft long reactor that had been used during ACWA demonstration tests (General Atomics, 1999b). High-pressure oxygen was used as the oxidant. Coupons of various materials were attached to a coupon tree placed along the reactor length. In this way, coupons could be placed in different regions of the reactor along its length, including a quench zone at the end of the reactor. The coupons were subsequently examined under a microscope to determine microstructural effects and thicknesses as a function of location in the SCWO reactor. The following materials were tested:

- noble metals (platinum, Pt-20%Ir, Pt-10%Rh), Au-8%Pd, Rh)
- platinum-plated metals, ceramics (magnesium-stabilized zirconia [MSZ], YSZ)
- nickel-steel alloys (C276, I617, I625, I690, HA25, HA188)
- refractory tantalum and niobium (for quench zone and downstream components only)

The preliminary results of the tests are shown in Table 3-1.

The results described in the General Atomics report can be summarized as follows (Jensen et al., 1999):

- platinum and Pt-20%Ir and Rh showed the least corrosion with no evidence of intergranular corrosion, suggesting them as prime candidates for MOCs
- Pt-10%Rh and Au-8%Pd exhibited intergranular attack as a corrosion mechanism
- platinum-plated coupons exhibited delamination in all but the quench zone, indicating that higher quality platinum plating or other liner materials will be necessary in zones where the coating components have complex shapes
- Ni-based steels, including Ni/Co alloys, showed much greater corrosion than the Pt alloys in zones other than the quench zone
- YSZ and MSZ ceramic coupons displayed coating

TABLE 3-1 Preliminary Results of Corrosion Testing with Noble Metals and Superalloys

Material	Inlet Heat-up Section	High-Temperature Reaction Section	Quench Section
NOBLE METALS Maximum Corrosion (mils/year)			
Pt	55	47	8
Pt-20%Ir	68[a]	21	5
Pt–10%Rh[b]	68	81	5
Au–8%Pd[b]	> 500	> 500	16
Rh	39	[c]	[c]
SUPERALLOYS Maximum Corrosion (mils/year)			
C276 (Ni-steel)	[c]	2,360	26
I617 (Ni-steel)	[c]	1,940	[c]
I625 (Ni-steel)	[c]	2,070	26
I690 (Ni-steel)	2,100	2,180	131
HA25 (Ni-steel)	2,500	2,200	26
HA188 (Ni-steel)	[c]	1,550	[c]
Tantalum	[c]	[c]	52
Niobium	[c]	[c]	1,940

Notes:
[a] Nozzle deterioration caused direct impingement that may have caused higher corrosion
[b] Displayed intergranular corrosion
[c] Not tested

Source: Adapted from Jensen et al., 1999.

spallation and corrosion in the reaction zones

- all metals and alloys except niobium exhibited little corrosion in the quench zone

Corrosion rates of the platinum and Pt-based alloys ranged from 50–65 mils/year, based on micrographs revealing uniform recession (i.e., no intragranular attack) (Jensen et al., 1999). On the basis of these preliminary estimates and the absence of undesirable corrosion mechanisms, such as intergranular attack, General Atomics has recommended a platinum liner (20 mils thick) for the EST and a Pt-20%Ir alloy liner (12 mils thick) as a backup (Burchett, 1999; Jensen et al., 1999). MOC testing and performance evaluation will have to be an intrinsic part of the EST program, both for the reactor and for downstream components exposed to aggressive environments (e.g., pressure and temperature reduction components). The selection and reliability of all primary components will have to established by a thorough demonstration of process performance.

Current plans for the NECDF call for installing two full-scale SCWO reactors (both sized for 100 percent of the planned throughput of VX hydrolysate) and one uninstalled reactor in reserve. All three reactors will have platinum liners 20 mil thick (Jensen et al., 1999). However, a thickness of 20 mil is substantially thinner than the required liner thickness based on test results for 100-percent throughput of VX hydrolysate for a single reactor, with no conservative design margin. This change in thickness would significantly reduce the capacity margin provided for in the original design. Because of the high level of uncertainty associated with the performance of MOCs, a highly conservative design margin, either by an increase in liner thickness or through reserve reactor liners, will be essential.

Another factor to be considered is the time required to fabricate additional liners if the initial supply is depleted. Recently, General Atomics claimed it was able to fabricate 20-mil thick liners of the required diameter for the reactor. General Atomics plans to "float" a precious-metal liner in a cylindrical Hastelloy pressure vessel and use cooled elastomeric O-rings that have performed satisfactorily on other SCWO systems to form the SCWO reactor. The annular space between the liner and the vessel wall will be monitored for leaks to indicate when change-out of the liner is required. No decision has been made yet on whether to use platinum or Pt-20%Ir. Because they have markedly different mechanical properties, these two liner materials may require significantly different fabrication methods. Platinum is relatively weak and very ductile; Pt-20%Ir is less ductile but 10 times stronger. Final selection of the liner material for use at the NECDF was scheduled for early 2000. Fabrication times for the liner and reactor assembly have been reported to be at least two to three months. Production of hydrolysate for the EST was begun in 1999 to ensure that sufficient VX hydrolysate would be available for the EST planned for the spring of 2000.

In summary, parallel with plans for further MOC tests, the Army has elected to move forward with a design that will incorporate a liner in the SCWO reactor system that may or may not be used in a sacrificial capacity. Batch testing suggests that significant corrosion occurs even with precious-metal liners. The preliminary analyses of the General Atomics tests are useful for identifying materials that might be viable but are not sufficient for making a final selection, which will require reconciling the significant disparities between the General Atomics and SWEC test results. In addition, the drastic differences in the performance of platinum materials during batch and continuous tests should be explained and verified. Although oxygen-depleted zones may have been present in the batch tests, they might also be present in a full-scale SCWO system processing VX hydrolysate because of poor mixing. Therefore, this issue must be resolved. The EST should provide further information on corrosion kinetics and mechanisms, as well as a demonstration of the viability of liner fabrication and the reliability of MOCs. In short, the Army must have a more fundamental and practical understanding of MOCs.

Using a liner, or an entire reactor, in a sacrificial capacity may become a viable alternative, but this decision must be based on an understanding of reactor lifetime and the development of maintenance, monitoring, and change-out/replacement procedures and schedules. Considerable testing, including pilot-scale tests, will be necessary to address these issues.

Corrosion of the metal components in the reactor will cause these metals to appear in the effluent from the SCWO reactor and, ultimately, in the salt stream from the evaporator. The ADP design does not describe how these metals, some of which are regulated (e.g. chromium, nickel), could affect the disposal of the salt streams, which consist largely of sulfate and phosphate salts. The Army will have to determine the amounts and effects of metals in the SCWO effluent as a result of corrosion, including their effects downstream of the SCWO reactor and how their presence will affect handling and disposal, overall effluent quality, monitoring, and regulatory and permitting issues.

Operational Issues

Problems with salt/solids management, including the potential for plugging and difficulties in pressure let-down, was thoroughly described in the *SCWO* report (NRC, 1998) but have not been resolved in the ADP as currently modified. Further development work and testing will be necessary to address this important issue. The tests carried out by General Atomics to demonstrate high destruction efficiencies of VX hydrolysate indicated potentially significant operational problems with the handling of solids in the SCWO reactor and with the robustness of the pressure let-down system (NRC, 1998).

General Atomics has since carried out two additional tests with a modified pressure let-down system to improve system reliability (General Atomics, 1999b; Jensen et al., 1999). The new design calls for gas/liquid separation after quench at high pressure and prior to pressure let-down. Test results have shown satisfactory continuous operation for up to 40 hours without plugging or corrosion/erosion of the pressure let-down system (General Atomics, 1999b). However, the system did not operate without interruption for extended periods during demonstration tests for the ACWA program. The interruptions were attributed to plugging by entrained solids in the aqueous reactor effluent from corrosion of the liner materials that did not redissolve during temperature and pressure let-down. A platinum liner was not available for the test because of schedule constraints.

Recommendation 3-1a. The Army should develop criteria and a schedule for resolving design and operational issues raised in the 1998 report, *Using Supercritical Water Oxidation to Treat Hydrolysate from VX Neutralization,* that have not yet been resolved for supercritical water oxidation operation at Newport. These issues include materials of construction, fabrication methods, system plugging, pressure let-down, and the duration of successful continuous pilot-scale operations.

Recommendation 3-1b. The Army should pursue the testing of materials of construction for treating VX hydrolysate by supercritical water oxidation (SCWO) more aggressively to finalize materials selection, design, and fabrication methods for critical components, including the SCWO reactor, inlet, and pressure let-down system. This testing should clearly define mechanisms and rates of corrosion and erosion under the range of anticipated process conditions. An independent panel of experts in materials of construction should evaluate testing to date and identify further needs to ensure that the reliability of the SCWO system is adequate to meet the processing objectives.

SAFETY ISSUES

Several safety issues for processing VX hydrolysate by SCWO must still be resolved. First, the SCWO reactor operates at 1,200°F and 4,000 psig pressure. Protective measures should be put in place to protect personnel in case of a rupture of the reactor. The system should be anchored and barricaded to protect workers and the environment. Second, current plans call for high-purity oxygen to be used as the oxidant being supplied to the reactor at high pressure, which will require appropriate safeguards. The evaluation of safeguards for using pure oxygen should include compatibility with MOCs, both during normal operating conditions and potential failure modes (e.g., exposure of SCWO reactor components at high temperature and pressure if leakage of the primary liner occurs).

The current ADP calls for carbon filtration of the off-gases from the SCWO reactor. The purpose of the carbon beds is not clear to the Stockpile Committee. SCWO off-gas enriched in oxygen relative to air (as a result of excess oxygen input to the reactor) would create a potential fire hazard. If carbon filtration is required to remove trace organic species from the SCWO off-gas prior to emission, added safeguards may be required to prevent carbon fires that could result from reaction of the carbon with unreacted oxygen in the SCWO off-gas. If the carbon beds are intended to remove traces of agent, they appear to be superfluous because no agent should be present in the off-gas after hydrolysis and the subsequent reaction of the hydrolysate in the SCWO reactor. In the committee's opinion, treatment by carbon filtration to guard against the emission of chemical agent could increase complexity and risk without providing any benefit. Trace constituents in SCWO effluent gases can be quantified during the EST.

Recommendation 3-2. For worker protection and secondary containment, the final design package for the Newport facility must include the physical hazard controls (e.g., protective barricades) common to industrial operations involving high pressure and stored energy. Systems must be designed to minimize leaks, plugging, and ruptures of the supercritical water oxidation reactor and associated plumbing and protective barriers. Secondary containment equipment will also be necessary, including safety systems for handling high-purity oxygen at high pressure, such as protection against downstream fires and explosions caused by contact between combustible materials (e.g., activated carbon) and oxygen-enriched gas streams under normal and potential upset conditions.

4

Management of Process and Nonprocess Wastes

At both the ABCDF and NECDF, several waste streams will be generated from the treatment processes. Although they differ appreciably because of the differences in the processes, they also have many features in common. An underlying principle in the plant designs for managing these waste streams is that they be treated on site only to the point that they can be safely shipped to outside organizations for final treatment and disposition. On-site final treatment is avoided if possible. Although this approach makes economic sense, it also means that management systems to ensure safe and efficient handling will be necessary. To that end, the Army should take the following steps:

- define criteria for off-site shipment
- validate technologies and measurement techniques for meeting these criteria
- define and install backup on-site systems where warranted
- obtain necessary permits
- obtain public acceptance
- institute operating practices and systems for maintaining safe and efficient handling of wastes, on and off site

At both the ABCDF and NECDF, agent destruction is accomplished during the neutralization process step, the product of which is hydrolysate. Although each hydrolysate contains reaction products that require monitored destruction under the CWC, their characteristics are similar to those of common industrial hazardous wastes. The committee believes that off-site treatment of hydrolysate may offer significant cost and schedule benefits to the CSDP and could at least be considered a contingency plan in case difficulties arise during start-up and pilot testing of the on-site hydrolysate (postneutralization) process steps. Off-site treatment of the hydrolysates will require that suitable treatment and disposal facilities be identified and that public acceptance of this approach be obtained. Identification and public acceptance of off-site treatment and disposal facilities for hydrolysates may be challenging. However, at the Tooele, Utah, facility, aqueous waste streams that do not contain agent are currently being disposed of off site. Thus, the committee believes that it is prudent to maintain both on-site and off-site treatment and disposal options where possible.

Besides process effluent streams, the operation and eventual closure of both the ABCDF and NECDF will produce agent-contaminated wastes. Some examples are listed below:

- demilitarization protective ensemble suits and breathing air hoses used by workers doing maintenance in plant areas subject to agent contamination
- activated carbon and other filter materials used to control emissions
- waste materials (e.g., particles, sludges, and fluids) removed from process lines, filters, and strainers during maintenance operations
- plant equipment operations (e.g., small parts, liners, etc.) removed and replaced during maintenance
- maintenance equipment and supplies (e.g., pails, tools, absorbent spill pillows, wipes)
- primary plant equipment and agent-contaminated surface coatings or layers removed during closure of the facility

A comprehensive list of nonprocess wastes that will require treatment and disposal management can be found in the documentation for closure of the Johnston Atoll facility (U.S. Army, 1999b).

The Army must make provisions for storing, decontaminating, shipping, and disposing of nonprocess waste materials, including the plant itself, which must be treated as waste. In the baseline incineration system, contaminated wastes can be disposed of in the various incinerators. But comprehensive plans will have to be developed for the disposal of all contaminated waste streams at the Aberdeen and Newport facilities. For example, waste might be decontaminated with the neutralizing medium used to hydrolyze agent and the resulting stream treated in the plant. At the ABCDF, the subsequent treatment would involve removal of VOCs followed by treatment in the SBR; the SCWO system would be used at the NECDF. However, the compatibility of the waste stream with the disposal equipment and operation would have to be investigated. Several waste streams at baseline incineration system facilities—especially plastic hoses, parts, and containers—have been especially difficult to decontaminate.

Operations at both the ABCDF and NECDF will result in a waste stream of used activated carbon from areas that are known to be or could potentially be contaminated with agent. Used activated carbon from process steps and areas where agent is not present (e.g., VOC removal from hydrolysate at the ABCDF) will also have to be disposed of. This carbon is intended to be regenerated and reused. Process monitoring, record keeping, and segregation will be necessary to differentiate the carbon that is or has potentially been exposed to agent from that which has not. Carbon exposed (or potentially exposed) to agent is not being considered for regeneration, whereas unexposed carbon is expected to be regenerated. Activated carbon that has been used to treat exhaust from areas known to be contaminated or areas that could potentially be contaminated with agent will have to be shown to be free of agent to below required levels for shipment off site. Sampling and analytical methods will be necessary to meet this challenge.

For off-site shipment of decontaminated wastes, provisions will have to be made for qualifying the wastes for shipment, as well as arrangements made for receiving and disposing of them. Attempts to ship agent decontamination brines (that were tested to be free of agent) from the Johnston Atoll facility met with extreme public resistance and were eventually disallowed. Off-site shipment of nonprocess wastes from the ABCDF and NECDF may present an even more complex problem because of the diversity of physical waste forms, a known history of agent contact for some wastes, and the Army's inability to demonstrate definitively that the waste stream is truly free of agent. If the traditional headspace analysis used to establish 3X conditions is not sufficient to release materials from Army control,[1] nonincineration methods for the complete decontamination of nonprocess wastes and analytical methods for verifying that materials are free of agent will be necessary. Testing of MOCs and performance evaluations will have to be an intrinsic part of the EST program, both for the SCWO reactor and for downstream components exposed to aggressive environments (e.g., pressure and temperature reduction components). The selection and reliability of all primary SCWO components will have to be thoroughly demonstrated by process performance.

Recommendation 4-1. The Army should evaluate off-site management of hydrolysates both for potential cost and schedule benefits and as a contingency plan in case difficulties arise during start-up and pilot testing of the on-site (postneutralization) process steps.

Recommendation 4-2. The Army should define the quantity and characteristics of anticipated nonprocess waste streams and evaluate options for the final disposition of each type of waste. On-site processes should be developed and demonstrated for treating all agent-contaminated wastes. Methods of representative sampling and characterization of wastes should be developed to verify that treated wastes are free of agent to below appropriate risk thresholds.

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[1] The 3X decontamination level refers to solids decontaminated to the point that the agent concentration in the headspace above the encapsulated solid does not exceed the health-based, eight-hour, time-weighted average limit for worker exposure. The levels for HD and VX are 3.0 and 0.01 μg per cubic meter of air, respectively. Materials classified as 3X may be handled by qualified plant workers using appropriate procedures but are not releasable to the environment or for general public reuse. In specific cases in which approval has been granted, a 3X material may be shipped to an approved hazardous waste treatment facility for disposal in a landfill or for further treatment.

5

Agent and Nonagent Monitoring and Measurements

MONITORING AGENT IN THE VAPOR PHASE

Monitoring for the presence of agent is a critical step in ensuring worker and public safety, which is the number one priority of the PMCD. Monitoring for agent in the vapor phase is common to baseline incineration system facilities and alternative technology bulk-site facilities. The committee anticipates that the Army's considerable experience in monitoring techniques for both mustard and VX will be directly transferable to the ABCDF and NECDF. The committee has already recommended several potential improvements for vapor-phase agent monitoring that are applicable to the ABCDF and the NECDF (NRC, 1994a, 1996b).

Insofar as the committee can ascertain, the ADPs for the ABCDF and NECDF include adequate agent monitoring of the air in areas where elevated agent concentrations are likely, such as agent storage and neutralization process areas. However, provisions for monitoring for agent in areas downstream of the toxic cubicles may not be adequate. These areas include hydrolysate storage and downstream processing areas (VOC treatment and biochemical treatment at the ABCDF; air stripping and SCWO at the NECDF). The committee considers it prudent for the Army to monitor ambient air and gaseous exhausts to the atmosphere for agent in processing areas downstream of the toxic cubicles where process upset conditions (e.g., operator error) could potentially result in agent contamination.

Recommendation 5-1. Plant ventilation air at each facility should be monitored to verify the absence of agent in processing areas downstream of the toxic cubicles. Gaseous exhausts to the atmosphere should also be monitored to verify the absence of agent. Agent monitoring at each facility should incorporate the lessons learned from agent monitoring at the other disposal facilities.

MONITORING AGENT IN CONDENSED PHASES

Monitoring for the presence of agent in process streams is necessary to verify agent destruction after neutralization and to verify the absence of agent prior to final release or disposal. Monitoring process streams and nonprocess waste streams for the presence of agent is also required to ensure worker safety and compliance with environmental regulations. The following main processing streams will require monitoring:

- vapor streams from scrubbing operations or activated carbon treatment (see discussion above on vapor-phase monitoring)
- hydrolysate from neutralization of agent
- ton container clean-out solutions
- aqueous effluents from the secondary treatment processes (biological treatment for HD hydrolysate; SCWO for VX hydrolysate) prior to discharge or disposal (e.g., after crystallization and separation of salts from SCWO effluent)
- solid salts or sludges separated from aqueous streams
- ton containers and metal parts following clean-out operations

Analysis for agent contamination of solid phases will also be required for nonprocess wastes, including spent activated carbon, process sludges, dunnage, and personal protective equipment.

Monitoring Solid Materials

Monitoring for residual agent in solid materials is required for both baseline and bulk-site disposal facilities. Establishing that ton containers, metal parts, and other solid materials (e.g., personal protective equipment) have been decontaminated to a 3X level requires confining the material in an enclosed space and analyzing the vapor for agent concentrations that do not exceed the eight-hour time-weighted average limit for worker exposure. A 3X decontamination level must be demonstrated prior to the shipment of materials for off-site treatment or disposal. In many cases, establishing a 3X decontamination level is an intermediate step prior to thermal treatment to achieve a 5X condition (either by on-site treatment at baseline facilities or by off-site treatment for 3X decontaminated ton containers at bulk-site facilities).[1] Thermal treatment to a 5X standard will not be an option for on-site treatment of solid wastes at the ABCDF and NECDF.

Measuring potential agent contamination in certain solid process streams (e.g., sludges and dried salts) and in spent activated carbon presents challenges unique to the alternative technology sites. For baseline sites, these materials can be thermally treated in one of the incinerators (e.g., the metal parts furnace) to achieve the 5X decontamination level prior to disposal of the ashes. Because this is not an option for the alternative technology bulk sites, measurement techniques must be developed and demonstrated to certify that these materials have no detectable agent prior to off-site shipment for disposal.

Vapor-phase monitoring to meet the 3X specification will most likely not be sufficient to verify agent destruction in the solid materials from the bulk sites because these materials could contain either strongly adsorbed agent or occluded agent that could be released in the future. Because of the unique analytical interferences resulting from the composition of particular waste streams, the measurement methods will have to be specific to each waste stream, and each method will have to be validated for the specific waste matrix. Criteria for determining the detection limit for each method should be based on the hazard and risk evaluations for that waste stream.

Monitoring Liquids

Measuring agent at low levels in aqueous phases, such as hydrolysate, decontamination fluids, and final waste streams, is particularly challenging because of the presence of organic and inorganic constituents that interfere with measurement techniques. Measuring for residual agent in the hydrolysate from the neutralization step may present the greatest challenge. Nevertheless, measurement is necessary for the following purposes:

- to verify the efficacy of the primary agent destruction step (e.g., 99.9999 percent destruction)
- to delist the hydrolysate so that it can be managed as a nonhazardous waste in subsequent processing steps (biological treatment for HD hydrolysate, SCWO for VX hydrolysate)
- to clear any hydrolysate to be shipped off site for process development and testing purposes

From operational experience obtained during the development of the neutralization process and pilot operations conducted at the Chemical Agent Munitions Disposal System (near Tooele, Utah), the Army is aware of the potential presence of residual agent in poorly mixed or occluded zones of the hydrolysis reactor. This agent may not be detected during analysis of a batch of hydrolysate following neutralization but may be detected after the hydrolysate has been transferred to downstream storage vessels. Operator error could also result in the transfer of hydrolysate that still contains residual agent to downstream processing. Reactor designs for the ABCDF and NECDF have been carefully developed to avoid poorly mixed or occluded reaction zones, and interlocks are provided to prevent operator errors. However, the committee considers it prudent for the Army to analyze the contents of hydrolysate storage tanks following the transfer of each batch of hydrolysate to the tanks.

Recommendation 5-2. After the transfer of each batch of hydrolysate from the reactors to hydrolysate storage tanks, the hydrolysate should be sampled from the hydrolysate storage tank and analyzed before subsequent processing to verify that it is free of detectable agent.

The hydrolysate testing requirements are distinctly different from the requirements for the verification of agent destruction for final release of process effluents because the hydrolysate is an "in-process" stream that requires further treatment (in accordance with both the CWC and environmental regulations). The Army's initial specifications for testing hydrolysate, which were based on standards for battlefield drinking water, were unrealistic for three reasons: (1) hydrolysate will not be discharged without further treatment; (2) human consumption is highly unlikely; and (3) the complexity of the analytical techniques makes measurements unusually time consuming and unreliable. More appropriate criteria for detection limits would be based on the required destruction efficiency and a risk-based evaluation of subsequent hydrolysate management. Analysis of the hydrolysate should also include measurements of potentially active species, such as sulfonium ions from HD hydrolysis and EA2192 from VX hydrolysis.

Criteria for detection limits to verify the absence of agent in the aqueous waste streams after final on-site treatment should be based on a risk evaluation that assumes either discharge to public waters or another final disposal method. Risk evaluations must address both worker and public risks. Evaluations of public risks from the accidental release of hydrolysate at less than 330 ppb (99.9999 percent destruction level) of agent have shown that public risk is minimal (Garrick and Gekler, 1999). However, an evaluation of worker risk and potential mitigation measures has not been completed.

Recommendation 5-3. The criteria for releasing VX hydrolysate to subsequent processing steps at the Newport

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[1] Solids are treated to a 5X decontamination level by holding them at 1,000°F for 15 minutes, which results in complete decontamination. The material can then be released for general use or sold in accordance with applicable federal, state, and local regulations.

facility should be based on the 99.9999 percent agent destruction level required by the state of Indiana and an evaluation of worker and public risks associated with releases of agent in hydrolysate equal to or less than this destruction level (i.e., 330 ppb). Assessments of worker risk should include a thorough evaluation of risk mitigation measures and additional design and operating requirements necessary to satisfy this release criterion.

Measurement of VX at Low Concentrations

Measuring very low levels of VX in hydrolysate has been extremely difficult because of the presence of reaction products from the neutralization of VX, organic impurities, stabilizers present in the stockpile grade VX that remain after neutralization, and the high concentrations of excess sodium hydroxide necessary for neutralization. The hydrolysate from the neutralization process thus remains a highly reactive matrix. Representative sampling of the hydrolysate is also difficult because it consists of two phases (aqueous and organic) that must be maintained in a thoroughly mixed condition during sampling, and the organic phase can wet the sample container walls. Thus, the Army has had difficulty reliably meeting the initial target of 20 ppb (the battlefield drinking water standard). The required VX destruction efficiency for the neutralization process (> 99.9999 percent) necessitates a detection limit of less than 330 ppb. By contrast, a detection limit of less than 20 ppb has been demonstrated for VX in effluents from SCWO treatment of hydrolysate. Measurement of VX in SCWO effluents is much simpler because concentrations of organic constituents are low and the pH is near neutral.

The following approaches have been developed for measuring VX in hydrolysate (NIVA Consultants et al., 1999):

- nuclear magnetic resonance (NMR) carried out directly on hydrolysate
- chloroform extraction followed by gas chromatography-ion trap mass spectroscopy (GC-ITMS)
- methylene chloride extraction followed by high-performance liquid chromatography-mass spectroscopy-mass spectroscopy (HPLC-MS-MS)
- solid-phase extraction followed by gas chromatography-mass selective detection (GC-MSD)
- hexane/acid liquid-liquid extraction followed by GC-ITMS-MS

Multiple variations of these approaches have been investigated. NMR requires approximately 16 hours to achieve a detection limit of 500–1,000 ppb. Because of the long analytical time and high detection limit, this method is considered unsuitable. The chloroform extraction method has been found to achieve a detection limit between 80 and 200 ppb and requires approximately four hours to complete. Methylene chloride extraction followed by HPLC-MS-MS has been found to achieve a detection limit of approximately 100 ppb and requires less than one hour to complete. The solid-phase extraction technique has been found to achieve a detection limit of between 4 and 35 ppb and requires several hours. However, analytical recoveries from this method have been erratic, ranging from less than 50 percent to greater than 350 percent. Evaluation of this approach suggests the possible formation of VX under the analysis conditions from the original agent impurities.

Recent results obtained during analysis of the VX hydrolysate produced at the Chemical Agent Munitions Disposal System for the planned SCWO EST indicate that the hexane extraction method can repeatably achieve a detection limit of less than 20 ppb (NIVA Consultants et al., 1999). However, this method not only requires approximately six hours to complete under optimum conditions, it also appears to be sensitive to both the technician performing the analysis and the laboratory (e.g., instrumentation model) where the analysis is carried out. These limitations suggest that further specification and validation will be necessary to achieve reliable results for routine use.

Recommendation 5-4. The Army should develop and demonstrate methods of chemical analysis to confirm the destruction of VX in the hydrolysate at the Newport facility. These methods should include procedural specifications and provisions for training so that confirmation at the required detection limits can be confirmed by testing by different analysts at multiple laboratories.

Reliability and Efficiency of Measurement Methods

Verification of the destruction of mustard in HD hydrolysates has not presented the same technical challenges as VX, but it does require the use of NMR analysis, which takes four to six hours to measure both mustard and sulfonium ions (U.S. Army, 1998b). Verification of agent destruction also constitutes a critical path item in the operational cycle of each facility. Currently, analysis of each batch of hydrolysate takes six hours, provided that reliable analytical results are obtained from the first analysis. Thus, reducing the time required to verify agent destruction in process streams would significantly improve the overall processing efficiency and schedule.

Recommendation 5-5. The Army should develop and demonstrate innovative analytical techniques that require significantly less time than present techniques to verify agent destruction for both VX and HD hydrolysates at the required detection limits.

6

Risk Management

RISK ASSESSMENTS AND HAZARD EVALUATIONS

Several of the recommendations in this report deal with concerns about safety and risk. The Army established a procedure in 1997 for dealing with these matters, which was published in *A Guide to Risk Management Policy and Activities* (U.S. Army, 1997). The *Guide* calls for the following safety and risk evaluations to be completed at the design stage:

- hazard fact sheets in support of the final programmatic environmental impact statement
- Phase 1 and Phase 2 QRAs[1]
- appropriate HEs

A health risk assessment (HRA) must also be prepared but has less impact on operational risk and safety. Figure 6-1 is a diagram showing how these analyses and assessments interact.

In previous reports, the committee has stressed that the safety of operations and storage can best be understood and managed based on the results and models developed in a Phase 2 site-specific QRA (NRC, 1993, 1994a, 1996a, 1996b, 1997, 1999a, 1999b, 1999c). The QRA in turn must be based on a thorough understanding of plant hazards identified in HEs, knowledge of operating practice and campaign management, knowledge of agent toxicology, and models of agent dispersion following accidents. Special attention must be paid to unusual design features and particular hazards at each facility.

A key component for the Phase 2 QRA is a detailed, dynamic model of the cascade ventilation system for each facility that can be used to determine possible agent concentration levels at various points in a plant in case of a power failure, interlock failure, or other exigency. Parallel systems and single systems can be compared to define the alternative with the lowest risk, which would then be included in the final plant design. From the information made available during this study, the committee could not be certain that development of these kinds of analyses (leading to a Phase 2 QRA and associated HEs) is proceeding at a proper pace during the design stage. The committee believes that HE and QRA development must be initiated as soon as possible to ensure that the results are available for the final design and operation of the facilities.

Recommendation 6-1. Quantitative risk assessments and hazard evaluations for both the Newport and Aberdeen facilities should be carried out as early as possible during facility design, and the findings from these assessments should be used in both the design and future operation of the facilities to minimize risk and maximize safety.

SCHEDULE RISK

Another risk that must be considered is associated with the schedule of project development and design, construction, start-up, operations, and shutdown. Schedule delays not only affect cost and meeting CWC obligations, but also increase public risk due to continued storage. The committee is particularly concerned about the schedule for the NECDF because of uncertainties associated with the SCWO process. The committee believes the workability of this process may very well be proved out during a pending EST. However, it is not clear if the EST can be completed quickly enough to prevent causing serious delays to the NECDF project. If the EST takes longer than expected, or if further development in

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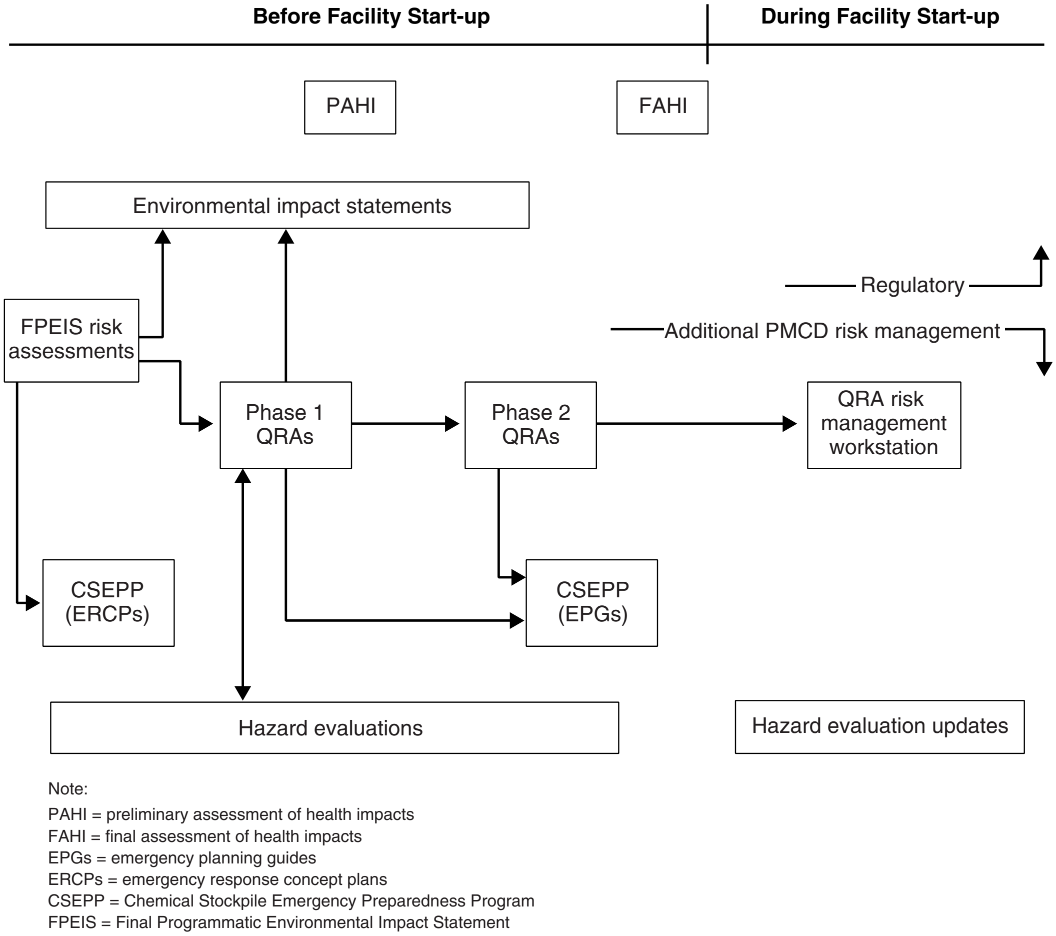
[1] A Phase 1 QRA evaluates public risks from a proposed facility before it is constructed. A Phase 2 QRA is a detailed evaluation of the risks and consequences of accidental releases of agent to workers and the community based on the site-specific design and operations.



Note:
PAHI = preliminary assessment of health impacts
FAHI = final assessment of health impacts
EPGs = emergency planning guides
ERCPs = emergency response concept plans
CSEPP = Chemical Stockpile Emergency Preparedness Program
FPEIS = Final Programmatic Environmental Impact Statement

FIGURE 6-1 Timing of risk management activities. Source: Adapted from U.S. Army, 1997.

some process area is required, continued storage of the agent would be necessary, which would probably increase public risk. The ABCDF schedule is less likely to be delayed because biodegradation is better known and more developed. In 1998, Arthur Anderson reported that although the schedules for all PMCD disposal facilities could conceivably meet PMCD's plans, there was a high probability that the schedules for several facilities, including the ABCDF and the NECDF, would be exceeded (Arthur Anderson, 1998).

Recommendation 6-2. Schedule risk should be evaluated for the Aberdeen and Newport facilities in the context of program implications and public risks and appropriate contingency plans developed if needed.

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Appendixes

Appendix A

Process Description for the Aberdeen Chemical Agent Disposal Facility

The following excerpt is accompanied by a simplified flow diagram for the ABCDF (Figure A-1) and mass balance tables of input and output streams (Tables A-1 and A-2). The numbered streams in Tables A-1 and A-2 correspond to the numbered diamonds in Figure A-1 (U.S. Army, 1998).

1. HD PROCESS OVERVIEW

The process design consists of the following process modules:

HD Ton Container Cleanout (TCC) - One Unit
Secondary Heat Transfer Fluid Circulation - Three Units
Water Storage - One Unit
Reagent Storage and Preparation - One Unit
Neutralization - Three Units
VOC Treatment - One Unit
Biotreatment - One Unit
Effluent Filtration - One Unit

1.1 HD Ton Container Cleanout (One Unit)

The HD TCC module transfers the ton container contents to the Neutralization module and prepares the ton containers for shipment off-site. Two holes are punched in the ton container (TC), agent is drained from the container and transferred to the Agent Holding Tank in the Neutralization Module. After draining, the container is rinsed with a series of three sprays using hot water to remove residual agent and heel in the container. Each container is the cut in two and washed, inside and out, with hot spray water to achieve an XXX decontamination condition. Each container half is steamed and air dried before monitoring to verify the XXX decontamination condition. The XXX container halves are then bagged and readied for shipment to off-site smelting. The heated spray water make-up is supplied from the Hot Process Water Tank in the Reagent Storage and Preparation Module. Container rinse effluent is sent to the TCC Effluent Tank in the Neutralization Module. Wash water is recycled through the Spray Water Tank. Spray water is monitored for pH and, when required, a portion of the Spray Water Tank contents is purged to the TCC Effluent Tank. TCC vent gases are vented to the TCC vent scrubber where caustic is used to scrub agent vapors and acid vapors from the vent path. Spent scrubber liquor is sent to the TCC Effluent Tank. The ventilation exhaust is sent to a dedicated carbon filter and then to the site cascade ventilation system.

1.2 Secondary Heat Transfer Fluid Circulation Module (Three Units)

The Secondary Heat Transfer Fluid Circulation module meets the heat transfer needs of the processes carried out in the Neutralization module. There are three identical units in the Secondary Heat Transfer Fluid Circulation Module. Each unit serves one of the three identical neutralization unit toxic cubicles. The Secondary Heat Transfer Fluid Circulation Module provides a boundary between toxic systems and the environment and prevents contamination of plant cooling water by toxic process fluids in the event of heat exchanger leaks.

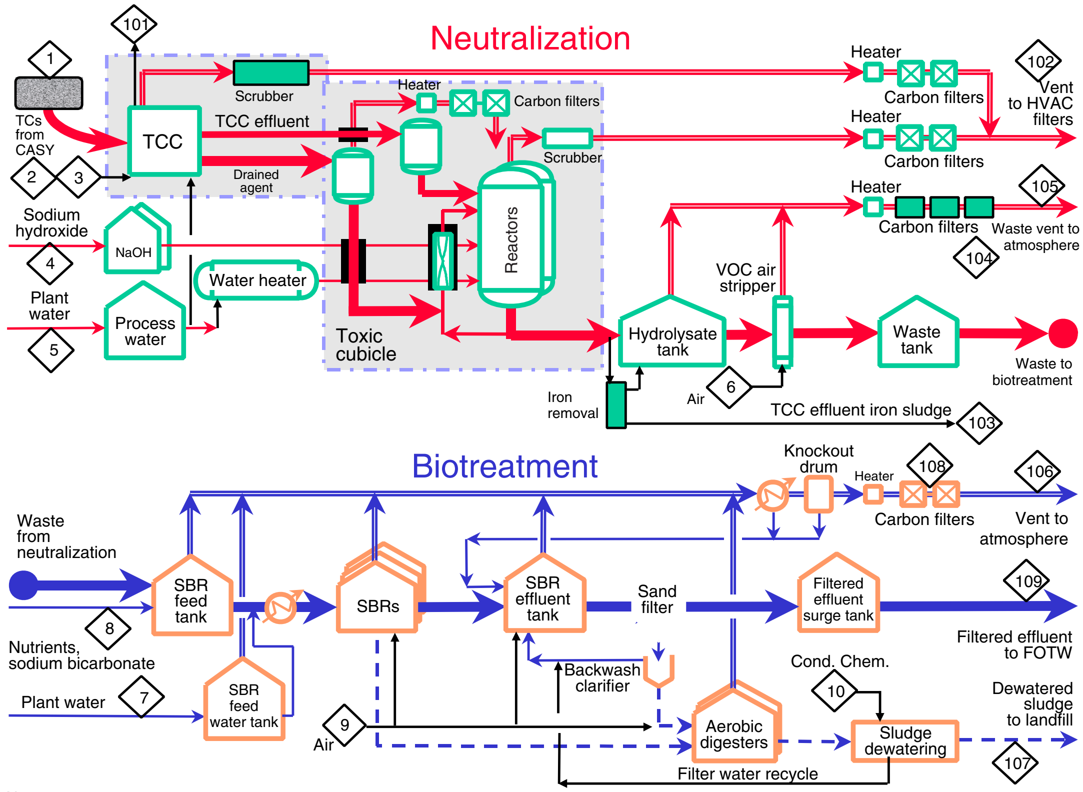
1.3 Water Storage Module (One Unit)

The Water Storage Module stores water for use in the TCC, Reagent Storage and Preparation, Neutralization, Biotreatment, and Effluent Filtration Modules. Process water is received from the plant water supply and is stored in the Process Water Tank.

1.4 Reagent Storage and Preparation Module (One Unit)

The Reagent Storage and Preparation Module heats process water for use in the TCC and Neutralization Modules, stores sodium hydroxide for use in the neutralization reaction, and dilutes and stores sodium hydroxide for decontamination uses.

Cold process water is received from the Water Storage Module. Eighteen percent sodium hydroxide is delivered by truck and stored undiluted for use in the neutralization reaction.



Note:
CASY = chemical agent storage yard

Single-digit numbers in diamonds refer to the input streams in Table A-1.
Triple-digit numbers in diamonds refer to output streams in Table A-2.

FIGURE A-1 Simplified flow diagram for the ABCDF. Source: U.S. Army, 1998.

1.5 Neutralization Module (Three Units)

The Neutralization Module accepts the ton container contents from the TCC module and destroys the agent batchwise through hydrolysis with water followed by caustic addition. The Neutralization Module consists of three units, each located inside a Containment Level A toxic cubicle. There are two HD Reactors and one TCC Effluent Tank in each of the three neutralization units. In each neutralization unit, drained agent held in the Agent Holding Tank is processed in batch neutralization reactions. The rinse and spray water from the TCC Module and spent decontamination solution are stored in the TCC Effluent Tank and process in separate batch reactions. In the neutralization reaction HD reacts with water to yield the principal hydrolysis products of thiodiglycol and hydrochloric acid. After the hydrolysis is complete and sample analysis results confirm the destruction of HD, 18 percent sodium hydroxide is added to the reactor to raise pH in order to increase the hydrolysate biodegradability. The hydrolysate is then pumped to the Hydrolysate Tank in the VOC Treatment Module.

The vent streams from the HD reactors and the TCC Effluent Tank are combined, scrubbed with a sodium hydroxide scrubber, condensed and passed through dedicated carbon filters before entering the site cascade ventilation system.

1.6 VOC Treatment Module (One Unit)

The VOC Treatment Module is designed to reduce VOC sufficiently to avoid producing Biotreatment sludge that would be a hazardous waste.

The VOC Treatment Module consists of two air stripping units and three portable carbon filter units. One air stripper is in use and the other is on standby or being cleaned. Hydrolysate from the Hydrolysate Tank is cooled to reduce temperature in the carbon beds before being fed to the air stripper. After air stripping the hydrolysate is stored in the Waste Tank for transfer to the Biotreatment Module. The VOC laden air stream from the air stripper is passed through three carbon beds to remove the VOC before the air stream is vented to the atmosphere. The carbon filters are recycled to the portable carbon filter unit vendor for regeneration.

TABLE A-1 Process Inputs for HD/Water Neutralization with Biodegradation

	TCC Clean-out, Neutralization, and VOC Air Stripping						Biodegradation				
Stream Number Description	1 TCs with Agent	2 TCC Air	3 TCC Steam	4 Neutral NaOH (aq)	5 Neutral Water	6 VOC Air Stripper	7 Biodegradation Water	8 Nutrients and Buffer	9 Air	10 Conditioning Chemicals	Total Inputs
Total flow (kg/1,000 kg)	1,000.00	1,152	234	262	24,066	4,261	58,770	1,039	68,310	72	159,166
Pressure (psig)			125								
Temperature (°F)	70	70	351	70	70	70	70	70	70	70	
Physical state (solid (S), liquid (L), gas (G))	S,G & L	G	G	L	L	G	L	S & L	G	L	
Components											
HD	828.00										828
Sodium hydroxide				47							47
Water		6	234	215	24,066	21	58,770		342	52	83,706
Sulfur containing impurities	131.00										131
Chlorinated aliphatic hydrocarbons	6.00										6
$NaHCO_3$								963			963
KNO_3											
KCl								14			14
Na_2SO_4											
NH_3								35			35
Na_3PO_4											
Wolin salts								8			8
H_3PO_4								19			19
Organics											
Polymer										4	4
Iron chlorides	35.00										35
O_2		266				985			15,797		17,048
N_2		879				3,253			52,140		56,272
CO_2		1				2			31		34
Biosolids											
$Fe(OH)_3$										16	16
Activated carbon (66 kg Estimated)											
TC shells (No./1,000 kg)	1.23										
TC valves (No./1,000 kg)	2.49										
TC plugs (No./1,000 kg)	7.45										
TC cuttings (3 lb./TC estimated)											

1.7 Biotreatment Module (One Unit)

The Biotreatment Module is designed to biologically degrade thiodiglycol and other compounds in the stripped hydrolysate using sequencing batch reactors (SBRs).

The stripped hydrolysate is transferred from the Waste Tank in the VOC Treatment Module to the SBR Feed Tank. Nutrients are added, the pH is adjusted and the hydrolysate is diluted with water in preparation for the transfer to the SBRs. Sodium bicarbonate solution is added to the SBRs to neutralize the acids formed during biotreatment and to maintain pH. Biodegradation of the organics in the stripped hydrolysate produces carbon dioxide, water, biomass

TABLE A-2 Process Outputs for HD/Water Neutralization with Biodegradation

	TCC Clean-out, Neutralization, and VOC Air Stripping						Biodegradation			
Stream Number	101	102	103	104	105	106	107	108	109	
Description	TC Metals	Vent Gas	Iron Clarifier Sludge	Activated Carbon	VOC Stripper Vent	Vent Gas	Biomass	Activated Carbon	Water Effluent	Total Outputs
Total flow (kg/1,000 kg)		1,152	964	67	4,261	68,876	894	4	83,014	159,232
Pressure (psig)										
Temperature (°F)	70	110	90	70	140	100	90	70	93	
Physical state (solid (S), liquid (L), gas (G))	S	G	L	S	G	G	S	S	L	
Components										
TDG			8							8
Sodium hydroxide										
Water		6	875		21	689	695		81,467	83,753
Sulfur containing impurities			28							28
Chlorinated aliphatic hydrocarbons				5						5
NaCl			34				5		671	710
$NaNO_3$									29	29
$NaNO_2$									8	8
KNO_3									2	2
KCl									7	7
Na_2SO_4							5		746	751
NH_3										
Na_3PO_4									7	7
Wolin salts									8	8
H_3PO_4										
Organics							1		69	70
Polymer							4			4
Iron chlorides			19							19
O_2		266			985	14,907				16,158
N_2		879			3,253	52,212				56,344
CO_2		1			2	1,068				1,071
Biosolids							168			168
$Fe(OH)_3$							16			16
Activated carbon (estimated)				62				4		66
TC shells (No./1,000 kg)	1.23									
TC valves (No./1,000 kg)	2.49									
TC plugs (No./1,000 kg)	7.45									
TC cuttings (3 lb./TC estimated)										

(sludge), and salt. Sludge, which is settled and withdrawn daily from the SBRs, is biologically stabilized in aerobic digesters and then thickened and dewatered prior to disposal. The supernatant from the SBRs is pumped to the Effluent Filtration Module. Vent gases from the aerated tanks in the Biotreatment Module are passed through carbon filters prior to their release to the atmosphere.

1.8 Effluent Filtration Module (One)

The Effluent Filtration Module removes most of the remaining suspended solids from the SBR supernatant in the Continuous Backwash Sand Filter. The filter effluent is stored in the Filtered Effluent Surge Tank for transfer to the Federally Owned Treatment Works.

SUPPLEMENTAL INFORMATION

Modification of the Treatment Steps for Volatile Organic Compounds

In the original acquisition design package (ADP), the volatile organic compound (VOC) stream was processed by wetted-wall evaporation followed by hydrogen peroxide mediated ultraviolet irradiation and liquid-phase carbon adsorption (shown in Figure A-2). However, this process was subsequently replaced by a simpler, cheaper process—air stripping followed by gas-phase carbon adsorption (shown in Figure A-3). VOC treatment facilities are located in the process auxiliary building.

Biotreatment

The biotreatment plant is designed to operate 24 hours per day, seven days per week, and to handle hydrolysate from 7,000 pounds of agent per day. The design throughput is less than the overall plant design capacity of 10,000 lbs of agent per day, which means the biotreatment plant can handle the effluent from the neutralization section only if the latter is operated five days per week, or at 70 percent of capacity. The four sections of the biotreatment plant are described below.

In the feed preparation section, the air-stripped hydrolysate is biotreated so it can eventually be handled in the same federally owned treatment works (FOTW) that handles other wastes from Aberdeen Proving Ground. Nutrients are added to the hydrolysate to promote biological growth in the reactors; sodium bicarbonate is added as a buffer to keep the reaction mixture from acidifying. Aqueous ammonia, phosphoric acid, potassium chloride, and other nutrients are then added. The sequencing biological reactor (SBR) feed is diluted to a concentration of 1 volume percent, the maximum concentration of organics or salts that can be treated in the reactors.

In the SBRs, the soluble organics in the hydrolysate are converted to a mixture of insoluble biomass (that can be settled and separated), water, and carbon dioxide. The ADP calls for three parallel SBR systems to ensure flexibility for low or high feed rates. Each SBR system has its own aeration system. The design criteria call for a hydraulic retention

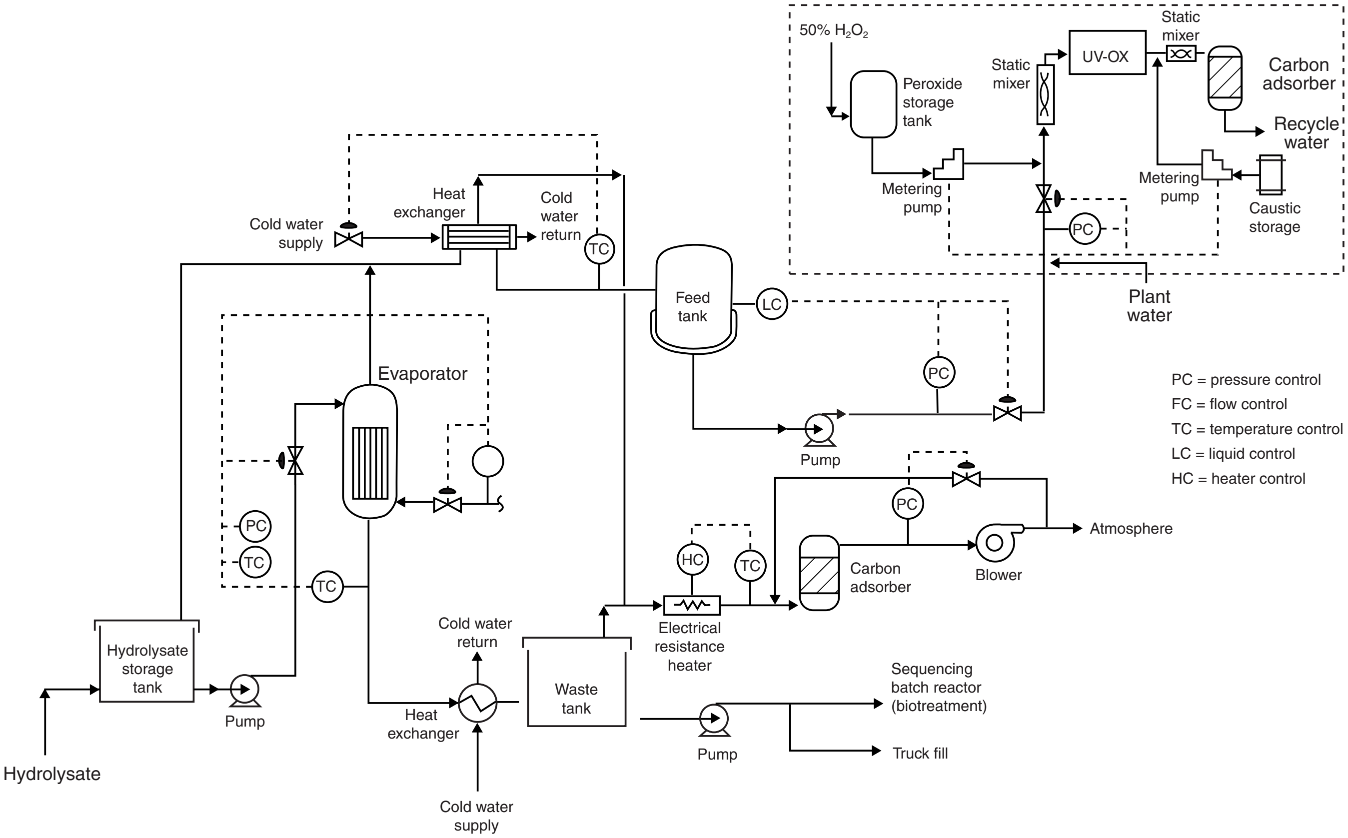


FIGURE A-2 ADP VOC treatment system, wetted-wall evaporation followed by ultraviolet radiation and liquid-phase carbon adsorption. Source: Myler, 1999.

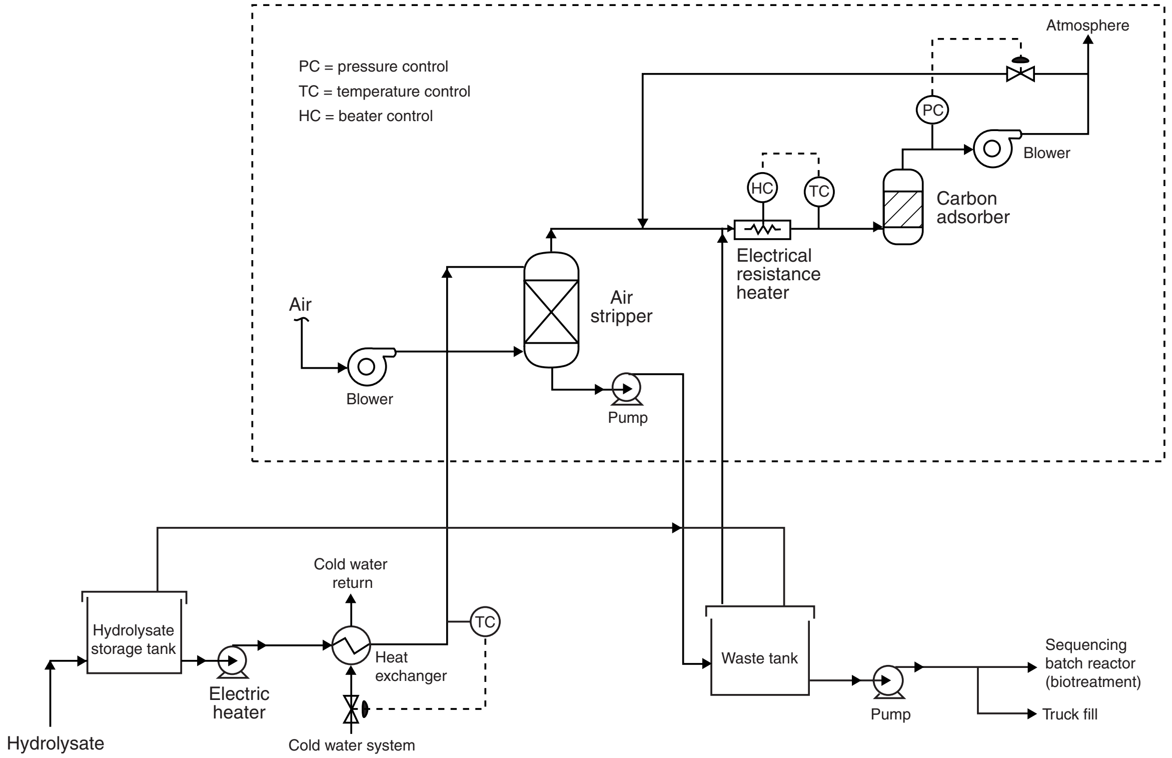


FIGURE A-3 Current VOC treatment system, air stripping followed by gas-phase carbon adsorption. Source: Myler, 1999.

time of 10 days, a solid retention time of 15 days, and a mixed-liquor suspended-solids concentration of 4,000 mg/liter.

Aerobic digestion reduces the amount of solids generated, as well as the odors produced in sludge-handling facilities. Aerobic digestion can also be a source of biomass to seed the SBRs. The design basis is for a 10-day residence time, which should meet the standards of the Environmental Protection Agency for a specific oxygen consumption of less than 1.5 mg oxygen/gm of suspended solids per hour.

In the sludge-thickening and dewatering section, polymer is added to thicken the sludge from 1 percent total suspended solids content to about 3 percent. Ferric chloride and diatomaceous earth are then added, prior to dewatering in conventional filter presses. The filter cake will have a total solids content of about 20 percent, which is high enough that no free liquids will be present.

Filtration of Effluent

A continuously backwashed sand filter is provided to remove 90 percent of the suspended solids in the supernatant liquids from the SBRs and the filtrates from the thickener and filter press. The filtered effluent is discharged to the FOTW via truck or pipeline. The settled solids are recycled to the aerobic digesters.

Waste Streams

The process produces three waste streams—air emissions, wastewater, and solid waste.

Air Emissions

Four gaseous process streams are vented to the atmosphere: (1) gases from the toxic ventilation system handling exhaust from the toxic cubicles, (2) gases from a similar ventilation system treating exhaust from the clean-out of ton containers (TCC), (3) gases from a separate ventilation system for the VOC system, and (4) gases from a fourth ventilation system that processes biotreatment off-gas. To remove any traces of agent, the gas from the toxic cubicles is scrubbed with caustic soda before it passes through carbon filters and into the atmosphere. The TCC system is similar.

TABLE A-3 Volume of Off-Gas

Source	Standard Cubic Feet per Minute
Toxic cubicles	84
Ton container clean-out (TCC)	6,000
VOC treatment (with ultraviolet treatment step)	88
Biotreatment	5,406

Source: Adapted from U.S. Army, 1998.

Air stripper off-gas from the VOC system in the process auxiliary building is passed through carbon filters. The gases from the biotreatment plant also pass through deep-bed carbon filters. The amounts of off-gas handled by these four systems in standard cubic feet per minute (scfm) are shown in Table A-3.

In addition, nonprocess air emissions are produced by three boilers, two diesel-driven auxiliary generators, a laboratory filter vent, the medical decontamination area, and a vent from the sodium bicarbonate storage tank.

Wastewater

Wastewater from sumps in each toxic cubicle is treated with caustic and then handled as hydrolysate. The 9,540 lbs/day of bottom deposits from the hydrolysate clarifier is a slurry of solids that is hauled to an off-site hazardous waste treatment facility. The 581,000 lbs/day of effluent from the effluent filtration module is sent by pipeline to the Aberdeen FOTW (U.S. Army, 1998).

Nonprocess wastewater is discharged directly into the existing system at Aberdeen Proving Ground. However, all wastewater from the utility areas is passed through an oil-water separator before being discharged. Sanitary wastewater is handled in the normal treatment system.

Solid Waste

Decontaminated ton containers are sent to the Rock Island Arsenal for smelting and reclamation. The 6,260 lbs/day of biosolids filter cake is sent to an off-site disposal facility. Spent activated carbon from the various filters, estimated to be generated at a rate of 600 lbs/day, will be tested for 3X surety and sent to the same contractor that takes other carbon filter material from facilities at Aberdeen Proving Ground. Demilitarization protective ensembles, which are at the 3X level after normal decontamination procedures, will be placed in 55 gallon drums, stored in a solid waste storage area, and then shipped off site.

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Appendix B

Process Description for the Newport Chemical Agent Disposal Facility

The following excerpt is accompanied by a simplified flow diagram for the NECDF (Figure B-1) and mass balance tables of input and output streams (Tables B-1 and B-2). The numbered streams in Tables B-1 and B-2 correspond to the numbered diamonds in Figure B-1 (U.S. Army, 1998).

1. VX PROCESS OVERVIEW

The Newport Chemical Agent Disposal Facility is designed to destroy the stockpile of nerve agent VX stored in ton containers at the Newport Chemical Depot. The overall process employed in the facility design is divided into three major process steps:

Ton Container Cleanout (TCC) - removal of agent from ton containers and decontamination of the ton containers prior to shipment

Neutralization - destruction of VX resulting in a non-agent product (hydrolysate)

Post Treatment - oxidation of organic compounds in the hydrolysate through supercritical water oxidation (SCWO) and removal of inorganic salts through evaporation

1.1 Ton Container Cleanout (TCC)

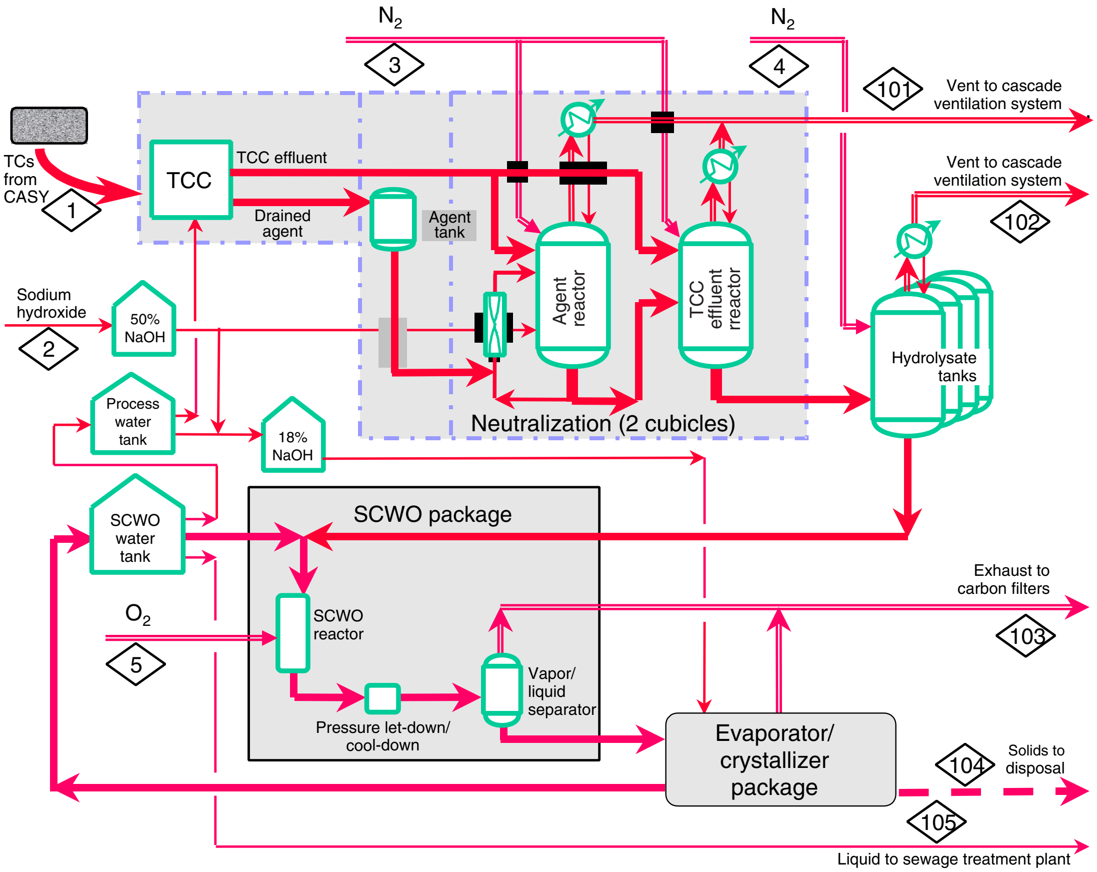
The ton container cleanout process consists of draining agent from ton containers, decontaminating the containers to a 3X condition, and preparing the containers for off-site shipment. The draining of bulk agent is accomplished in the Bulk Drain Station by punching two holes in each ton container and inserting a drain tube into one hole. Liquid agent remaining after draining is removed with a vacuum nozzle inserted through one hole. After removal of liquid agent, any residual agent is washed from the surface of the ton container by halving the container and spraying it with hot process water directed through high pressure spray nozzles. Each container is steamed and air dried, monitored to verify that a 3X condition has been achieved, and readied for shipment to offsite smelting. Agent drained from the ton containers is referred to as drained agent, while the effluent from the washing of ton containers is termed TCC effluent. All vent gases from the TCC area are passed to the building cascade ventilation system where they are filtered prior to release to the atmosphere.

1.2 Neutralization

Drained agent is neutralized through batchwise reaction with caustic at 194°F. The neutralization reaction converts VX to several non-agent organic products, and the resulting mixture is referred to as drained agent hydrolysate. After reaction, the drained agent hydrolysate is combined with TCC effluent. The combined mixture is reacted to destroy agent in the TCC effluent, and the resulting mixture is called hydrolysate. The hydrolysate is sampled and held until it is confirmed that the VX is 99.9999% destroyed (no more than 330 ppb VX) and cannot be detected. The concentration of EA 2192 must be below 20 ppm. Vent gases from the reactors are contained during the reaction, but are vented and partially condensed during the initial filling of each reactor. The vent condensate is returned to the reactors, and the vapor effluent ultimately routed to the building cascade ventilation system.

1.3 Post Treatment

Non-agent organic compounds in the hydrolysate are destroyed with supercritical water oxidation (SCWO), which mineralizes the organics at high temperature and pressure to form water, innocuous gases, and inorganic salts. Water is added to the hydrolysate prior to the SCWO reaction to adjust the heating value of the feed. Oxygen, the oxidant for the reaction, is injected directly into the SCWO reactor. A quench stream of water is added to the reactor effluent to cool the effluent below its critical temperature. After separation of the product gases, the pH of the SCWO aqueous effluent is adjusted with sodium hydroxide and the stream is evaporated to remove salts. The evaporator condensate is recycled to supply SCWO feed and quench water and process



Note:
CASY = chemical agent storage yard
Single-digit numbers in diamonds refer to the input streams in Table B-1.
Triple-digit numbers in diamonds refer to output streams in Table B-2.

FIGURE B-1 Simplified flow diagram for the NECDF. Source: U.S. Army, 1998.

water for washing ton containers and diluting caustic. The salt-concentrated evaporator bottoms are crystallized and filtered, then sent to a properly permitted offsite landfill. The excess evaporator condensate is sent to a sewage treatment plant (STP). Post treatment vent gases are passed through carbon filters prior to discharge to the atmosphere. Since the SCWO package is vendor-supplied, the final configuration may differ from what is described. The process description of SCWO provided in this document represents a typical configuration.

SUPPLEMENTAL PROCESS INFORMATION

Operation of Supercritical Water Oxidation

Two independent supercritical water oxidation (SCWO) reactor trains are located in separate rooms in the SCWO building. Normally, one train is in operation and the other on standby. The SCWO reactors are plug-flow tubular reactors about 10 inches in diameter and 15 feet long. At the start of each batch run, fuel oil is used as a start-up feed to bring the unit up to operating temperature and pressure. The feed is then switched to hydrolysate at a rate of 2,092 lbs/hr, along with 937 lbs/hr of water. Oxygen is introduced separately at a rate of 1,313 lbs/hr, 150 percent of the stoichiometric requirement. The unit operates under severe conditions of 1,200°F and 4,000 psig. Residence time is about 60 seconds. The reaction stream is quenched with 7,555 lbs/hr of water as it leaves the reactor, the pressure is let down, and vapor streams totaling 1,170 lbs/hr are mixed with air and sent through a carbon filter before being released to the atmosphere. The 10,727 lb/hr liquid-solid outlet stream is mixed with caustic to maintain a pH of 9 before it is fed to an evaporator feed tank.

TABLE B-1 Process Inputs for VX Neutralization/Posttreatment

Stream number Description	1 Agent	2 NaOH (aq)	3 N_2 to Reactors	4 N_2 to Hydrolysate Tanks	5 Oxygen	Total Inputs
Total flow		1197.00	5.78	0.89	3,500.00	5,703.67
Pressure (psia)					3,500.00	
Temperature (° F)	70	80	70	70	70	
Physical state (solid (S), liquid (L), gas (G))	L	L	G	G	G	
Components						
$CH_3P(O)(OEt)(SR)$ VX	956.50					956.50
Water						598.50
NaOH						598.50
Air						0.00
Oxygen					3,500.00	3,500.00
Nitrogen			5.78	0.89		6.67
Agent impurities						
Diisopropylamine	0.03					0.03
Diisopropylcarbodiimide (stabilizer)	11.90					11.90
O-ethyl methylethylphosphinate	0.21					0.21
Diethyl methylphosphonate	0.41					0.41
2-(diisopropylamino) ethane thiol	6.50					6.50
O,O-diethyl methylphosphonothioate	0.98					0.98
O,S-diethyl methylphosphonothioate	0.46					0.46
2-(diisopropylamino) ethyl ethyl sulfide	0.81					0.81
Diethyl dimethylpyrophosphonate (Pyro)	7.20					7.20
O,O-diethyl dimethylpyrophosphonothioate	1.24					1.24
O-(2-diisopropylaminoethyl) O-ethylmethylphosphonate	1.70					1.70
1,2-bis(ethyl methylphosphonothiolo)ethane	4.50					4.50
Unknowns	7.56					7.56
Ton containers (No./1,000kg)						1.47
Ton container valves (No./1,000kg)						3.06
Ton container plugs (No./1,000kg)						9.19

Source: U.S. Army, 1998.

Evaporator/Crystallizer

Material in the evaporator feed tank goes to an evaporator/crystallizer. The SCWO product contains about 4 weight percent salt. The evaporator system can handle about 125 percent of the 11,122 lbs/hr of caustic-treated feed from each SCWO reactor. It operates continuously, producing 24,870 lbs/hr of condensate and 574 lbs/hr of solids.

WASTE STREAMS

Air Emissions

The ton container clean-out (TCC) system, neutralizers, and hydrolysate storage sections all produce small amounts of vent gas, totaling about 2.7 lbs/hr. In the cascade ventilation system, these gases are passed through two-stage carbon filters and are then exhausted to the atmosphere. A separate gas-handling system with carbon filters processes the 1,170 lbs/hr of posttreatment gases.

Liquid Waste

No liquid waste is produced under normal operating conditions. Although most of the requirements for the SCWO operation are met by the evaporator/crystallizer condensate, some supplemental plant water is required.

Solid Waste

Salt produced in the evaporator/crystallizer is sent to a suitable hazardous waste treatment, storage, and disposal facility. The clean ton containers and various metal parts exiting the TCC are sent to the Rock Island Arsenal for

Table B-2 Process Outputs for VX Neutralization/Posttreatment

Stream number	101	102	103	104		105	
Description	Neutralization Vent	Hydrolysate Vent	SCWO/Evaporation Vent	Solid Effluent	TC Clean-out Parts	Liquid Effluent	Total Outputs
Total flow (kg/1,000kg)	5.80	0.93	3,121.00	1,535.00	See below	1,042.00	5,704.73000
Pressure (psia)							
Temperature (°F)	46	40	80	95		100	
Physical state (solid (S), liquid (L), gas (G))	G	G	G	S		L	
Components							
$CH_3P(O)(OEt)(SR)$ VX							
Water	0.0317	0.00345	70.22	462.00		1,042.00	1,574.25520
NaOH							
Air							
Oxygen			1,165.22				1,165.22000
Nitrogen	5.74	0.922					6.66200
CO_2			1,803.44				1,803.44000
N_2O			82.11				82.11000
Nox			0.022				0.02200
CO			0.0089				0.00890
Volatile organic compound			0.00056				0.00056
$(NO_3)^-$				0.691			0.69100
Ethyl methylphosphonic acid				0.277			0.27700
Methylphosphonic acid				0.137			0.13700
Thiols				0.00778			0.00778
$(HCO_3)^-$				9.45			9.45000
Total organic carbon				0.113			0.11300
Na_2SO_4				531.26			531.26000
Na_2HPO_4				531.04			531.04000
Total Metals				0.0702			0.07020
O,O-diethyl methylphosphonothioate							
O,S-diethyl methylphosphonothioate							
2-(diisopropylamino) ethyl ethyl sulfide							
Diethyl dimethylpyrophosphonate (Pyro)							
O,O-diethyl dimethylpyrophosphonothioate							
O-(2-diisopropylaminoethyl)							
O-ethylmethylphosphonate							
1,2-bis(ethyl methylphosphonothiolo)ethane							
Unknowns							
Ton containers (No./1,000kg)					1.47		1.47
Ton container valves (No./1,000kg)					3.06		3.06
Ton container plugs (No./1,000kg)					9.19		9.19

Source: U.S. Army, 1998.

smelting and recycling. Spent filter carbon is checked for detectable agent before being sent to an outside contractor for disposal. Demilitarization protective ensembles, which are at the 3X level after normal decontamination procedures, will be packed in 55 gallon drums, stored in a solid waste storage area, and shipped off site.

REFERENCE

U.S. Army. 1998. Acquisition Design Package, Vol. 2. Newport Chemical Agent Disposal Facility. February 1998. CD-ROM prepared by Stone and Webster Engineering Corporation for the Department of the Army. Aberdeen Proving Ground, Md.: Office of the Product Manager for Alternative Technologies and Approaches.

Appendix C

Biographical Sketches of Committee Members

David S. Kosson *(chair)* has a B.S. in chemical engineering, an M.S. in chemical and biochemical engineering, and a Ph.D. in chemical and biochemical engineering from Rutgers, The State University of New Jersey. Currently, he is chairman and professor of the Department of Civil and Environmental Engineering and professor of chemical engineering at Vanderbilt University. Previously, he was professor of chemical and biochemical engineering at Rutgers. Dr. Kosson has carried out research and published extensively on subsurface contaminant transport phenomena, leaching phenomena, physical, chemical, and microbial treatment processes for hazardous waste, and waste management policy. He has been a member of the National Research Council (NRC) Committee on Alternative Chemical Demilitarization Technologies and the Panel on Review and Evaluation of Alternative Chemical Disposal Technologies.

Charles E. Kolb *(vice chair)* is president and chief executive officer of Aerodyne Research, Inc. He earned an S.B. degree in chemistry from the Massachusetts Institute of Technology and an M.A. and Ph.D. in physical chemistry from Princeton University. Since 1971, his principal research interests at Aerodyne have included atmospheric and environmental chemistry, combustion chemistry, materials chemistry, and the chemical physics of rocket and aircraft exhaust plumes. He has served on several National Aeronautics and Space Administration panels dealing with atmospheric chemistry and global change, as well as on five NRC committees and boards dealing with environmental issues. From 1996 to 1999, Dr. Kolb was atmospheric sciences editor for *Geophysical Research Letters.* In 1997, he received the Award for Creative Advances in Environmental Science and Technology from the American Chemical Society.

David H. Archer, a member of the National Academy of Engineering, has a Ph.D. in chemical engineering and mathematics from the University of Delaware. He is a retired consulting engineer with the Westinghouse Electric Company and is currently adjunct professor at Carnegie Mellon University. Dr. Archer has worked in both industry (at Westinghouse as an engineer, supervising engineer, department manager, and consulting engineer) and academia (at the University of Delaware and Carnegie Mellon University for almost 10 years). He has considerable experience in research and management related to chemical engineering, as well as experience with combustion and plant management.

Piero M. Armenante has a Ph.D. in chemical engineering from the University of Virginia and is currently professor of chemical engineering at the New Jersey Institute of Technology. He is also the director of the Northeast Hazardous Substance Research Center, a multi-university research center funded by the Environmental Protection Agency (EPA). Dr. Armenante's research interests include multiphase mixing in agitated systems, the biological treatment of hazardous waste, industrial sterilization processes, and biomedical engineering. He has an extensive list of peer-reviewed and other publications and has administered numerous grants, studies, and projects.

Dennis C. Bley is president of Buttonwood Consulting, Inc., and a principal of The WreathWood Group, a joint venture company that supports multidisciplinary research in human reliability. He has more than 25 years of experience in nuclear and electrical engineering, reliability and availability analysis, plant and human modeling for risk assessment, diagnostic system development, and technical management. Dr. Bley has a Ph.D. in nuclear engineering from the Massachusetts Institute of Technology and is a registered professional engineer in the state of California. He has served on a number of technical review panels for U.S. Nuclear Regulatory Commission and U.S. Department of Energy programs and is a frequent lecturer in short courses for universities, industries, and government agencies. He is active in many professional organizations and is on the Board of Directors of the International Association for Probabilistic Safety

Assessment and Management. Dr. Bley has published extensively on subjects related to risk assessment. His current research interests include applying risk analyses to diverse technological systems, modeling uncertainties in risk analyses and risk management, technical risk communication, and human reliability analyses.

Jerry L.R. Chandler has a Ph.D. in biochemistry from Oklahoma State University and has done extensive postgraduate studies in mathematics. He is currently a research professor at the Krasnow Institute for Advanced Study at George Mason University. During his long career, Dr. Chandler served with the U.S. Public Health Service, the National Institute for Occupational Safety and Health (NIOSH), the Food and Drug Administration, and the National Cancer Institute Epidemiology Program. More recently, he was a neuropharmocologist in the Epilepsy Branch of the National Institute of Neurology and Stroke at the National Institutes of Health. Dr. Chandler is a founding member and president of the Washington Evolutionary Systems Society and has published extensively on using mathematical category theory to understand the origins of disease. He previously served as a NIOSH observer with the National Academy of Sciences/ NRC Panel on Risk Assessment.

Frank P. Crimi is a part-time consultant and retired vice president of Lockheed Martin Advanced Environmental Systems Company. He has a B.S. in mechanical engineering from Ohio University and has done graduate studies in mechanical engineering at Union College in Schenectady, New York. In addition to his appointment to the NRC Committee on Decontamination and Decommissioning of Uranium Enrichment Facilities, Mr. Crimi has firsthand knowledge and experience with radioactive and hazardous waste treatment and disposal technologies.

J. Robert Gibson is global director, Facilities Planning and Administration, DuPont Life Sciences, E.I. du Pont de Nemours and Company, and an adjunct associate professor of marine studies at the University of Delaware. Since receiving his Ph.D. in physiology from Mississippi State University, Dr. Gibson has specialized in toxicology. He has been certified by the American Board of Toxicology and is the author of numerous publications.

Michael R. Greenberg is a professor in the Department of Urban Studies and Community Health at Rutgers, The State University of New Jersey, and an adjunct professor of environmental and community medicine at the Robert Wood Johnson Medical School. His principal research and teaching interests include urbanization, industrialization, and environmental health policy. Dr. Greenberg has a B.A. in mathematics and history, an M.A. in urban geography, and a Ph.D. in environmental and medical geography.

Kathryn E. Kelly received her doctorate in public health from Columbia University, with a concentration in environmental toxicology and the health effects of hazardous waste incineration. She also studied toxicology at the New York University Institute of Environmental Medicine. Dr. Kelly is the founder and president of three companies: Delta Toxicology, Inc., Crystal Bay, Nevada; Environmental Toxicology International, Seattle, Washington; and Alden Analytical Laboratories, Seattle, Washington. She has broad experience in toxicology, waste combustion, environmental policy, and risk communication.

Peter B. Lederman is executive director of the Hazardous Substances Management Research Center, executive director of the Office of Intellectual Property, and research professor of chemical engineering and environmental policy at the New Jersey Institute of Technology. He received his Ph.D. in chemical engineering from the University of Michigan. Dr. Lederman has 47 years of experience in all facets of environmental management, control, and policy development; hazardous substance treatment and management; process engineering; and more than 18 years of experience as an educator. He is a registered professional engineer and a diplomate of the American Academy of Environmental Engineers. Dr. Lederman has worked on environmental policy at the federal and state levels and has served on several NRC committees, most recently the Committee on Decontamination and Decommissioning of Gaseous Diffusion Plants.

James F. Mathis, a member of the National Academy of Engineering, graduated from the University of Wisconsin with a Ph.D. in chemical engineering. Dr. Mathis was vice president of science and technology for Exxon Corporation, where he was responsible for worldwide research and development programs, and chair of the New Jersey Commission on Science and Technology until his retirement in 1997. Dr. Mathis' expertise is in research and development and chemical engineering.

Charles I. McGinnis has an M.E. from Texas A&M University. He is a registered professional engineer in Texas and Missouri, and a Fellow of the American Society of Civil Engineers. He retired from the U.S. Army as a major general and former director of civil works for the U.S. Army Corps of Engineers and has recently served in senior positions at the Construction Industry Institute in Austin, Texas. He was director of engineering and construction for the Panama Canal Company and subsequently vice president of the company and lieutenant governor of the Canal Zone. As director of civil works for the U.S. Army Corps of Engineers, he was responsible for a $3 billion per year budget for the planning, design, construction, operation, and maintenance of public works nationwide.

Charles F. Reinhardt has an M.D. from Indiana University School of Medicine and an M.Sc. in occupational medicine from Ohio State University School of Medicine. He recently retired after more than 30 years with the DuPont Company, where he was a plant physician, a physiologist, chief of the physiology section, research manager for environmental sciences, assistant director, and finally director of DuPont's Haskell Laboratory, a position he held until his retirement in 1996. Dr. Reinhardt has considerable expertise and experience in occupational medicine and toxicology and has served on numerous NRC panels and committees, including the Committee on Toxicology.

H. Gregor Rigo attended Ohio University and earned his Ph.D. in mechanical and environmental engineering from the University of Illinois. He is currently president of Rigo & Rigo Associates, Inc., in Berea, Ohio. He has extensive experience in plant start-up, process and environmental engineering, and applied statistics focused on the use and control of emissions from nontraditional fuels; technical, environmental, and economic evaluations; and multipathway health risk assessments.

Kozo Saito has a Ph.D. in mechanical engineering from Seikei University in Tokyo and is currently professor of mechanical engineering in the Department of Mechanical Engineering at the University of Kentucky. Dr. Saito's expertise and experience are in experimental combustion studies, thermal sensing and control, and lean manufacturing and control. He is a member of the Combustion Institute, the American Society for Engineering Education, and the American Society of Mechanical Engineers.

W. Leigh Short earned his Ph.D. in chemical engineering from the University of Michigan. He recently retired as a principal and vice president of Woodward-Clyde, where he was responsible for management and business development associated with the company's hazardous waste services in Wayne, New Jersey. Dr. Short has expertise in air pollution, chemical process engineering, hazardous waste services, feasibility studies, site remediation, and project management. He has taught courses in control technologies, both to graduate students and as a part of the EPA's national training programs. He has also served as chairman of the EPA's NO_x Control Technology Review Panel.

Arnold F. Stancell, a member of the National Academy of Engineering, graduated from the Massachusetts Institute of Technology with a Sc.D. in chemical engineering. Dr. Stancell is currently a professor of chemical engineering at the Georgia Institute of Technology and was a recent visiting professor in chemical engineering at the Massachusetts Institute of Technology. For many years, he worked for Mobil Oil, where he started in research and eventually became vice president of Mobil Chemical and then vice president in the crude oil/natural gas exploration and production business, both domestic and international. Dr. Stancell's expertise is in the management of large businesses, including chemical operations.

Steven R. Tannenbaum, a member of the Institute of Medicine, has a Ph.D. in food science and technology from the Massachusetts Institute of Technology, where he is currently the codirector and Underwood-Prescott Professor, Division of Bioengineering and Environmental Health, and professor of chemistry, Department of Chemistry. Dr. Tannenbaum's research interests include the chemistry and pathophysiology of nitric oxide, the quantitative measurement of human exposure to carcinogens, and tissue-based microsensors for toxin detection and drug metabolism. He has been a member of the NRC Board on Environmental Studies and Toxicology and has served on several NRC committees.

Chadwick A. Tolman received his Ph.D. in physical chemistry from the University of California at Berkeley and is currently a program officer in organic and macromolecular chemistry in the Division of Chemistry at the National Science Foundation. He has extensive experience and expertise in chemistry and chemical process development. Dr. Tolman spent 31 years in central research and development, DuPont Experimental Station. His work has spanned a broad range of subjects, including hydrocarbon oxidation, organometallic chemistry, and the destruction of toxic organic compounds in wastewater.

William Tumas graduated from Ithaca College with a B.A. in chemistry and earned his Ph.D. in organic chemistry from Stanford University, with a National Science Foundation and Hertz Foundation Fellowship. After conducting postdoctoral research in organometallic chemistry at the California Institute of Technology as a National Institutes of Health and Chaim Weizman Postdoctoral Fellow, he worked for six years at DuPont Central Research and Development. Since 1993, Dr. Tumas has been at Los Alamos National Laboratory, where he is currently group leader of the Chemical and Environmental Research and Development Group in the Chemical Sciences and Technology Division. He has previously served on the NRC Panel on Review and Evaluation of Alternative Chemical Demilitarization Technologies (1995–1996) and one other NRC committee. His research interests include catalysis, supercritical fluids, environmental chemistry, and waste treatment technology assessment.