Evaluation of Demonstration Test Results of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: A Supplemental Review for Demonstration II

Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: Phase II, Board on Army Science and Technology,National Research Council

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A Supplemental Review for Demonstration II

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Demilitarization of Assembled Chemical Weapons: Phase II

Board on Army Science and Technology
Division on Engineering and Physical Sciences
National Research Council

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Preface

The United States has been in the process of destroying its chemical munitions for well over a decade. Initially, the U.S. Army, guided by recommendations from the National Research Council (NRC), decided to use incineration as its destruction method at all sites. However, citizens in some states with stockpile storage sites oppose incineration on the grounds that the exact nature of the effluents escaping from the stacks cannot be determined. The Army has continued to pursue incineration at four of the eight storage sites in the continental United States where that process seemed appropriate. Nevertheless, influenced by growing public opposition to incineration and the 1996 NRC report *Review and Evaluation of Alternative Chemical Disposal Technologies*, the Army has also been developing technologies based on chemical hydrolysis for the remaining sites. These processes will be used to destroy the VX nerve agent stored at Newport, Indiana, and the mustard agent stored at Aberdeen, Maryland, both of which are stored only in bulk one-ton containers and not in assembled munitions.

In 1996, persuaded by public opposition in Lexington, Kentucky, and Pueblo, Colorado, Congress enacted Public Law 104-201, which instructed the U.S. Department of Defense (DoD) to "conduct an assessment of the chemical demilitarization program for destruction of assembled chemical munitions and of the alternative demilitarization technologies and processes (other than incineration) that could be used for the destruction of the lethal chemical agents that are associated with these munitions." In response, the Army established the program manager for the Assembled Chemical Weapons Assessment (PMACWA). In Public Law 104-208, the PMACWA was required to "identify and demonstrate not less than two alternatives to the baseline incineration process for the demilitarization of assembled chemical weapons." During the first phase of the Assembled Chemical Weapons Assessment (ACWA) program, seven technologies were evaluated. Three of them proceeded to demonstration testing (Demo I) and one was dropped completely. In August 1999, the PMACWA selected two of the Demo I technologies as candidates for the destruction of the assembled munitions weapons at Pueblo Chemical Depot. The two packages, General Atomics Total Solution (GATS) and Parsons/Honeywell (formerly Parsons-Allied Signal) water hydrolysis of explosives and agent technology (WHEAT), were advanced to the engineering design study phase of the ACWA program.

The PMACWA has involved the citizen stakeholders in every aspect of the program, including the procurement process. The Keystone Center, a nonprofit organization, was hired to facilitate public involvement through a process known as the Dialogue, which has become a model for public involvement in matters of public concern.[1]

The Congress mandated that the Army coordinate with the NRC during the ACWA program. In response, the NRC established the Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons (ACW I committee) in 1997 to oversee this program. The question before the committee was not whether incineration was an adequate technology for destroying assembled chemical weapons but whether other chemical processes acceptable to the stakeholders could be

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[1] The U.S. Department of Energy and the National Aeronautics and Space Administration have both adopted this approach. For example, at the NASA Jet Propulsion Laboratory, the Dialogue process will be used in developing a Mars sample-return mission, which is scheduled for 2012.

used. The second NRC committee (ACW II committee) was established in the spring of 2000 to evaluate the two engineering design studies for the destruction facilities at Pueblo, Colorado, and Richmond, Kentucky, and to evaluate the demonstration testing of the three technology packages that had not been selected for those sites or for previous demonstration testing.

Although the PMACWA had no intention of demonstrating these three technologies, Public Law 106-79 (2000) mandated that the PMACWA "conduct evaluations of [the] three additional alternative technologies under the ACWA program." Furthermore, the PMACWA was directed to "proceed under the same guidelines as contained in Public Law 104-208 and continue to use the Dialogue process and Citizens' Advisory Technical Team and their consultants." Accordingly, the PMACWA initiated a program commonly referred to as Demo II to demonstrate the three technologies (AEA SILVER II™, the Foster Wheeler/Eco Logic/Kvaerner integrated demilitarization process, and Teledyne-Commodore's solvated electron process) that had not been selected during the first phase. The ACW II committee was asked to determine if and how the Demo II results affected its commentary, findings, and recommendations and the steps that were suggested for implementation in the ACW I report. This report presents the committee's evaluation of the second set of demonstration tests.

I wish to gratefully acknowledge the hard work of members of the ACW II committee, all of whom served as volunteers and provided the expertise necessary to carry out this enormous task. They gave relentlessly and unselfishly of their time and effort throughout the study. Their areas of expertise included chemical processing, biological remediation, environmental regulations and permitting, energetic materials, and public acceptance. Committee members attended plenary meetings, visited the technology providers' headquarters and test sites, observed design-review sessions, and studied the extensive literature, including engineering charts and diagrams, provided by the technology providers.

On behalf of the committee, I would like to also express appreciation for the extensive support of the Army ACWA team and its interactions with stakeholders and the Dialogue, particularly the group's Citizens Advisory Technical Team, whose members attended all open meetings of the committee and shared information and views with it. The committee also appreciated the openness and cordiality of the representatives of the technology providers. They and the Army provided early drafts of their test reports and other documentation to facilitate the committee's evaluation.

A study such as this requires extensive logistic support; the committee is indebted to the NRC staff for their assistance. I would particularly like to acknowledge the close working relationship I had with the NRC study director, Patricia Paulette. We worked as a team in leading this study. We spoke on the phone daily and e-mailed each other incessantly. The efforts of William Campbell, who took extensive notes and provided real-time report corrections at all our meetings as well as suggestions on how to best organize the report, were invaluable to the committee and to me. Gwen Roby provided the logistic support that enabled us to concentrate on our task. I am also indebted to my colleagues in the Chemistry Department at the University of Southern California who willingly took over my teaching duties while I traveled on behalf of this study.

Robert A. Beaudet, *Chair*
Committee on Review and Evaluation
of Alternative Technologies for
Demilitarization of Assembled
Chemical Weapons: Phase II

Acknowledgments

This report has been reviewed in draft form by individuals chosen for their diverse perspectives and technical expertise, in accordance with procedures approved by the NRC's Report Review Committee. The purpose of this independent review is to provide candid and critical comments that will assist the institution in making its 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 review comments and draft manuscript remain confidential to protect the integrity of the deliberative process. We wish to thank the following individuals for their review of this report:

Steven Konkel, Eastern Kentucky University
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Martin B. Sherwin, Chemical Engineer (retired)
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Although the reviewers listed above have provided many constructive comments and suggestions, they were not asked to endorse the conclusions or recommendations nor did they see the final draft of the report before its release. The review of this report was overseen by Royce Murray, University of North Carolina, appointed by the National Research Council. He was responsible for making certain that an independent examination of this report was carried out in accordance with institutional procedures and that all review comments were carefully considered. Responsibility for the final content of this report rests entirely with the authoring committee and the institution.

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Acronyms, Chemical Symbols, and Abbreviations

ACWA	Assembled Chemical Weapons Assessment (program)
ACW I	Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons
ACW II	Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: Phase II
AEA	AEA Technologies Corporation
Ag^{2+}	silver II ions
AgCl	silver chloride
a-HAX	solution containing potassium hydroxide and humic acid
BIF	boiler and industrial furnace
CATOX	catalytic oxidation
CEES	chloroethyl ethyl sulfide
CEM	continuous emission monitor
CO	carbon monoxide
CO_2	carbon dioxide
Composition B	an energetic material that contains (nominally) 59.5 percent RDX, 39.5 percent TNT, and 1.0 percent wax
CWC	Chemical Weapons Convention
DAAMS	depot area air monitoring system
Demo I	Demonstration I (demonstration testing of three technologies selected for the first phase of ACWA technology testing)
DMMP	dimethyl methylphosphonate
DoD	U.S. Department of Defense
DPE	demilitarization protective ensemble
DRE	destruction and removal efficiency
ECBC	Edgewood Chemical and Biological Center
EDP	engineering design package
EDS	engineering design study
EPA	Environmental Protection Agency

FEK or FW/EL/K	Foster Wheeler/Eco Logic/Kvaerner
GATS	General Atomics Total Solution
GB	a nerve agent
GC/MS	gas chromatography/mass spectrometry
GPCR™	gas-phase chemical reduction
H_2	hydrogen
HD	distilled mustard agent
HF	hydrofluoric acid
HNO_3	nitric acid
HPLC	high-performance liquid chromatography
HRA	health risk assessment
ICI	Imperial Chemical Industries
IMPA	isopropyl methylphosphonic acid
IRS	impurities removal system
KOH	potassium hydroxide
LMIDS	Lockheed Martin Integrated Demilitarization System
MACT	maximum achievable control technology
MDM	multipurpose demilitarization machine
MPA	methylphosphonic acid
M28	energetic material used for propulsion of certain assembled chemical weapons
N_2	nitrogen
NO_x	nitrogen oxides
N_2O	nitrous oxide
NRC	National Research Council
O_2	oxygen
PA	picric acid
PCP	pentachlorophenol
PGB	product gas burner
PMACWA	program manager for the Assembled Chemical Weapons Assessment
PMD	projectile mortar demilitarization (machine)
POTW	publicly owned treatment works
ppm	parts per million
PTFE	polytetrafluoroethylene (Teflon)
QRA	quantitative risk assessment
RCRA	Resource Conservation and Recovery Act
RDX	cyclotrimethylenetrinitramine
RFP	request for proposals
SCWO	supercritical water oxidation

SET™	solvated electron technology
SILVER II™	electrochemical oxidation using silver II ions in nitric acid
SO_x	sulfur oxides
SO_2	sulfur dioxide
SVOC	semivolatile organic compound
TBA	tributylamine
TC	Teledyne-Commodore
TCLP	toxicity characteristic leachate procedure
TNB	trinitrobenzene
TNBA	trinitrobenzoic acid
TNT	trinitrotoluene, an energetic material
TOC	total organic carbon
TRBP	thermal reduction batch processor
TW-SCWO	transpiring-wall supercritical water oxidation
VOC	volatile organic compound
VX	a nerve agent
WHEAT	water hydrolysis of explosives and agent technology
3X	At the 3X decontamination level, solids are decontaminated to the point that 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 level for mustard agent is 3.0 mg per cubic meter in air. 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.
5X level	Treatment of solids to a 5X decontamination level is accomplished by holding a material at 1,000°F for 15 minutes. This treatment results in completely decontaminated material that can be released for general use or sold (e.g., as scrap metal) to the general public in accordance with applicable federal, state, and local regulations.
5X treatment unit	This unit is used to heat chemical solid waste materials to a level of decontamination where no residual contamination is detectable.

Executive Summary

By direction of Congress, the U.S. Department of Defense's (DoD's) program manager for the Assembled Chemical Weapons Assessment (PMACWA) asked the National Research Council (NRC) Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: Phase II (the ACW II committee) to conduct an independent scientific and technical assessment of three alternative technologies (referred to as Demo II) under consideration for the destruction of assembled chemical weapons at U.S. chemical weapons storage sites. The three technologies are AEA Technologies Corporation's (AEA's) electrochemical oxidation process; the transpiring-wall supercritical water oxidation and gas-phase chemical reduction processes of Foster Wheeler/Eco Logic/Kvaerner (FW/EL/K); and Teledyne-Commodore's solvated electron process. Each of these technologies represents an alternative to incineration for the complete destruction of chemical agents and associated energetic materials. The demonstration tests were approved by the PMACWA after an initial assessment of each technology. The results of that initial assessment were reviewed by an earlier NRC committee, the Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons (the ACW I committee) (NRC, 1999).

For the present review, the committee conducted an in-depth examination of each technology provider's data, analyses, and demonstration test results for the critical components tested. This review report supplements the ACW I report and considers the demonstration performance of the Demo II candidate technologies and their readiness for advancement to pilot-scale implementation. Because testing in these areas is ongoing, the committee decided to cut short its fact-finding efforts for input to this report as of March 30, 2001. This cutoff was necessary in order to provide the sponsor with the needed information in a timely fashion.

In 1996 the U.S. Congress enacted two laws, Public Law 104-201 (authorization legislation) and Public Law 104-208 (appropriation legislation), mandating that DoD assess alternative technologies to the baseline incineration process for the demilitarization of assembled chemical munitions. In December 1996 the deputy to the commander of the Soldier Biological Chemical Command was appointed as the PMACWA. Subsequently, seven technologies designed for the complete destruction of assembled chemical weapons were evaluated (ACW I report), and on July 29, 1998, three of them were selected for the Demonstration I (Demo I) phase of the ACWA program.

The PMACWA requested that the NRC perform an independent evaluation of the seven technology packages that had been selected originally during earlier phases of the Assembled Chemical Weapons Assessment (ACWA) program and deliver a report by September 1, 1999. However, to meet that deadline, the NRC ACW I committee had to terminate its data-gathering activities on March 15, 1999, before the demonstration tests had been completed (NRC, 1999).

In September 1999, the PMACWA asked the ACW I committee to examine the results of tests demonstrating the operations of three of the original seven alternative technologies and to determine if they had changed the committee's original findings, recommendations, and comments. Accordingly, the NRC published a supplemental report in March 2000 (NRC, 2000), at which time the ACW I committee was disbanded.

In 1999, Congress passed Public Law 105-261, mandating as follows:

> The program manager for the Assembled Chemical Weapons Assessment shall continue to manage the development and testing (including demonstration and pilot-scale testing) of technologies for the destruction of lethal chemical munitions that are potential or demonstrated alternatives to the baseline incineration program. In performing such management, the program manager shall act independently of the program manager for Chemical Demilitarization and shall report to the Under Secretary of Defense for Acquisition and Technology.

The Army was also directed to continue its coordination with the NRC.

Congress extended the PMACWA's task through Public Law 106-79 by mandating that he "conduct evaluations of [the] three additional alternative technologies under the ACWA program, . . . proceed under the same guidelines as contained in Public Law 104-208 and continue to use the Dialogue process and Citizens' Advisory Technical Team and their consultants." In response, the PMACWA initiated a new test program, commonly referred to as Demo II, to investigate whether three of the alternative technologies remaining from the original testing were ready to proceed to an engineering design phase.[1] The remaining technologies were from AEA, FW/EL/K, and Teledyne-Commodore. The seventh of the original technologies had been judged to be too immature for further testing during the original multitiered selection process.

In response to Congress, a second NRC committee, the Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: Phase II (ACW II committee), was formed and tasked to produce three reports: (1) an evaluation of the Demo II tests (Task 1), (2) an evaluation of two engineering design studies (EDSs) and tests for use at the Pueblo, Colorado, storage site (Task 2), and (3) an evaluation of EDS packages and tests for the Blue Grass, Kentucky, site (Task 3).

The statement of task for Task 1 is as follows:

> At the request of the DoD's Program Manager for Assembled Chemical Weapons Assessment (PMACWA), the NRC Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons will provide independent scientific and technical assessment of the Assembled Chemical Weapons Assessment (ACWA) program. This effort will be divided into three tasks. In each case, the NRC was asked to perform a technical assessment that did not include programmatic (cost and schedule) considerations.
>
> Task 1
>
> To accomplish the first task, the NRC will review and evaluate the results of demonstrations for three alternative technologies for destruction of assembled chemical weapons located at U.S. chemical weapons storage sites. The alternative technologies to undergo demonstration testing are: the AEA Technologies electrochemical oxidation technology, the Teledyne Commodore solvated electron technology, and the Foster Wheeler and Eco Logic transpiring wall supercritical water oxidation and gas phase chemical reduction technology. The demonstrations will be performed in the June through September 2000 timeframe. Based on receipt of the appropriate information, including: (a) the PMACWA-approved Demonstration Study Plans, (b) the demonstration test reports produced by the ACWA technology providers and the associated required responses of the providers to questions from the PMACWA, and (c) the PMACWA's demonstration testing results database, the committee will:
>
> - Perform an in-depth review of the data, analyses, and results of the unit operation demonstration tests contained in the above and update as necessary the 1999 NRC report, Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons (the ACW report).
> - Determine if any of the AEA Technologies, Teledyne Commodore, and Foster Wheeler/Eco Logic technologies have reached a technology readiness level sufficient to proceed with implementation of a pilot-scale program.
> - Produce a report for delivery to the PMACWA by July 2001 provided the demonstration test reports are made available by November 2000. (An NRC report delivered in March 2000 covered the initial three technologies selected for demonstration phase testing.)

In this current supplemental review, which responds to Task 1, the ACW II committee provides an extensive review of the data, analyses, and demonstration test results for critical components of the demilitarization processes of AEA, FW/EL/K, and Teledyne-Commodore. Like the first supplemental review (NRC, 2000), this review evaluates the effects of the new test results on the findings and recommendations in the original ACW I committee report (NRC, 1999) and assesses the level of maturity attained by each technology for proceeding to the engineering design phase of development. A separate chapter is devoted to each technology, and the chapters are organized as follows: descriptions of the demonstrated unit operations; descriptions of the tests used in the study, including committee commentary; a discussion of the effects of the demonstration results on previous findings; and, finally, new findings derived from this supplemental review. Chapter 5 considers the earlier general findings and recommendations and presents new ones in light of the demonstration test results.

In general, very few of the original findings and recommendations were changed as a result of the new tests. In some cases, the original findings and recommendations were confirmed. The new findings and recommendations are presented below by technology. The level of development of unit operation processes from the candidate technologies is summarized in Table ES-1. General findings and recommendations are also presented below.

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[1] The AEA, Eco Logic, and General Atomics technology packages were chosen by the PMACWA to undergo engineering design studies for the destruction of the assembled chemical weapons at the Blue Grass Army depot. This decision was made by the PMACWA prior to the issuance of this NRC report.

TABLE ES-1 Summary Evaluation of the Maturity of Demo II Unit Operations and Processes

	Hydrolysates			Agent Munitions			
Technology Provider/Unit Operation or Process	VX/GB	HD	Energetics	VX/GB	HD	Energetics	Other
AEA							
SILVER II™[a]				C	C	C	
Solid/liquid waste treatment				C	C	C	
Gaseous waste treatment				D	D	D	
Foster Wheeler/Eco Logic/Kvaerner							
TW-SCWO	B	B	C				
GPCR™				B	B	B	B[b,c]
Teledyne-Commodore							
Ammonia fluid jet cutting and washout system				D	D	E	
SET™				D	D	D	C[b]
Persulfate oxidation (agent)				D	D	D	
Peroxide oxidation (energetics)				D	D	D	
Metals parts and dunnage shredding							A[b,c]

NOTE: Environmental and safety issues were considered in assigning maturity categorizations. Schedule and cost issues were not considered. The letter designations are defined as follows (a blank space indicates that categorization was not applicable for that material): A, demonstration provides sufficient information to justify moving forward to full-scale design with reasonable probability of success; B, demonstration provides sufficient information to justify moving forward to the pilot stage with reasonable probability of success; C, demonstration indicates that unit operation or process requires additional refinement and additional demonstration before moving forward to pilot stage; D, not demonstrated, and more R&D is required; and E, demonstrated unit operation or process is inappropriate for treatment.

[a] Includes integrated gas polishing system to support demonstration.

[b] Dunnage.

[c] Metal parts.

SUPPLEMENTAL FINDINGS AND RECOMMENDATIONS

AEA Demonstration Test

Finding DII AEA-1. The overall process flow has been further complicated by major design changes in response to the Demo II testing. These changes include the addition of the impurities removal system (IRS), catalytic oxidation (CATOX) units, and a flow return circuit from the catholyte to the anolyte circuit. All three changes require small-scale and pilot-scale testing. Such modifications further complicate the interfaces between process units, which increases the time required for development, start-up, and commissioning of the full-scale system. Integration of the operating units will make achievement of a viable total solution very difficult.

Finding DII AEA-2. The discovery of organic material migration across the electrochemical cell membrane will require major modifications in design and operation, such as recycling of the catholyte material to the anolyte circuit and the addition of hydrocyclones in the catholyte circuit.

Finding DII AEA-3. The formation of intermediate oxidation by-products raises operational issues, including slower processing rates and reduced electrochemical efficiency. During the testing with tetrytol in the 12 kW unit, the problems were severe enough to cause the runs to be extended well beyond the planned processing times.

Finding DII AEA-4. The generation of new energetic compounds trinitrobenzoic acid, picric acid, and trinitrobenzene (TNBA, PA, and TNB) in the course of processing increases the complexity and hazards of the SILVER II™ process. Although the explosion hazard is reduced as the energetic feed is consumed, it is not completely eliminated until all energetic intermediates are destroyed.

Finding DII AEA-5. During the treatment of M28 in the Demo II test, lead oxide and other materials accumulated on cell anodes. The committee believes that a maintenance procedure for routine cleaning of the anodes will be required.

Finding DII AEA-6. Low steady-state electrochemical efficiencies (20 to 30 percent) were observed during treatment of tetrytol. These low efficiencies will decrease the throughput per cell and increase processing time and energy consumption.

Finding DII AEA-7. Volatile organic compounds (VOCs) were detected in the off-gas of the AEA process technology. AEA has now included a CATOX unit in the preliminary design. The committee believes that the introduction of this additional unit operation will further complicate the scale-up and integration.

Finding DII AEA-8. The IRS for removing salts (sulfates, phosphates, silver fluoride), excess water, and any metals that may be present requires extensive development and integration. The IRS has not yet been described in sufficient detail to allow for a meaningful assessment.

Recommendation DII AEA-1. The possible formation of lead picrate when mixed energetic feeds are treated must be investigated before any processing of lead-containing propellant, TNT-based energetics, or tetryl is undertaken.

Recommendation DII AEA-2. The IRS, the CATOX units, the return flow, and all other major modifications to the system must be tested and proven during the EDS design phase.

Recommendation DII AEA-3. AEA must validate complete destruction of all energetic intermediates during the EDS design phase.

Recommendation DII AEA-4. AEA must conduct additional tests to identify suitable materials of construction to overcome corrosion problems encountered owing to the formation of hydrofluoric acid (HF) in the treatment of GB.

Foster Wheeler/Eco Logic/Kvaerner Demonstration Tests

Finding DII FEK-1. The proposed full-scale TW-SCWO system has design and operating conditions significantly different from those tested in Demo II. These include the temperature of the transpiration water at the inlet; pH of the feed; turbulence in the reactor; and use of pure oxygen, not air, as the oxidant.

Finding DII FEK-2. The proposed full-scale design for the TW-SCWO system involves a scale-up in reactor cross-sectional area by a factor of 2 from the Demo II test unit and an increase in reactor throughput by a factor of 35. Performance under these full-scale design conditions has not been demonstrated.

Finding DII FEK-3. Aluminum present in the hydrolysates, which could lead to the formation of slurries and plugging, could be a problem. The proposed changes for mitigating this problem (e.g., changing operating conditions and/or removing aluminum during weapon disassembly) must be tested.

Finding DII FEK-4. Demo II tests confirmed that firing tubes and other solids could be treated to a 5X condition by the GPCR™ process.

Finding DII FEK-5. All waste streams have been or can be characterized sufficiently for engineering design to proceed.

Finding DII FEK-6. The current sampling and monitoring systems for agent in gaseous streams have not been certified or validated for use with the GPCR™ process off-gas.

Finding DII FEK-7. The product gas from the GPCR™ process does not meet the EPA syngas requirements because of high benzene and polyaromatic hydrocarbon content.

Finding DII FEK-8. While no agent was detected in the scrubbing solutions and scrubber filters, the ability of the GPCR™ process to destroy HD in mortars and neat GB could not be confirmed because sampling and analysis problems hampered the gathering of gas-phase data.

Finding DII FEK-9. Little evidence of soot formation was indicated when the GPCR™ unit was tested separately with PCP-spiked wood, HD mortars, M55 rocket firing tubes, and neat GB.

Finding DII FEK-10. The full-scale SCWO reactor design has not been tested and is different in size and in the flow rates of the feed streams from those used in the Demo II tests. The full-scale design treats hydrolysate at a rate per unit volume of reactor that is almost 10 times higher than that used during the Demo II tests. In addition, the ratio of the flow rates of all other streams to the flow rate of hydrolysate in the full-scale unit has decreased by approximately a factor of 10 from those used during the Demo II tests. These changes in hydrolysate processing per unit of reactor volume and the reduction of other feed streams relative to the hydrolysate may reduce the efficacy of the SCWO reactor and may be expected to exacerbate problems of corrosion and plugging.

Finding DII FEK-11. The experience of multiple shutdowns during Demo II testing of the TW-SCWO and the resulting thermal stresses and crack generation in the liner indicate a potential reliability issue, which must be significantly reduced or eliminated.

Recommendation DII FEK-1. Since the hydrolysate/total feed ratio and flow velocity used in Demo II testing are so different from those of the proposed design, the TW-SCWO reactor must be tested at a hydrolysate/total feed ratio and flow velocities close to the proposed design conditions.

Recommendation DII FEK-2. Long-term testing of appropriately designed SCWO reactor liners under the new operating conditions for the proposed full-scale operation will be necessary to prove the reliability and effectiveness of the TW-SCWO unit.

Recommendation DII FEK-3. Long-term testing of the TW-SCWO should include feeds containing chlorine, phosphorus, and sulfur and be at residence times and flow velocities close to the proposed design conditions.

Recommendation DII FEK-4. The Army or the technology provider must develop analytical methods to determine the quantities of agent in the gas streams containing hydrogen.

Teledyne-Commodore Demonstration Tests

Finding DII TC 1. Demo II tests were delayed and could not be completed for the Teledyne-Commodore process because of incidents in which the immaturity of the process became apparent. For example, an exothermic reaction between ammonia vapor and M28 propellant led to an ignition incident. At another time, Composition B, dissolved in liquid ammonia, leaked through flanges into valves and piping that were intended to transfer the material from the ammonia fluid jet-cutting vessel to the SET™ reactor. These incidents revealed serious safety problems associated with the Teledyne-Commodore process.

SUPPLEMENTAL GENERAL FINDINGS

General Finding DII 1. The demonstration tests were not operated long enough to show reliability in long-term operation. The PMACWA's Demo II tests were required to be of the same duration as the Demo I tests. The technology providers had neither the time nor the resources for extensive systemization (preoperational testing) in Demo II. Consequently, these tests were simply proof-of-concept demonstrations that indicate whether or not a particular unit operation (with more development) might be applicable to the disposal of assembled chemical munitions.

General Finding DII 2. The AEA technology package is a very complex, immature chemical processing system. Several new unit operations required to address problems revealed in the Demo II tests will significantly increase the complexity of an integrated processing system and extend the time required for its development.

General Finding DII 3. The demonstrated components of the FW/EL/K technology package are ready to progress to the EDS phase. However, certain key units were not tested (or the results were inconclusive). Additional testing will be needed to verify the ability of the transpiring-wall technology to minimize corrosion; the testing should be carried out in parallel with development of an engineering design.

General Finding DII 4. Because of fire and safety problems, the basic process for the Teledyne-Commodore technology was not tested in Demo II. The Army decided against going forward because the Demo II goals could not be met in time. As a result, the committee had no technical basis on which to evaluate the process any further.

General Finding DII 5. As was true for Demo I, none of the unit operations tested in Demo II has been integrated into a complete system. The lack of integration is a major concern and a significant obstacle to full-scale implementation.

SUPPLEMENTAL GENERAL RECOMMENDATIONS

General Recommendation DII 1. Further development of the Teledyne-Commodore technology package for the destruction of assembled chemical weapons should not be pursued under the ACWA program.

General Recommendation DII 2. Before the AEA technology proceeds to the EDS phase, extensive testing should be performed on the SILVER II™ process, including all the new unit operations that are being proposed to address the shortcomings identified in Demo II results.

General Recommendation DII 3. For the FW/EL/K technology package, additional testing should be performed in the EDS phase to complete GPCR™ off-gas characterization and demonstrate long-term operation of the modified TW-SWCO unit.

1

Introduction

BACKGROUND

In 1996 Congress enacted two laws, Public Law 104-201 (authorization) and Public Law 104-208 (appropriation), mandating that the U.S. Department of Defense (DoD) assess technology alternatives for the baseline incineration process for the demilitarization of assembled chemical weapons and conduct demonstration tests of at least two of them. The laws included the following stipulations:

- All funds for the construction of baseline incineration facilities at the Blue Grass Army Depot in Richmond, Kentucky, and Pueblo Chemical Depot in Pueblo, Colorado, should be frozen until the effectiveness of alternative technologies and their ability to comply with safety and environmental requirements were reported to Congress.
- DoD should select a program manager who was not and had never been associated with the baseline incineration program.
- DoD should "coordinate" its activities with the National Research Council (NRC).

In December 1996, DoD appointed the deputy to the commander of the Soldier and Biological Chemical Command to be the program manager for the Assembled Chemical Weapons Assessment (PMACWA). On July 28, 1997, after organizing a staff and establishing a program plan, the PMACWA published a request for proposals (RFP) for a total system solution for the destruction of assembled chemical weapons without using incineration. Twelve proposals were submitted in September 1997. Of these, seven passed the threshold requirements stipulated in the RFP. These seven technologies are summarized in Table 1-1. One of the seven was rejected during the next phase of the selection process. On July 29, 1998, after an elaborate multitiered selection process, three of the remaining six technology packages were selected for demonstration testing (Burns and Roe, 1999; General Atomics, 1999; and Parsons-Allied Signal, 1999). Detailed descriptions of the selection process and of all seven technologies are available in the PMACWA's two annual reports to Congress (DoD, 1997, 1998).

Under both time and budget constraints, the PMACWA decided to focus the demonstration tests on the unit operations in each technology package that were "most critical and least proven," that is, operations that had not been previously used in the disposal of chemical munitions and/or had not been integrated into a complete system for this application. Systemization (preoperational testing) for the unit operations to be tested was conducted from January to March 1999. The demonstration tests were conducted from March to May 1999. On June 30, 1999, reports from the demonstration tests were submitted to the PMACWA by the technology providers. These reports were used to prepare the *Supplemental Report to Congress* that was submitted on September 30, 1999 (DoD, 1999). In 1999, Congress passed Public Law 105-261 (1999) mandating that:

> The program manager for the Assembled Chemical Weapons Assessment shall continue to manage the development and testing (including demonstration and pilot-scale testing) of technologies for the destruction of lethal chemical munitions that are potential or demonstrated alternatives to the baseline incineration program. In performing such management, the program manager shall act independently of the program manager for Chemical Demilitarization and shall report to the Under Secretary of Defense for Acquisition and Technology.

It also directed the Army to continue coordination with the NRC. In response, the PMACWA authorized engineering design studies for the two technologies that successfully completed demonstration testing, the Parsons/Honeywell technology package (hydrolysis followed by biotreatment) and the General Atomics technology package (hydrolysis followed by supercritical water oxidation (SCWO)). The engineering design studies (EDSs) for both Parsons/

TABLE 1-1 Description of the Seven Technology Packages That Passed DoD's Initial Evaluation

Technology Provider[a]	Access to Munitions	Treatment of Agent	Treatment of Energetics	Treatment of Metal Parts	Treatment of Dunnage
AEA	Modified reverse assembly (high-pressure wash, new rocket shearing).	Electrochemical oxidation using silver ions in nitric acid (SILVER II™).	Treated with SILVER II™ process.	High-pressure acid wash; thermal treatment to 5X.[b]	Shredded and treated with SILVER II™ process.
ARCTECH	Modified reverse assembly.	Hydrolysis with a-HAX (humic acid and strong base, KOH).	Hydrolysis with a-HAX.	Hydrolysis with a-HAX; shipped to Rock Island Arsenal for 5X treatment.	Hydrolysis with dilute a-HAX; shipped to landfill.
Burns and Roe	Modified reverse assembly.	Plasma arc.	Plasma arc.	Melted in plasma arc.	Shredded; processed in plasma arc.
General Atomics	Modified reverse assembly; cryofracture for projectiles.	Hydrolysis; supercritical water oxidation (SCWO).	Hydrolysis, SCWO.	Hydrolysis; thermal treatment to 5X.	Shredded; destroyed in SCWO.
Lockheed Martin (Foster/Eco Logic/Kvaerner)	Modified reverse assembly (multiple lines, compact layout, new drain and wash).	Hydrolysis; SCWO; Eco Logic gas-phase chemical reduction (GPCR).	Hydrolysis, SCWO, GPCR.	Hydrolysis; GPCR to 5X.	Hydrolysis; GPCR to 5X.
Parsons	Modified reverse assembly (fluid-jet cutting and energetic washout for rockets).	Hydrolysis; biotreatment. .	Hydrolysis, biotreatment.	Thermal treatment to 5X.	Thermal treatment to 5X.
Teledyne-Commodore	Fluid-jet cutting; access and drain agent; wash out energetics with ammonia.	Solvated electron process in ammonia for reduction; chemical oxidation with sodium persulfate.	Solvated electron process in ammonia for reduction; chemical oxidation with sodium persulfate.	Wash in solvated electron solution; oxidation to 3X;[c] ship to Rock Island Arsenal for 5X treatment.	Crushed or shredded; treated in solvated electron solution; shipped to landfill.

[a] Allied Signal was purchased by the Honeywell Corporation. Therefore, Parsons-Allied Signal is referred to as Parsons/Honeywell in this report. Lockheed Martin decided not to continue as technology provider for its process, so this technology development is continuing with Kvaerner John Brown as the integrator. Foster Wheeler is developing the supercritical water oxidation (SCWO) unit and Eli Eco Logic International is developing the Gas-Phase Chemical Reduction unit (GPCR™); all three were originally teamed with Lockheed Martin.

[b] Treatment of solids to a 5X decontamination level is accomplished by holding a material at 1,000°F for 15 minutes. This treatment results in completely decontaminated material that may be released for general use or sold (e.g., as scrap metal) to the general public in accordance with applicable federal, state, and local regulations.

[c] At the 3X decontamination level, solids are decontaminated to the point that agent concentration in the headspace above the encapsulated solid does not exceed the health-based, 8-hour, time-weighted average limit for worker exposure. The level for mustard agent is 3.0 μg per cubic meter in air. 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.

Honeywell and General Atomics were configured for use at the Pueblo Army Depot in Pueblo, Colorado (EDS I). However, only the engineering design study for General Atomics (EDS II) will be considered for use at the Blue Grass Army Depot in Richmond, Kentucky.

The purpose of the EDSs was to (1) support the certification decision of the Under Secretary of Defense for Acquisition and Technology, as directed by Public Law 105-261; (2) support the development of an RFP for a pilot facility; and (3) support the required documentation for the National Environmental Policy Act and the Resource Conservation and Recovery Act (RCRA) permit application. Each EDS required an engineering design package (EDP) and tests to generate data that had not been obtained during the demonstration test phase.

In 2000, Congress passed Public Law 106-79 mandating that the PMACWA

> conduct evaluations of [the] three additional alternative technologies under the ACWA program, . . . proceed under the same guidelines as contained in Public Law 104-208 and

> continue to use the Dialogue process and Citizens' Advisory Technical Team and their consultants.

The PMACWA then initiated a program, commonly referred to as Demo II, to demonstrate the three technologies that had not been selected during the first phase. The Demo II tests were performed between July and September 2000 by three technology providers: (1) AEA Technologies, (2) Foster Wheeler/Eco Logic/Kvaerner, and (3) Teledyne-Commodore. Based on the test results, these technologies could be considered for the destruction of the chemical weapons at the Blue Grass Army Depot and would progress to the engineering design phase.[1]

ROLE OF THE NATIONAL RESEARCH COUNCIL

The PMACWA requested that the NRC independently evaluate alternative technologies and submit a report by September 1, 1999, a month before the Army's report to Congress was due. After agreeing on a statement of task in March 1997, the NRC formed the Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons (the ACW I committee). The study was officially begun on May 27, 1997. The committee evaluated all seven technology packages that had passed the threshold requirements stipulated in the first RFP. As per the statement of task, the committee did not recommend a best technology or compare any of the technologies with the baseline incineration process in use at some storage sites. Members of the committee visited the demonstration sites prior to systemization of the unit operations in January 1999, but data-gathering activities had to be terminated on March 15, 1999 (before the results of the demonstration tests had been received), to produce a final report by September 1, 1999. The committee's report was submitted for peer review on May 1, 1999, and released to the sponsor and the public on August 25, 1999 (NRC, 1999).

In September 1999, the PMACWA requested that the tenure of the committee be extended to review the results of the demonstration tests (Demo I). The committee was asked to determine if and how the results affected its original findings and recommendations, as well as the suggested steps for implementation (NRC, 1999). In March 2000, the committee published a supplemental report (NRC, 2000) documenting its review of the Demo I test results and the impact of those results on the conclusions of the initial report (NRC, 1999). The committee completed its task at the end of March 2000 and was disbanded.

A second NRC committee, the Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: Phase II (the ACW II committee) was formed in the spring of 2000 and asked to produce three reports: (1) an evaluation of the new demonstration tests (Demo II) and their impact on the findings and recommendations presented in the NRC's ACW I report (NRC, 1999); (2) an evaluation of the EDSs for Pueblo (EDS I); and (3) an evaluation of the EDSs for Blue Grass Depot (EDS II). This report is an evaluation of the Demo II tests and responds to the first task.

In addition to evaluating the test results, the committee was asked to update the findings for these technologies in the ACW I committee's original report (NRC, 1999).

STATEMENT OF TASK

The complete statement of task for the ACW II committee study is given below. The current supplemental review addresses only Task 1.

> At the request of the DoD's Program Manager for Assembled Chemical Weapons Assessment (PMACWA), the NRC Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons will provide independent scientific and technical assessment of the Assembled Chemical Weapons Assessment (ACWA) program. This effort will be divided into three tasks. In each case, the NRC was asked to perform a technical assessment that did not include programmatic (cost and schedule) considerations.
>
> Task 1
>
> To accomplish the first task, the NRC will review and evaluate the results of demonstrations for three alternative technologies for destruction of assembled chemical weapons located at U.S. chemical weapons storage sites. The alternative technologies to undergo demonstration testing are: the AEA Technologies electrochemical oxidation technology, the Teledyne Commodore solvated electron technology, and the Foster Wheeler and Eco Logic transpiring wall supercritical water oxidation and gas phase chemical reduction technology. The demonstrations will be performed in the June through September 2000 timeframe. Based on receipt of the appropriate information, including: (a) the PMACWA-approved Demonstration Study Plans, (b) the demonstration test reports produced by the ACWA technology providers and the associated required responses of the providers to questions from the PMACWA, and (c) the PMACWA's demonstration testing results database, the committee will:
>
> - perform an in-depth review of the data, analyses, and results of the unit operation demonstration tests contained in the above and update as necessary the 1999 NRC report, *Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons* (the ACW report)

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[1] The AEA, Eco Logic, and General Atomics technology packages were chosen by the PMACWA to undergo engineering design studies for the destruction of the assembled chemical weapons at the Blue Grass Army depot. This decision was made by the PMACWA prior to the issuance of this NRC report.

- determine if any of the AEA Technologies, Teledyne Commodore, and Foster Wheeler/Eco Logic technologies have reached a technology readiness level sufficient to proceed with implementation of a pilot-scale program
- produce a report for delivery to the PMACWA by July 2001 provided the demonstration test reports are made available by November 2000. (An NRC report delivered in March 2000 covered the initial three technologies selected for demonstration phase testing.)

Task 2

For the second task, the NRC will assess the ACWA Engineering Design Study (EDS) phase in which General Atomics and Parsons/Honeywell (formerly Parsons/Allied Signal) will conduct test programs to gather the information required for a final engineering design package representing a chemical demilitarization facility at the Pueblo, Colorado, stockpile site. The testing will be completed by September 1, 2000. Based on receipt of the appropriate information, including: (a) the PMACWA-approved EDS Plans, (b) the EDS test reports produced by General Atomics and Parsons/Honeywell, (c) PMACWA's EDS testing database, and (d) the vendor-supplied engineering design packages, the committee will:

- perform an in-depth review of the data, analyses, and results of the EDS tests
- assess process component designs, integration issues, and overarching technical issues pertaining to the General Atomics and the Parsons/Honeywell engineering design packages for a chemical demilitarization facility design for disposing of mustard-only munitions
- produce a report for delivery to the PMACWA by March 2001 provided the engineering design packages are received by October 2000

Task 3

For the third task, the NRC will assess the ACWA EDS phase in which General Atomics will conduct test programs to gather the information required for a final engineering design package representing a chemical demilitarization facility at the Lexington/Blue Grass, Kentucky, stockpile site. The testing will be completed by December 31, 2000. Based on receipt of the appropriate information, including: (a) the PMACWA-approved EDS Plans, (b) the EDS test reports produced by General Atomics, (c) PMACWA's EDS testing database, and (d) the vendor-supplied engineering design package, the committee will:

- perform an in-depth review of the data, analyses, and results of the EDS tests
- assess process component designs, integration issues, and overarching technical issues pertaining to the General Atomics engineering design package for a chemical demilitarization facility design for disposing of both nerve and mustard munitions
- produce a report for delivery to the PMACWA by September 2001 provided the engineering design package is received by January 2001.

SCOPE AND APPROACH OF THIS STUDY

After reviewing the results of the Demo II tests, the committee reviewed and updated the findings and recommendations from the initial ACW I report, as necessary, and made new findings and recommendations. The committee also reviewed and updated the steps recommended by the ACW I committee that would be necessary before each technology could be implemented. The committee was not requested to review cost, schedules, or public acceptability in this report.

In August 2000, the committee began gathering information through briefings by PMACWA staff and representatives of the technology providers, site visits to the facilities where the demonstration tests were being carried out, and attendance at various progress reviews and status updates held by the PMACWA. Draft reports from technology providers on the results of the Demo II tests were made available to the committee on November 17, 2000. The NRC data analysis and report development took place between November 2000 and April 2001. Although the current report is largely based on those data, the final reports from the technology providers were reviewed as they became available to verify that they did not differ from the versions used in writing this report. Appropriate minor changes were made to the draft report as needed.

ORGANIZATION OF THIS REPORT

Chapter 2 describes AEA SILVER II™ technology (electrochemical oxidation process). Chapter 3 presents the FW/EL/K technology (neutralization followed by transpiring-wall supercritical water oxidation and gas-phase chemical reduction). Chapter 4 discusses Teledyne-Commodore's solvated electron process. For each technology package, the test objectives for unit operations are quoted, the steps for completing the process are reevaluated, the pertinent findings of the ACW I committee are reviewed, and the committee's evaluations of the Demo II results are presented. Chapter 5 evaluates the impact of the Demo II tests on the general findings and recommendations of the ACW I committee and presents some new general findings and recommendations. The committee's site visits and meetings are listed in Appendix A, and Appendix B contains biographical sketches of the committee's members.

2

AEA SILVER II™ Technology Process

The AEA SILVER II™ technology is based on the highly oxidizing nature of silver II ions (Ag^{2+}), which are generated by passing an electric current through a solution of silver nitrate and nitric acid in a standard electrochemical cell. Figure 2-1 shows a block diagram of the AEA SILVER II™ total system solution. The first step in the system is a modified reverse-assembly process in which the energetics, agents, and metal parts are separated. The energetic materials are then reduced in size to less than one-eighth-inch particles before further treatment. In the case of the high-pressure washout of bursters, the particle size was reduced to less than 500 microns (µm). The agent and energetic components are destroyed in separate electrochemical processing units. Metal parts and dunnage are thermally treated in a metal parts treater. The solid, liquid, and gaseous effluents are separated and treated to remove reagents so that they can be recycled and to clean the emissions prior to discharge. The AEA Demo II program tested two SILVER II™ processing plants. One used a 2 kW electrochemical processing unit and the other used a 12 kW unit. The 2 kW SILVER II™ plant was used to test the destruction of agents and agent simulants, and the 12 kW plant was used to test destruction of energetic materials and agent simulants.

The 2 kW SILVER II™ system was at the Edgewood Chemical and Biological Center (ECBC) facility and the 12 kW SILVER II™ system was at the Aberdeen Test Center Firebox. More detailed descriptions of the total system solution and the unit operations can be found in the original NRC ACW I report (NRC, 1999).

DESCRIPTION OF THE SYSTEMS

2 kW SILVER II™ System

The 2 kW SILVER II™ demonstration unit installed in the toxic chamber at ECBC (Figure 2-2) consists of an Imperial Chemical Industries (ICI) FM-21 electrochemical cell with a single in-cell flow channel and a single electrode pair. This unit and the associated gas treatment system are not as complex as the 12 kW unit, which is described in detail later in this chapter.

The system was designed to validate the capability of the SILVER II™ process to destroy chemical agents VX, GB (both are nerve agents), and HD (distilled mustard). It was also intended to demonstrate the effective destruction of agent simulants chloroethyl ethyl sulfide (CEES) and dimethyl methylphosphonate (DMMP) for comparison with results from the 12 kW system.

This system was also tested for its ability to handle and treat the silver chloride (AgCl) formed during the processing of HD and CEES to a 5X level. The chlorinated feeds from which the silver chloride was formed were either agents or agent simulants, which were introduced into a premix vessel from which they were metered into the anolyte vessel. A single hydrocyclone was installed in the anolyte circuit to remove the solids and prevent their accumulation in the electrochemical cell. During part of the test, the hydrocyclone discharge was sent back to the anolyte vessel; the rest of the time, discharges were sent to the settling/collection vessel. AgCl crystals that were removed from the recirculating anolyte circuit were later transferred to a 5X treatment unit.

The silver chloride 5X treatment unit was an electrically heated oven with forced-air circulation to prevent cold spots. The temperature of the silver chloride was measured with a thermocouple mounted in the unit. The vent air from the unit containing nitric acid vapors was passed through a condenser prior to flowing into the main plant off-gas system.

12 kW SILVER II™ System

The full-scale unit for the treatment of energetics and agent simulant operates at 300 to 750 kW; thus, the 12 kW test unit is only 2 to 4 percent of the size of the full-scale unit. The process flow diagram for the test unit is shown in Figure 2-3. The 12 kW test unit, which was operated by Army personnel from the Aberdeen Test Center, is a larger

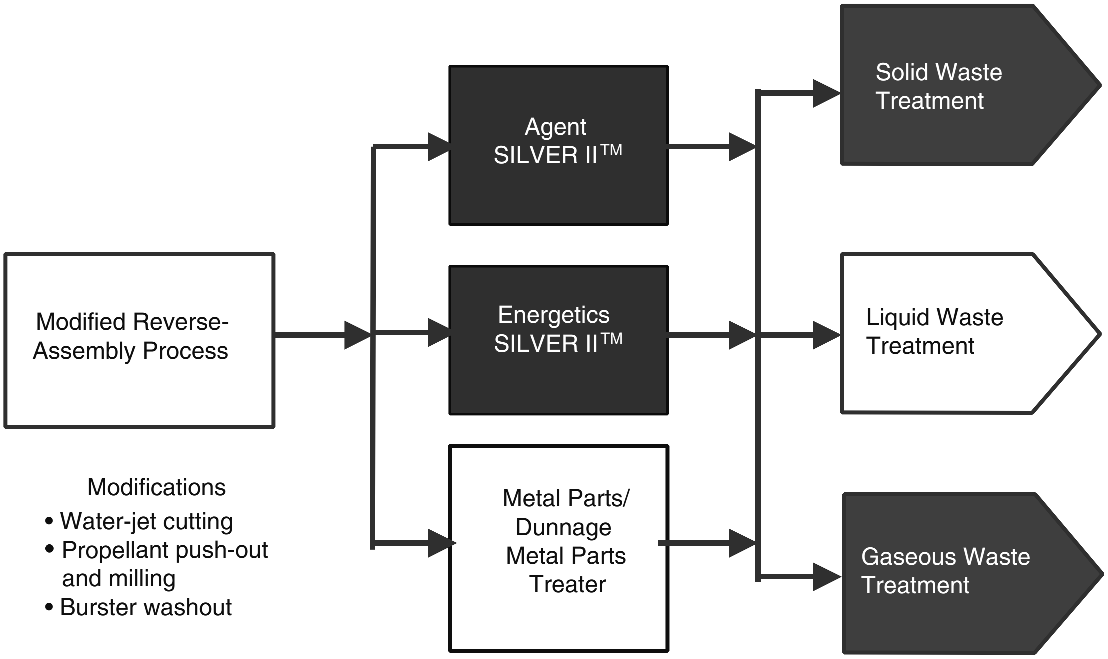

FIGURE 2-1 AEA SILVER II™ total system solution. Solid boxes represent unit operations for demonstration. SOURCE: Adapted from AEA (2000).

version of the 2 kW unit and consists of an ICI titanium electrolytic cell (2 V/6,000 A) with a Nafion™ membrane. The pressure drop across the cell is 0.5 bar during operation. The solution in the anode compartment circulates around a closed loop through a titanium anolyte vessel that has a volume of 600 L. The solution in the cathode compartment circulates around a separate closed loop. Both the anolyte and catholyte circuits are made of either glass or Teflon-lined components. The anolyte circuit, as designed, includes three hydrocyclones in parallel to remove AgCl particles (formed when chlorine-containing compounds are treated) from the liquid stream. The feed system for energetics is charged with premixed water slurries of fine (<500 µm) M28, Composition B[1] (an energetic material), and tetrytol (TNT and tetryl) in batches. These slurries are continuously fed to the anolyte vessel at a rate matching the destruction rate. To determine when the organics are completely destroyed, sensors measure the flow rates, CO (carbon monoxide), CO_2 (carbon dioxide), and the total organic carbon (TOC) content in the anolyte vessel.

The catholyte circuit contains 4M (molar) nitric acid that is reduced to NO_x (mixed oxides of nitrogen) during the process. The NO_x is reoxidized to nitric acid in a reformer that consists of two columns: a water (or dilute acid) absorption column and a distillation column to separate the acid from the water before recycling. The NO_x is absorbed in a column fed with the cooled acid condensate stream from the top of the distillation column. Oxygen is added to the system through the catholyte vessel to oxidize the NO_x to nitric acid. The liquid stream from the absorption column is passed to the distillation column, where the acidic condensate and the concentrated acid are separated.

The concentrated acid is removed from the base of the column and the distillate from the top of the column. The nitric acid produced is recycled to the SILVER II™ system. For the destruction of compounds containing nitrogen (e.g., energetics), a net excess of nitric acid is produced that is removed as concentrated acid from the base of the column. For organic feeds that do not contain nitrogen, all of the concentrated acid must be returned to the catholyte vessel to replace the acid reduced to NO_x in the cell. The anticipated nitric acid recovery is 99.5 percent.

The reformer off-gas is directed to a hypochlorite caustic scrubber. A continuous emission monitor (CEM) is used to monitor scrubber off-gas. The composition of this gas is expected to be primarily carbon dioxide (CO_2) and oxygen (O_2) with small amounts of water, nitrogen oxides (NO_x), sulfur oxides (SO_x), hydrogen (H_2), and carbon monoxide (CO).

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[1] Composition B contains (nominally) 59.5 percent RDX, 39.5 percent TNT, and 1.0 percent wax. The composition is specified in MIL-C-401E.

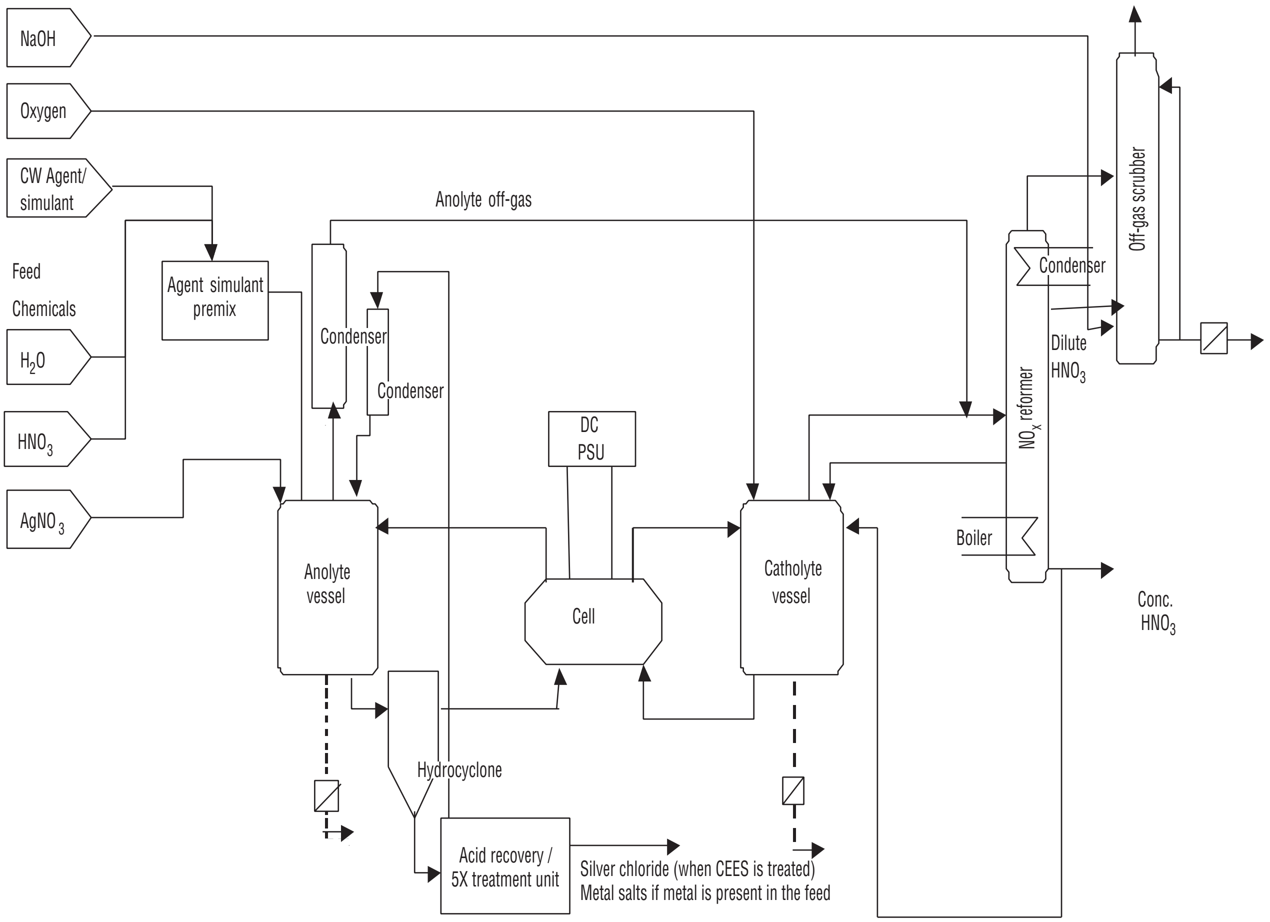

FIGURE 2-2 Process flow diagram of the AEA 2 kW demilitarization process. SOURCE: Adapted from AEA (2000).

TESTING

Agent Tests

Demo II testing of the AEA SILVER II™ 2 kW system with agent and agent simulant had the following objectives (DoD, 2001):

- Validate the ability to achieve a destruction and removal efficiency (DRE) of 99.9999 percent for HD, GB, and VX.
- Determine the impact of operations on materials of construction.
- Demonstrate the operation and performance of key process components for future scale-up.
- Develop the operational data for comparison with the 12 kW system.
- Characterize the silver-bearing residuals and determine potential silver recovery and disposal options for residuals from the silver recovery operation (HD only).
- Characterize gas, liquid, and solid process streams.

Sufficient agent or agent simulant was added to a premix vessel to complete an entire test.

Demo II testing of the SILVER II™ 12 kW system with agent simulant had the following objectives (DoD, 2001):

- Validate the ability to achieve a DRE of 99.9999 percent for agent simulants.
- Determine the impact of operations on materials of construction.
- Demonstrate the operation and performance of key process components.
- Develop operational data for comparison with the 2 kW system.
- Demonstrate the ability/inability to recycle, reuse, or dispose of nitric acid.
- Characterize gas, liquid, and solid process streams.

One agent simulant, DMMP, was tested in the 12 kW test unit (no agent was tested). For the DMMP validation, the organic feed was premixed with deionized water. This was to replace, in part, water lost from the anolyte during processing due to transfer across the membrane and consumption in oxidation reactions. Because of scheduling difficulties, the test originally planned for agent simulant CEES

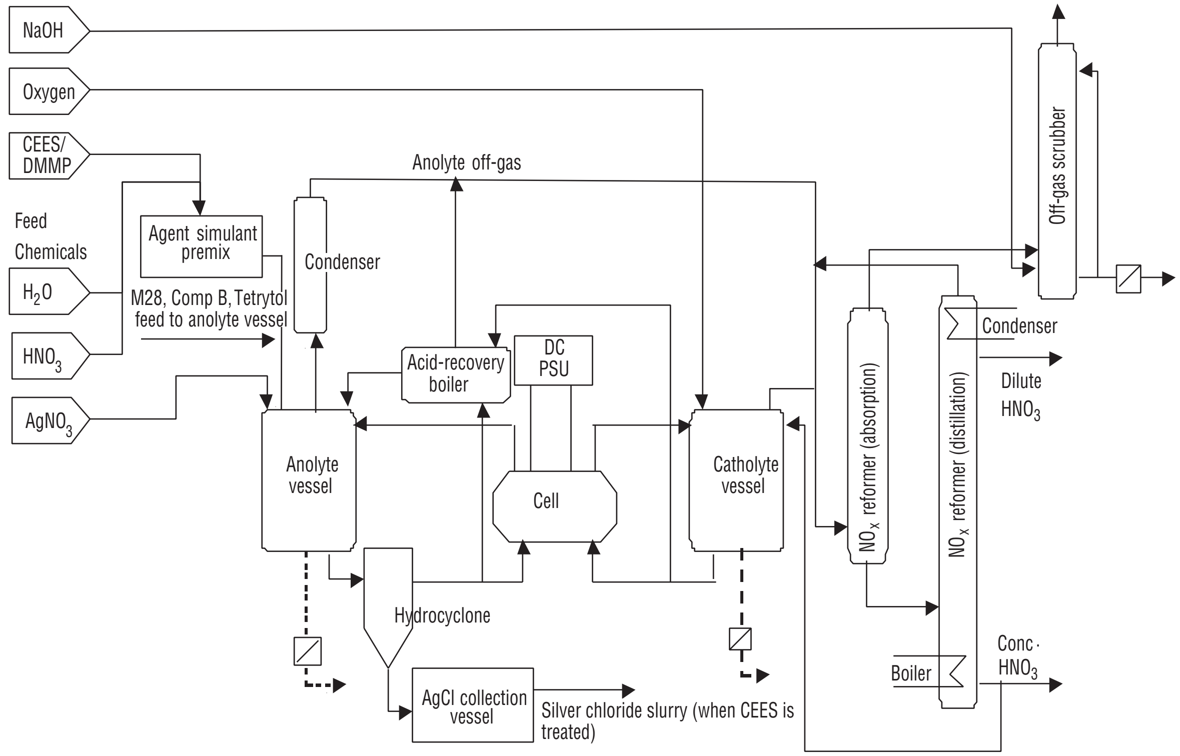

FIGURE 2-3 Process flow diagram of the AEA 12 kW demilitarization plant. SOURCE: Adapted from AEA (2000).

in the 12 kW unit was not performed. Once the DMMP had been premixed for 24 hours, it was discharged into a second vessel, from which it was continually mixed and metered into the anolyte vessel.

Energetics Tests

Demo II testing of the SILVER II™ 12 kW system with energetics had the following objectives (DoD, 2001):

- Validate the ability to achieve a DRE of 99.999 percent for tetrytol, Composition B, and M28 propellant.
- Determine the impact of operations on materials of construction.
- Demonstrate the operation and performance of key process components for future scale-up.
- Demonstrate the ability/inability to recycle, reuse, or dispose of nitric acid.
- Characterize gaseous, liquid, and solid process streams.

The tests on energetic material were conducted with premixed water slurries in the 12 kW unit. The average size of the energetics particles was 500 μm. The slurries were prepared in batches and kept well stirred in a storage vessel until they were pumped into the anolyte vessel for destruction. The feed rate was adjusted to maintain a minimum level of energetic material in the anolyte circuit at any time. The TOC in the anolyte vessel was monitored online to estimate the level of energetic in the anolyte vessel.

TEST RESULTS

The next sections summarize the results of the 2 kW and 12 kW tests and their relation to the stated objectives. These sections include discussions and comments from the committee on destruction rates, materials of construction, and gaseous, liquid, and solid process discharge. Other important committee observations germane to the operation and performance of key process components for future scale-up are also included.

Destruction and Removal Efficiency

In both the 2 kW and 12 kW systems, the overall instantaneous destruction rate was calculated from the volumetric flow rate and the measured composition of the anolyte off-gas. Analysis of the anolyte off-gas by continuous emission monitors provided the composition (volume percent, equivalent to mole percent) of the gas. The volumetric flow, corrected for ambient temperature and pressure, provided the total molar flow rate.

The DRE (in percent) for a feed of agent or energetic was defined by AEA as follows:

$$100 \times [(\text{total amount fed to plant}) - (\text{amount remaining in plant} + \text{discharges after campaign})] / [\text{total amount fed to plant}]$$

where the amounts are expressed in moles.[2]

In Appendix D of the AEA Draft Final Report (AEA, 2000), AEA gave a detailed calculation of the DRE (e.g., Table D-11 for DMMP), and it is clear that AEA calculated the discharges based on the flow rate of gas through the scrubber, the concentration observed in the scrubber off-gas, and the duration of the run. Post-test sampling of the anolyte, catholyte, and dilute and concentrated nitric acid and caustic scrubber solution was used to determine the amount remaining in the plant.

The goal for the destruction of agent simulant was 99.9999 percent; for energetics it was 99.999 percent. Determination of the DRE (as defined by AEA) depends on the detection limit of the analytical procedure for a particular agent or energetic and on the actual amount of material that was fed into the system. The total amount of agent, simulant, or energetic fed to the plant is known accurately from run records. The Environmental Protection Agency (EPA) reference test methods were used to determine concentrations of organics in solutions.

The 2 kW tests successfully demonstrated the destruction of HD, GB, and VX, as well as the destruction of simulant DMMP to 99.9999 percent DRE or greater. The amount of material treated, steady-state current efficiency, and DREs are shown in Table 2-1.

TABLE 2-1 Destruction Efficiency in the 2 kW Test Unit

Agent or Simulant	Amount Treated (kg)	Current Efficiency (%)	Destruction Efficiency (%)
DMMP	10	40–50	99.9998957
VX	4	70–90	99.9999886
HD	16	40–60	99.9999914
GB	15.7	60–80	99.999996

SOURCE: AEA (2000).

Tests originally planned for the simulant CEES were not conducted because of schedule constraints (the other demonstration tests took longer than had been expected, as discussed in more detail below).

In the 12 kW facility, 40 kg of the simulant DMMP was destroyed with a DRE similar to that in the 2 kW unit (99.99997 percent) and the same electrochemical efficiency (40 to 50 percent). Thus, AEA concludes that the two systems appear to be operating in a similar fashion.

All tests on energetics were conducted in the 12 kW plant. The Comp B was not tested owing to schedule constraints. Tests on M28 propellant successfully demonstrated high DREs of nitrocellulose (99.9999 percent) and nitroglycerine (99.99999 percent) and very high electrochemical efficiency (80 to 100 percent). More than 159 kg of M28 were treated in 8 days of operation.

In the tests conducted with tetrytol, solid material built up on the walls of the anolyte and catholyte circuits and forced operation at slower feed rates than anticipated. The difficulties encountered in processing tetrytol were attributed to the formation of recalcitrant intermediate products that either crystallize in the anolyte circuit or migrate through the membrane to the catholyte circuit before crystallizing. The accumulation of these solids, which obstructed filters and sample lines and accumulated in tanks, valves, and piping, forced periodic change-outs of the solutions in both circuits. This slowed the feed rate of tetrytol. Very low electrochemical efficiencies (20 to 30 percent) were measured for tetrytol due to the small amount of organic material that could be tolerated in the anolyte vessel. When the concentrations of organics in the anolyte solution were lowered, their level of oxidation was reduced. The measured DREs of the tetrytol components were relatively low—for example, 99.7 percent for TNT and 99.8 percent for tetryl. Though the tetrytol runs were scheduled to last 7 days, they actually lasted 18 days, because the feed rate had to be lowered, and only 73 kg of tetrytol was processed. AEA has suggested several changes to the plant design to reduce the accumulation of this intermediate product. These suggestions are discussed later in this chapter.

Preliminary results from gas chromatography/mass spectrometry and high-performance liquid chromatography analysis of a solid intermediate recovered from the tetrytol testing identified several energetic compounds, including trinitrobenzoic acid (TNBA) and trinitrobenzene (TNB) (Winkler, 2001). Also, picric acid (PA) was identified in the solid intermediate (personal communication between John Coffey, senior environmental chemist, CH2M Hill, Inc., and Darren Dalton, PMACWA, February 21, 2001).

Currently, AEA considers the indicators of complete reaction to be the absence of TNT and tetryl (compounds that are present in the original feed) and a low value of TOC in the anolyte solution. However, the presence of TNBA, PA, and TNB crystals in both the anolyte and catholyte circuits implies that the absence of TNT and tetryl is in and of itself not a valid indicator of the total destruction of all energetic compounds in the system. Also, the measurement of TOC in the anolyte solution is not equivalent to the measurement of total organics in the system, because there is no accounting for the solids that precipitate out or otherwise accu-

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[2] As noted, this definition does not agree with the standard regulatory definition understood by the committee. The regulatory definition of DRE is based on the measured feed rate of a constituent and its emission rate in the gas-phase effluent only.

mulate in the system. The explosive hazard should not be assumed to be eliminated as the reaction proceeds, because TNBA or other energetic compounds may still remain.

Only a small amount of PA was observed in the solids formed during the tetrytol testing (O'Neil, 2001). However, if a mixed feed stream containing an aromatic nitro compound (Composition B and tetryl or tetrytol) is processed with a propellant that contains lead, lead picrate (a very sensitive primary explosive) can be formed. During the M28 propellant tests, lead dioxide precipitated on the cell membrane, so it is possible that the precipitation of lead with the small amount of picric acid that might be present does not compete with the formation of lead dioxide in the electrochemical cell. However, the possibility of lead picrate precipitation cannot be dismissed a priori. A determination of the relative solubilities of lead dioxide and lead picrate in nitric acid solutions could provide some insight into whether the formation of lead picrate is likely to be a problem for mixed feeds.

Materials of Construction

Even given the relatively short duration of the Demo II tests, it was apparent that corrosion is a serious problem. Notable problems encountered during the Demo II tests are listed below:

- anolyte pump failures in the 2 kW tests
- leakages in the glass-to-glass joints in the 2 kW facility
- failure of the glass nonreturn valves in the 2 kW tests
- damage to glass during the processing of GB in the 2 kW test
- failure of glass components and joints in the12 kW unit
- cell gasket failure in the 12 kW unit

Materials of construction must be carefully selected to avoid leaks and failures that could interfere with full-scale operation.

The results of coupon tests updated by AEA (Table 2-2) demonstrate that serious issues remain concerning the selection of materials of construction. In these tests, coupons of different materials were exposed in the anolyte chamber during the Demo II program and were reweighed and photographed after exposure to the SILVER II™ environment for all of the runs. The approximate exposure times were just over 34 days. All materials tested showed significant weight losses during exposure to these environments. Corrosion was probably caused by the formation of hydrofluoric acid (HF) when treating GB. Even polytetrafluoroethylene (PTFE)-coated stainless steel showed high rates of corrosion. However, the technology provider has indicated that PTFE-lined stainless steel is still a viable alternative based on previous tests results and industrial experience. The technology provider attributed the coupon failure to micropores in the PTFE coatings, which it said should not be present in the PTFE-lined piping.

Characteristics of Gaseous, Liquid, and Solid Process Streams

All mass balances were obtained using the volumes and composition of input reactants and output products. Routine on- and off-line samplings were used to determine the concentrations of the intermediate species in the process streams. The inventory of materials in the system was estimated from the volume of material in each vessel. All streams leaving the process were analyzed and this, in conjunction with volume measurements, gave the total inventory of the species for the mass balance.

The organic feed material was well characterized and quantified, but the organic intermediates in the process were diverse and not quantified individually. However, the anolyte, catholyte, and reformer liquors were analyzed regularly to determine their TOC content. This TOC value, along with volume readings, yielded the inventory of dissolved organics in the plant. The organic feed rate; TOC determinations; and off-gas analyses for CO, CO_2, and volatile organic compounds (VOCs) were used to derive a continuous mass

TABLE 2-2 Anolyte Coupon Weights Before and After Testing

Coupon	Starting Weight (g)	Final Weight (g)	Change in Weight (g)	Change in Weight (%)
PTFE/304L #2	22.074	19.306	2.768	12.54
PTFE/304L #1	22.118	19.275	2.843	12.85
Titanium #4	20.086	16.343	3.743	18.64
Titanium #3	20.290	16.197	4.093	20.17
Glass #2	22.861	16.475	6.386	27.94
Glass #1	23.538	15.532	8.006	34.01
Zirconium #5	18.554	2.317	16.237	87.51
Zirconium #4	18.617	2.268	16.349	87.82

SOURCE: AEA (2000).

balance for carbon. Organic sulfur and phosphorus were determined from analysis of the sulfate and phosphate salts formed in the anolyte circuit after oxidation. All organic chlorine was assumed to be converted to AgCl at the end of a campaign. Any silver remaining in solution was neglected, because the solubility of silver chloride is less than 0.001M.

The following gaseous effluents were analyzed:

- the anolyte gases for CO and SO_2 (sulfur dioxide) by CEM; VOCs by EPA TO15; semivolatile organic compounds (SVOCs) by EPA 0010; agents by the depot area air monitoring system (DAAMS); and Schedule 2 decomposition compounds by EPA TO14
- the pre-reformer off-gas for O_2 and NO_x by CEM
- the post-reformer off-gas for O_2 and NO_x by CEM
- the discharged off-gas for CO_2, O_2, CO, N_2 (nitrogen), N_2O (nitrous oxide), H_2, SO_x, and NO_x by CEM and gas chromatography; VOCs by EPA TO15; SVOCs by EPA 0010; agents by DAAMS; and Schedule 2 decomposition compounds by EPA TO14

The Demo II tests revealed the presence of VOCs in the off-gas stream. AEA has indicated that the design will be changed to include a catalytic oxidation (CATOX) unit on the off-gas vents to control the emissions of these organics. In the EDS phase of the program it is important to evaluate the performance of the proposed CATOX unit with particular focus on how impurities such as phosphorus and fluorine affect the catalyst's oxidative reactivity.

The liquid discharges from the anolyte circuit, catholyte circuit, NO_x reformer, and caustic scrubber were sampled and analyzed for metals and organics. At the time of this report, not all of the data were available to the committee. The tests apparently validated that this technology did not generate Schedule 1 compounds or significant quantities of Schedule 2 compounds regulated under the Chemical Weapons Convention (CWC). The Army has concluded that the characterization of the products of agent and propellant destruction showed that acceptable treatment of most hazardous intermediates (to relatively low levels) was achieved and validated for this process. Additional treatment steps that should effectively destroy the remaining hazardous intermediates were proposed but not demonstrated.

AEA's effluent management system proposes to send the dilute nitric acid waste streams to a publicly owned treatment works (POTW) under a pretreatment exemption. Although the availability of a POTW has not been confirmed, the Demo II tests indicated that the concentrations of hazardous material in the liquid streams were sufficiently low to qualify for treatment at such a facility.

Solids from the scrubber filter and from the anolyte bag filter were analyzed at the end of each run. They were analyzed for agents, agent simulants, Schedule 2 compounds, other decomposition products, metals, SVOCs, VOCs, TOC, sulfates and phosphates, dioxins, and furans. Hazardous properties of the solids (ignitability, corrosivity, reactivity) were also examined. At the time the committee completed its evaluation for this report, the solid product characterization had not been completed.

Electrochemical Efficiency

AEA defines electrochemical efficiency as 100 times the ratio of current utilized to oxidize organic compounds in the feed to the sum of this current and the current used to electrolyze water. The current utilized to oxidize organic compounds is inferred from the amounts of CO and CO_2 produced; the current used for the electrolysis of water is inferred from the amount of oxygen produced. Thus,

$$\text{Electrochemical efficiency} = 100 \times [I_{CO} + I_{CO_2}] / [I_{CO} + I_{CO_2} + I_{O_2}].$$

I_{CO}, I_{CO_2}, and I_{O_2} are the currents (in amperes) used to form CO, CO_2, and O_2, respectively. This provides a measure by which to determine the relative amount of the current used to destroy the agent or the energetic versus the amount used in parasitic reactions.

The currents are not measured directly but are determined from the relative molar amounts of CO, CO_2, and O_2 measured in the off-gas. Using HD as an example, equations for the anodic reactions producing CO and CO_2 are:

$$C_4H_8Cl_2S + 8H_2O \rightarrow 4CO + 24H^+ + 2Cl^- + SO_4^{-2} + 20e^-$$
$$C_4H_8Cl_2S + 12H_2O \rightarrow 4CO_2 + 32H^+ + 2Cl^- + SO_4^{-2} + 28e^-$$

Thus, each mole of CO produced requires (20/4) faradays (F), or 5F, of charge to flow, while each mole of CO_2 requires (28/4)F, or 7F. The electrochemical inefficiency is related to the fraction of the current that results in the production of O_2:

$$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$$

Each mole of O_2 thus requires 4F of charge to pass.

The currents required to calculate the efficiency are therefore determined from measurements of the off-gas composition and solution of the following equations:

$$\begin{aligned} I_{CO} &= [(\text{moles/min CO}) \times 96{,}500 \times 5]\ \text{A}, \textit{ or} \\ &\quad [\text{volume percent of CO}] \times 5 \\ I_{CO_2} &= [\text{moles/min } CO_2) \times 96{,}500 \times 7]\ \text{A}, \textit{ or} \\ &\quad [\text{volume percent of } CO_2] \times 7 \\ I_{O_2} &= [(\text{moles/min } O_2) \times 96{,}500 \times 4]\ \text{A}, \textit{ or} \\ &\quad [\text{volume percent of } O_2] \times 4 \end{aligned}$$

The committee believes that this definition of electrochemical efficiency cannot be taken as an indicator of the amount of energy that will be required to run the plant. Nonelectrochemical oxidations, such as direct oxidation by nitric acid rather than Ag^{2+}, may contribute to the destruction of the feed, thus reducing the need for electrochemically driven reactions and consequently the energy requirements. On the other hand, other dissipative processes, such as resistive heating and electrochemical oxidation of impurities in the system, will increase the required energy. The only utility of the electrochemical efficiency as defined by AEA is to reflect the competition between oxidation of the feed and parasitic reactions.

AEA DESIGN CHANGES BASED ON TEST RESULTS

Major changes have been proposed to the process flow diagram based on the Demo II test results. Figure 2-4 shows the revised AEA process flow diagram. Some of the changes are listed below:

- addition of a CATOX unit to control VOCs in off-gases
- use of hydrocyclones in both the anolyte and catholyte circuits
- addition of a return flow of material (both solids and liquids) from the catholyte to anolyte to return organics that have migrated across the cell membrane
- addition of a high-shear vortex mixer in the anolyte circuit to achieve better mixing of the tetrytol to promote the oxidation of intermediate products that precipitate
- addition of chemical cleaning processes for electrodes to remove the buildup of lead and AgCl
- addition of an energetic rotary deactivator for the destruction of separated fuzes and supplemental charges

AEA proposes adding an impurities removal system (IRS) as a bleed stream on the anolyte circuit. The IRS would withdraw anolyte solution to control concentrations of impurities such as phosphates and sulfates and to remove solid precipitates (i.e., AgCl). In the IRS, liquids and gases would be separated from solids. The liquids and gases would be recycled back to the SILVER II™ anolyte circuits, and the solids would constitute a solid waste stream. The IRS includes five operations: (1) removal of solids, (2) fractional distillation of the solution, (3) separation of lower portions of the distillation column containing nitric acid enriched with phosphoric acid and sulfuric acid, (4) release of off-gases through a CATOX unit, and (5) recirculation of the condensate to the catholyte unit. The process flow diagram for the IRS was being modified when this report was written. Because the IRS is so complicated, further refinements and development will be necessary prior to its full-scale deployment.

The sizing of the IRS and the frequency of bleeds will be determined by the expected impurities in the feeds. AEA considers the lead in the burn rate modifiers in propellants to be an impurity that must be treated by the IRS. It has indicated that most of the lead oxide produced from lead in the propellants will be spalled to produce a suspension that will also need to be removed in the IRS. A smaller amount of lead precipitates out as lead oxide on the cell membrane, which keeps it from reaching the IRS. Fluoride, sulfate, and phosphate are expected to be present from agents but not energetics.

One of AEA's main concerns to be addressed by the IRS is the presence of small pieces of metal in the energetics originating from resonance rods, which in turn come from the rocket propellant removal operation. The resonance rods sometimes become dislodged when the propellant is being removed, and unless they are removed, they will be ground up with the propellant. These metal grindings will be present in the slurry that is fed into the anolyte vessel. Even if the grindings do not short out the cell electrodes, a metallic conductor placed between two electrodes from which it is electrically isolated (no electronic connectivity) will itself become a bipolar electrode—the surface of the metal facing the anode will become cathodic and the surface of the metal facing the cathode will become anodic. In the extreme, the potentials can be strong enough to drive faradaic processes. If this happens, productivity could be lost as a result of parasitic side reactions and reduced voltage on the principal electrodes themselves.

At the time of the committee's review, the IRS system was not well developed. The IRS system AEA proposes for agent is different from the system for energetics, and the design of the full-scale system is expected to be much different from the one that has already been tested. Because the IRS was not tested in Demo II, no other data were available on the characterization of solids. AEA plans to put the solids in containers and send them to a RCRA-approved disposal facility.

REEVALUATION OF STEPS REQUIRED FOR IMPLEMENTATION

The ACW I committee identified several key steps that would have to be implemented before the AEA SILVER II™ technology could be fully implemented (NRC, 1999). These steps are reevaluated on the basis of the results of the Demo II tests.

> 1. Modified shearing locations for M55 rockets and a new shearing machine must be tested to show routine segregation of components and reduction in particle sizes to less than 1 inch in diameter.

This still needs to be evaluated.

> 2. The modified mine shearing approach must be tested.

This is not applicable for the Blue Grass Stockpile, which has no land mines.

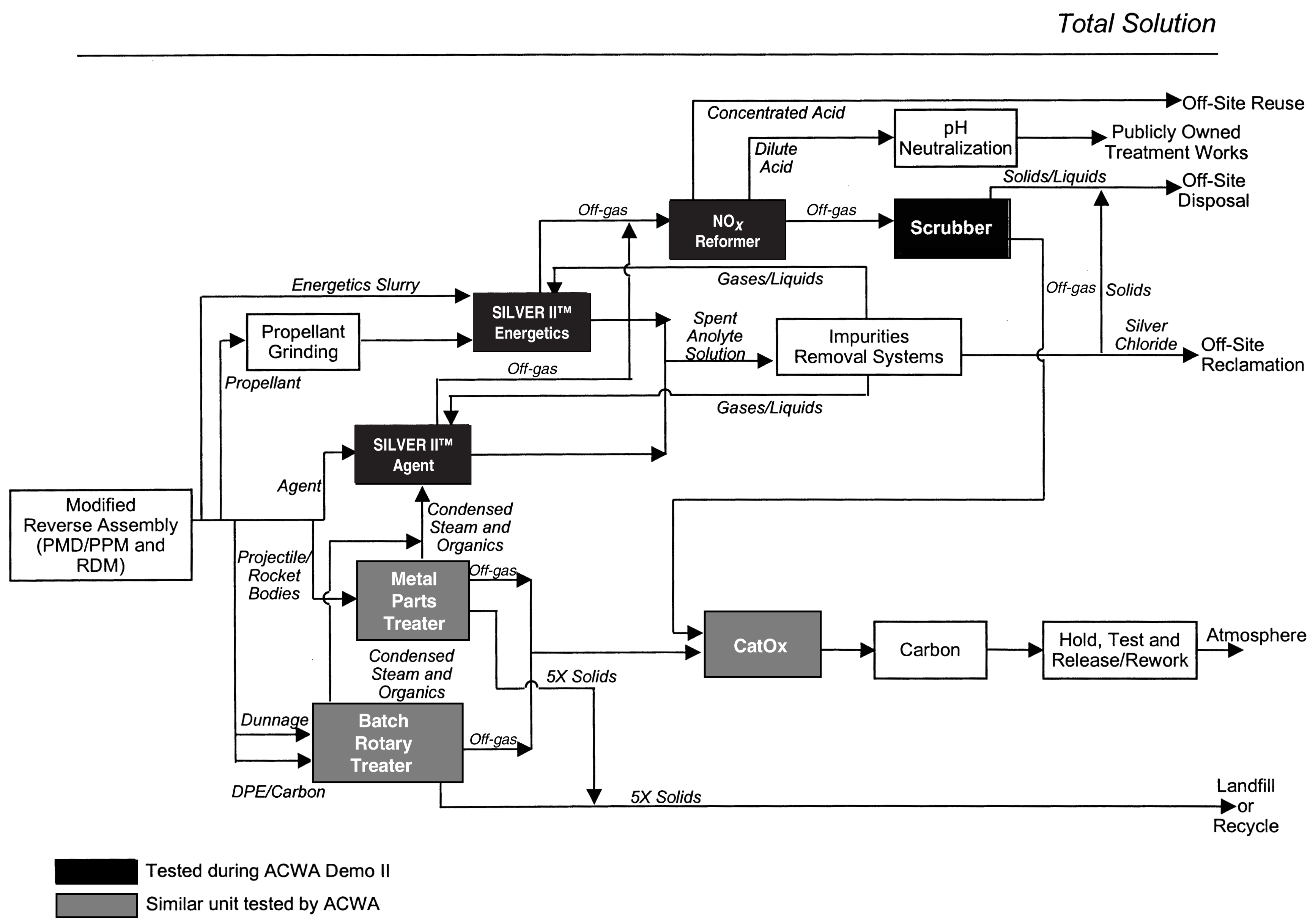

FIGURE 2-4 Revised process flow diagram of the AEA SILVER II™ demilitarization process. SOURCE: CH2MHILL (2001).

> 3. The dissolution of fuzes and mine bodies in nitric acid and SILVER II™ solution must be evaluated.

This is no longer relevant, because an energetic rotary deactivation unit has been added to the design.

> 4. All effluents must be characterized in detail when treating agents contaminated with metals from disassembled chemical weapons (i.e., potential trace species and reaction by-products, such as nitrated hydrocarbons, partially oxidized products, and metals, must be identified) and their environmental impacts evaluated.

The Demo II test evaluated all of the major effluent streams for a full suite of trace species and reaction by-products. Intermediate reaction products were found in tests with tetrytol, as described above. However, at the time this report was being prepared, not all of the data were available to the committee.

> 5. Demonstrations of the scale-up, development, and integration of hardware with real materials of construction must focus on the robustness of the parallel flow in multiple-cell reactors. The issues of cell blockage, hydrocyclone performance, and NO_x reformer performance must be addressed.

The ACW II committee repeats this recommendation and stresses its importance in light of the increased complexity of the process once changes have been made to address problems revealed in the Demo II tests, which were discussed in the preceding section.

> 6. The efficacy of high pressure-jet wash-out of agent and gelled agent from M55 sheared pieces must be tested.

This recommendation was not addressed in the Demo II tests, so it remains valid.

> 7. The treatment of burster charges and M28 propellant in the SILVER II™ reactor must be tested, and the material preparation required to ensure reasonable treatment times with no energetic events must be evaluated. This testing must also determine what happens to the lead stearate in the propellant during SILVER II™ treatment.

The tests with the 12 kW system in the Demo II confirmed that this technology is capable of destroying the components (nitrocellulose and nitroglycerine) of M28 propellant. For tetrytol, the destruction of TNT and tetryl was good. However, recalcitrant intermediate products were formed during the treatment of tetrytol; AEA was still evaluating them as this report was being written. Given the preliminary indication that the intermediates were energetics, the finding of the need to demonstrate that no energetic events can occur remains valid. The committee is also not aware of any new data on the fate of lead stearate from these Demo II tests.

> 8. The process must be developed and tested for the efficacy of submerged-bath dilute nitric acid treatment for metals parts, including the effects of agitation and temperature.

This recommendation is no longer relevant, because the current design uses a metal parts steam treater; the condensed steam and organics produced by this unit are introduced into the agent SILVER II™ circuit. Off-gas is directed to a CATOX unit.

> 9. The treatment of shredded dunnage material must be tested in a prototype-scale SILVER II™ reactor.

This is no longer relevant because the dunnage and demilitarization protective ensemble (DPE)/carbon will be treated in a batch rotary treater. The resulting condensed steam and organics are sent to the SILVER II™ agent circuit, and the off-gas from the batch rotary dunnage treater is passed through a CATOX unit.

> 10. Techniques for controlling particulate matter to prevent plugging of SILVER II™ electrolytic cell channels must be developed and demonstrated.

The committee's concern at the time of this recommendation was plugging in the cell channels due to formation of AgCl particles. Although no significant plugging problems were encountered in Demo II in the cell channels themselves due to AgCl particles, there was evidence of other types of particulate buildup in other components. For example, lead oxide precipitated in the cells. A wide range of unanticipated particulate and material blockages was encounered in the vessels and piping in the anolyte circuit. There was also a buildup of particulates in the catholyte circuit, caused by the migration of organics across the membrane. The potential for plugging as the units are scaled up remains a concern for the committee.

> 11. Materials of construction must be evaluated under corrosive and oxidizing conditions.

Severe corrosion was found in several materials, particularly those used to contain fluorine-bearing streams. This recommendation remains valid.

> 12. The realistic potential for off-site recycling/reuse of silver salts and concentrated nitric acid must be evaluated, including recyclers' ability to accept, handle, and treat these materials.

This issue was addressed in the Demo II tests through an analysis of SILVER II™ concentrated acid by Ensign Bickford Company. Additional confirmation of the capabilities of commercial facilities should be solicited.

REVIEW OF THE ACW I COMMITTEE'S FINDINGS

It may be worth noting that many of the same problems were identified in the NRC study *Review and Evaluation of Alternative Chemical Disposal Technologies* (NRC, 1996) and the ACW I report (NRC, 1999). The ACW I committee's earlier findings are updated below:

> **Finding AEA-1.** Significant barriers to the development of the sophisticated equipment and processes for segregation, materials handling, and size reduction (to reduce materials to less than 1 inch in diameter) must be overcome.

These barriers remain a concern to the ACW II committee. The tests with energetics in the 12 kW unit were done with material reduced to less than 500 μm (AEA, 2000) and then premixed into slurries. The full-scale design calls for one-eighth-inch (~3,000-μm) pieces in slurries. The equipment and process for segregating, handling, and size-reducing to this target size will have to be defined and evaluated for the full-scale equipment.

Propellant grinding has not been successfully demonstrated. Another concern is that increasing the particle size from 500 μm to 3,000 μm could substantially increase the processing time, decrease electrochemical efficiency, and increase the formation of by-products and associated blockage.

The description for AEA's energetic process is as follows (AEA, 2000).

> Using the Army's existing Projectile/Mortar Demil Machine (PMD), 155mm and 8-inch projectiles will be disassembled, but we propose to replace the existing Multipurpose Demil Machine (MDM) with a new punch, drain, and washout machine. Projectiles will be received in the unpack area and loaded into the existing feed equipment for transportation into the explosive containment room. There will be two identical disassembly equipment lines. For rockets, the PMD will remove the nose closure, burster, fuze, supplemental charge and miscellaneous parts. Fuzes and supplemental charges will be conveyed to the detonation chamber for deactivation. The bursters will be extracted and conveyed to a stand-alone burster washout machine to fluid jet out the burster, using conventional fluid jet technology. This will result in an energetic slurry with a nominal maximum particle size of one-eighth inch and a slurry concentration not to exceed 20 percent by weight. The Rocket Demilitarization Machine directs the rocket motor section to the propellant processing station. The propellant is mechanically extracted from the motor section. The propellant grain is then deposited into the energetics grinder for size reduction. The grinder design will be optimized during EDS testing. Blade configuration and speed will be selected to process the propellant, which has been described as having a consistency of "tire rubber." The grinding operation will be done "wet" to reduce the possibility of fire and aims to produce a one-eighth inch (maximum) particle size.

Wet cutting and grinding/shredding have been used safely by industry. However, there is always a possibility that metal shards or large chunks of propellant will become wedged in the cutting or shredding equipment and ignite. The final design is expected to address this possibility, and the cutting area will be designed to withstand propellant ignition.

> **Finding AEA-2.** Potential problems associated with plugging of the passages in the electrolytic cells will have to be addressed.

No blockages of the electrolytic cell were reported during normal operation in either the 2 kW or the 12 kW unit during Demo II. However, there were buildups of silver chloride particles and blockage in other areas: (1) the carryover from the 5X treatment unit and sample lines in the 2 kW unit and (2) failure of the cell gasket in the 12 kW unit. For this reason, the ACW II committee remains concerned that cell blockages are likely to occur with the equipment scale-up.

Other blockages occurred outside the electrochemical cells during the treatment of tetrytol. As was discussed above, these were apparently caused by the formation and precipitation of intermediate decomposition products with low solubility. The blockages were severe enough in the 12 kW unit to prevent completion of the demonstration tests for tetrytol. In addition, there was deposition of solids and blockage in the catholyte as a result of the movement of intermediate products across the cell membrane. AEA has proposed a number of changes in the process design to prevent this: (1) the addition of a hydrocyclone in the catholyte circuit and anolyte circuits, (2) the addition of a high-shear vortex mixer in the anolyte and catholyte circuits, (3) the addition of an IRS using a bleed from the anolyte, and (4) provisions for a return flow from the catholyte to the anolyte circuit. The ACW II committee reiterates that buildup of solid materials in the circuits and the potential for blockages is an area of high technical risk that will require continued attention during scale-up and engineering design.

The committee further notes that the formation of intermediate decomposition products was not anticipated. Until the unit is operated successfully with all possible feeds, this will remain another area of high technical risk. Furthermore, some of the feeds are expected to vary in composition, which will complicate the challenge to system operability.

AEA has determined that TNBA did form in the anolyte vessel and that solids formed on the membrane and sides of the anolyte vessel during the treatment of tetrytol. Some of the TNBA crystals were 0.25 inch across. AEA said that when the particles reached a certain size, they broke away from the walls and fell to the bottom of the anolyte vessel. In its EDS, AEA proposes using a high-shear mixer in the anolyte vessel to break up these crystals. The mixer would break up other particles and is expected to reduce most of the solids to less than 100 μm before they enter the cell where the Ag^{2+} is formed and destruction takes place. AEA does not plan to do a mass balance but will determine when the destruction is complete by measuring the amount of starting material left in the anolyte vessel. Although this will indicate when the tetryl is destroyed, it will not indicate when all of the intermediate energetics has been destroyed.

Finding AEA-3. The proposed chemical dissolution of fuzes is a complete unknown because no data on this process are available. This approach is complex in comparison to more conventional techniques of separation and detonation.

The dissolution of fuzes was not tested in the Demo II test program. The preliminary design now calls for the separation of fuzes followed by destruction in an energetics rotary deactivation unit, a common approach for destroying fuzes. Chemical dissolution of fuzes is no longer part of the design.

Finding AEA-4. Data are not available to assess the efficacy of the treatment of energetics at larger scales.

The 12 kW pilot-scale tests conducted during the Demo II tests specifically tested for the DRE of components in M28 propellant and tetrytol. These tests provided evidence that high DREs (99.9999 percent) could be achieved for components of M28 propellant (nitrocellulose and nitroglycerine). However, lower DREs (~99.7 percent) were achieved with the components of tetrytol.

Processing time for the destruction of energetics, particularly M28 propellants, in the SILVER II™ unit at full scale will certainly increase as a result of larger particles (3,000 μm). In addition, processing times for all energetics will increase as a result of slower mixing in the anolyte vessel, the need to treat intermediate by-products, and the impacts of recycling untreated material from the catholyte. The committee reiterates its concerns about the impact of scale-up on DREs for energetics.

Finding AEA-5. The ability to obtain a representative sample of gaseous effluents retained in the holding tanks prior to release has not been demonstrated.

The hold, sample, and release option was not included in the Demo II tests. All gas analyses were performed on samples taken from the flowing off-gases over 2-hour periods. Techniques for sampling and ensuring that no agents are present prior to release will have to be developed and confirmed in subsequent programs.

Finding AEA-6. Several issues need to be addressed during the scale-up of the process into a fully integrated system, including temperature control, reaction times, efficiency of the NO_x reformer, cell flow management, efficiency of the hydrocyclone, and the tolerance of cells to particulate matter. These issues are potentially serious enough to create processing problems in larger scale systems.

The 2 kW and 12 kW demonstration units were successfully operated, showing that small-scale units could operate properly with adequate process control. However, critical gaps in knowledge remain with respect to performance of the scale-up process. Several process features are expected to become more difficult to control and manage as the scale is increased from 12 kW to more than 300 kW in a full-scale system. The most pressing issues are the following:

- longer reaction times for larger particles
- mixing limitations in larger anolyte vessels
- less effective performance of larger hydrocyclones
- formation of additional soluble by-products
- need for recirculation of catholyte solution to the anolyte vessel to control organics passing through the cell membrane
- control issues associated with a fully integrated pilot plant

Although none of these issues is likely to preclude the use of this technology, resolving the uncertainties will require comprehensive, time-consuming, integrated, pilot-scale development and systemization.

Finding AEA-7. Identifying which corrosion-resistant materials would be compatible with HNO_3 and HNO_3/HF will require a significant development program.

The committee reiterates and reinforces the initial finding that corrosion from fluorine-containing agents—GB, for instance—will be a serious problem in the full-scale operation of the SILVER II™ process. AEA has recognized this issue and is considering other materials of construction and exploring techniques for removing fluorine from agents prior to their introduction to the anolyte circuit. The committee believes that further assessments will be necessary to determine the viability of these techniques and notes that a fluorine removal step further complicates the process.

Finding AEA-8. Limiting or controlling reactions between nitric acid and agent and energetics must be demonstrated.

The Demo II results show that reactions between nitric acid and agent and energetics can be safely controlled.

Finding AEA-9. Conversion of excess nitric acid into ammonium nitrate fertilizer may be complicated because of the potential for contamination and the liabilities this would entail.

Conversion of nitric acid was not tested or evaluated in this study. However, the excess nitric acid was analyzed and found to be free of organics, agents, Schedule 2 compounds, and decomposition products. Therefore, conversion of nitric acid to ammonium nitrate, a commercially viable process, appears to be feasible.

Finding AEA-10. A large inventory of silver is required for processing of chemical weapons, and finding an off-site recycler to accept the potentially contaminated materials could be a problem.

AEA provided a letter from an outside vendor indicating that the dried silver chloride product is suitable for recycling. However, the silver chloride that was acceptable to the vendor was produced from a well-controlled feed material. Impurities in the feed from assembled chemical weapons may be present in the silver chloride produced during their destruction and may affect the acceptability of this product to off-site recyclers.

SUPPLEMENTAL FINDINGS

Finding DII AEA-1. The overall process flow has been further complicated by major design changes in response to the Demo II testing. These changes include the addition of the IRS, CATOX units, and a flow return circuit from the catholyte to the anolyte circuit. All three changes require small-scale and pilot-scale testing. Such modifications further complicate the interfaces between process units, which increases the time required for development, start-up, and commissioning of the full-scale system. Integration of the operating units will make achievement of a viable total solution very difficult.

Finding DII AEA-2. The discovery of organic material migration across the electrochemical cell membrane will require major modifications in design and operation, such as recycling of the catholyte material to the anolyte circuit and the addition of hydrocyclones in the catholyte circuit.

Finding DII AEA-3. The formation of intermediate oxidation by-products raises operational issues, including slower processing rates and reduced electrochemical efficiency. During the testing with tetrytol in the 12 kW unit, the problems were severe enough to cause the runs to be extended well beyond the planned processing times.

Finding DII AEA-4. The generation of new energetic compounds (TNBA, PA, TNB) in the course of processing increases the complexity and hazards of the SILVER II™ process. Although the explosion hazard is reduced as the energetic feed is consumed, it is not completely eliminated until all energetic intermediates are destroyed.

Finding DII AEA-5. During the treatment of M28 in the Demo II test, lead oxide and other materials accumulated on cell anodes. The committee believes that a maintenance procedure for routine cleaning of the anodes will be required.

Finding DII AEA-6. Low steady-state electrochemical efficiencies (20 to 30 percent) were observed during treatment of tetrytol. These low efficiencies will decrease the throughput per cell and increase processing time and energy consumption.

Finding DII AEA-7. VOCs were detected in the off-gas of the AEA process technology. AEA has now included a CATOX unit in the preliminary design. The committee believes that the introduction of this additional unit operation will further complicate the scale-up and integration.

Finding DII AEA-8. The IRS for removing salts (sulfates, phosphates, silver fluoride), excess water, and any metals that may be present requires extensive development and integration. The IRS has not yet been described in sufficient detail to allow for a meaningful assessment.

SUPPLEMENTAL RECOMMENDATIONS

Recommendation DII AEA-1. The possible formation of lead picrate when mixed energetic feeds are treated must be investigated before any processing of lead-containing propellant, TNT-based energetics, or tetryl is undertaken.

Recommendation DII AEA-2. The IRS, the CATOX units, the return flow, and all other major modifications to the system must be tested and proven during the EDS design phase.

Recommendation DII AEA-3. AEA must validate complete destruction of all energetic intermediates during the EDS design phase.

Recommendation DII AEA-4. AEA must conduct additional tests to identify suitable materials of construction to overcome corrosion problems encountered owing to the formation of HF in the treatment of GB.

3

Foster Wheeler/Eco Logic/Kvaerner Integrated Demilitarization Process

The Foster Wheeler/Eco Logic/Kvaerner (FW/EL/K) system is based on the Lockheed Martin Integrated Demilitarization System (LMIDS). The technology development is continuing with Kvaerner John Brown as the project integrator. Foster Wheeler Corporation is overseeing the supercritical water oxidation (SCWO) unit, which uses the transpiring-wall (TW) technology developed by the Aerojet General Corporation. Eli Eco Logic International (commonly referred to as Eco Logic) is in charge of the gas-phase chemical reduction (GPCR™) unit. In the FW/EL/K system, the chemical agent, energetic materials, and metal parts are separated by a modified version of the Army's baseline disassembly process. Chemical agent, energetic materials, and agent remaining on metal parts and dunnage are decomposed in separate caustic hydrolysis units. The hydrolysates are then further treated in a TW-SCWO unit. At the same time, metal parts and dunnage are decontaminated to a 5X level and gaseous effluents from the hydrolysis processes are treated by GPCR™. A schematic diagram of the process is shown in Figure 3-1.

Based on the results of Demo II tests for the SCWO unit and the GPCR™ unit and concerns of the PMACWA and the ACW I committee, changes were made to the integrated full-scale system described in the earlier report (NRC, 1999). The main changes are described below.

The immersed conveyor system for moving solids in baskets through the hydrolysis reactor has been removed from the caustic bath and is now replaced by an overhead chain conveyor system. This move is expected to reduce the maintenance for the conveyor system, which requires the presence of workers in demilitarization protective ensemble (DPE) suits. The new design has not been tested.

A number of changes have been made to the reverse-assembly/munitions-access process originally proposed in the LMIDS. The changes for M55 rockets include (1) using an expansion collet instead of hydromining for the removal of M28 propellant, (2) grinding the propellant to granules of less than 0.25-inch-diameter under a deluge of water solution at ambient temperature, and (3) hydrolyzing the propellant slurry separately to avoid the possible formation of lead picrate, which is a very sensitive explosive when it is dry. The changes in handling projectiles include (1) eliminating the burster well pull-and-place station, (2) replacing the burster well boring station with a projectile punch machine for accessing the agent cavity, and (3) replacing the projectile wash bay with a less complex wash operation.

No major changes have been made to the SCWO system, but there are some differences between the SCWO unit previously tested and the SCWO units proposed for EDS and for full-scale operation. Changes have also been made to equipment downstream of the SCWO reactor to facilitate processing of the suspended solids in the reactor effluent, especially for aluminum-rich feeds. The effluent flows from the pressure letdown valves to a knockout drum that contains a venturi scrubber, which separates liquid and suspended solids from the uncondensed vapor. The slurry is pumped to an evaporator/crystallizer system that replaces the flash separator in the original design.

Two changes are planned for the GPCR™. First, a step will be added to remove the toxic phosphine gas formed in the processing of GB. Second, hydrogen sulfide formed in the processing of mustard and VX will be removed. The removal system will be designed and included in the engineering design package for the full-scale plant. This will require one or both of the following modifications: (1) minor enhancements to the projectile trains in Area 100 to maximize the removal of mustard from the projectiles and (2) optimizing conditions for removal or adsorption/absorption of hydrogen sulfide in the existing Area 400 scrubber.

TRANSPIRING-WALL SUPERCRITICAL WATER OXIDATION UNIT

The following objectives were established for the Demo II TW-SCWO tests (DoD, 2001):

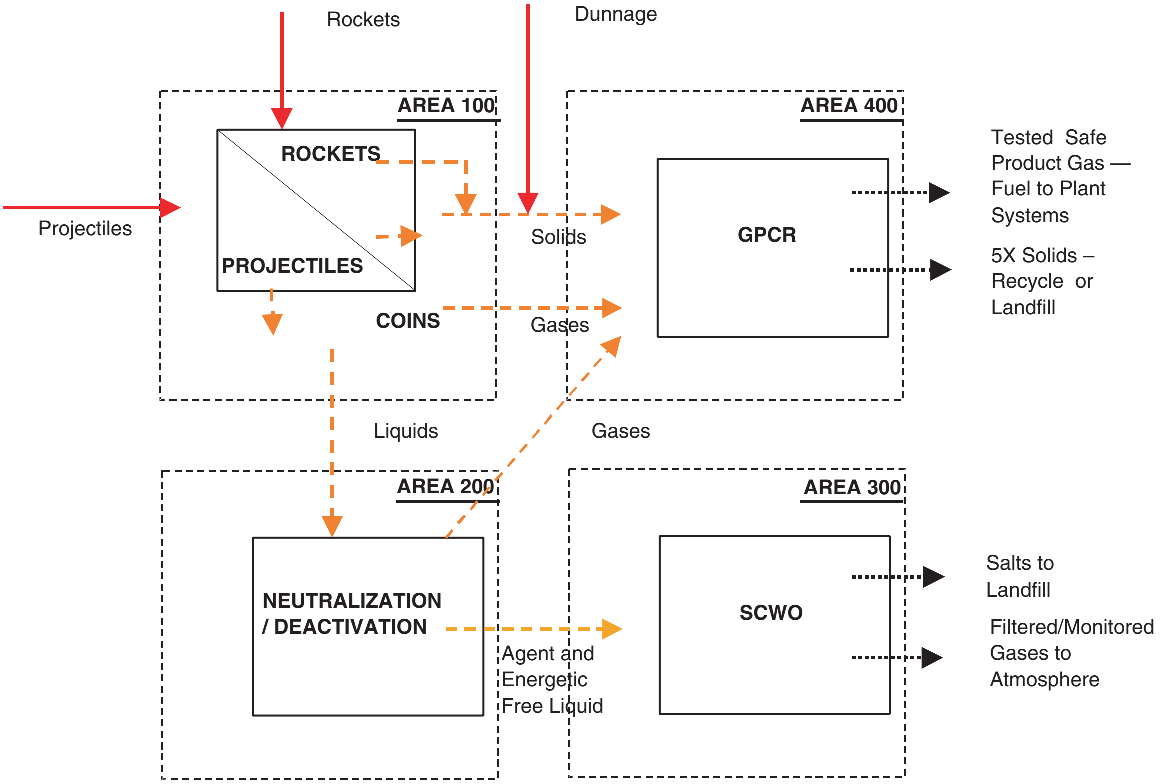

FIGURE 3-1 Schematic diagram of the FW/EL/K demilitarization process. SOURCE: FW/EL/K, 2000.

- Demonstrate the long-term, continuous operability of the TW-SCWO unit with respect to salt plugging, corrosion, integrity of the platelet liner, and erosion of the pressure control valve of the TW-SCWO reactor.
- Determine if aluminum from the energetic hydrolysis process can be processed by the TW-SCWO reactor without plugging.
- Demonstrate that the TW-SCWO can destroy CWC Schedule 2 compounds in the feed to below their detection levels.
- Characterize the gas, liquid, and solid process streams from the TW-SCWO process for selected chemical constituents and physical parameters and the presence or absence of hazardous, toxic, agent, and CWC Schedule 2 compounds.

To meet these objectives, four 100-hour validation tests of the TW-SCWO unit were planned, with each test preceded by a workup run. The tests were designed to demonstrate the DRE for Schedule 2 compounds and the long-term continuous operation of the TW-SCWO unit without salt plugging or corrosion. The feed for the first validation test was identical to that used by General Atomics in Demo I testing in 1999 to compare the performance of the TW-SCWO designed by Foster Wheeler with the General Atomics SCWO. The feeds for the remaining three tests were mixtures of agent and energetic hydrolysates in the same ratios expected from the hydrolysis of specific munitions. The four test runs had the following feeds:

- test 1: VX hydrolysate simulant
- test 2: HD/tetrytol/aluminum hydrolysate and simulant
- test 3: GB/Composition B/aluminum hydrolysate and simulant
- test 4: VX/Composition B/M28 propellant/aluminum hydrolysate and simulant

The first demonstration run lasted the planned 100 hours but was interrupted twice when the air compressor failed. The technology provider stated that this compressor unit would not be used in the full-scale system. Problems encountered during the other three validation tests included (1) deformation of the top edge of the liner, caused by poor distribution of transpiration water attributable to a manufacturing defect; (2) stress cracking of the upper liner, caused by corrosion and by thermal cycling during frequent unanticipated shutdowns; and (3) poor distribution of the feed into the reactor after a broken drill bit was left in the feed nozzle. Because of these problems, which are discussed in more detail below, the second run was terminated after 55 hours, the third was reduced from the scheduled 100 hours to 50 hours, and the fourth was reduced to 25 hours. Nevertheless, the technology provider considers that all of the tests were successfully completed and met the test objectives, with the exception of the length of the runs.

During the workup run for the second test, pressure fluctuations indicated that the upper section of the reactor liner had been damaged. This reactor had been previously used in testing for a Navy program and was not designed for the ACWA demonstration tests. Inspection of the upper section revealed a crack and corrosion damage. Therefore, immediately before the HD test, the upper section of the liner had been replaced with a liner of the same external dimensions but with a lower transpiration flow rate. Because the replacement liner was designed for a lower section of the reactor, it had smaller holes than the top sections and a slower flow rate; accordingly, a bleed hole for additional transpiration water flow was added to ensure the appropriate pressure drop and to increase the flow rate. This field modification led to a highly asymmetric flow of transpiration water and formation of a blister in the liner near the bleed hole, which was discovered during testing.

During the aluminum-rich HD test (test 2), the feed injector became plugged. To eliminate this problem in subsequent GB and VX tests, both of which contained aluminum-rich energetic hydrolysate (tests 3 and 4), the feed was not preheated and its pH was increased. No plugging of the feed injector occurred in these two tests. Visual observation of the reactor liner after the completion of all tests showed no evidence of salt plugging. Reasonably steady differential pressures across the liner for the duration of the runs also suggested that there was little or no salt buildup. Several problems were encountered during test 4, including difficulty in starting up the reactor, a distorted spray pattern from injector ports, and failures of the caustic feed pump.

Despite the problems encountered during testing, adequate destruction of CWC Schedule 2 compounds was demonstrated for the gaseous, liquid, and solid effluents from the TW-SCWO system. The concentrations of many chemical constituents in the gaseous effluent were highly variable.

Other Observations

During the workup run for test 2 (HD/tetrytol/aluminum hydrolysate and simulant), the technology provider discovered that no water was passing through the top 10 rows of transpiration holes (approximately the top 1.5 inches of the reactor). This problem was the result of a manufacturing defect. Because of the aggressive ACWA testing schedule, however, the Army and Foster Wheeler agreed that fabrication, testing, and replacement with a proper upper liner was not possible. Test 1 (VX hydrolysate simulant) was completed with no changes to the reactor. During the workup for test 2 (HD/tetrytol/aluminum hydrolysate and simulant), pressure fluctuations indicated the upper liner had been damaged. Inspection of the cracked upper liner after it was removed revealed severe corrosion caused by an absence of transpiration water flow in the area surrounding the crack. No evidence of corrosion was found in the region of the reactor liner that was protected by transpiration water flow. At that point the cracked upper liner was replaced with a spare lower liner that had to be modified by adding a bleed hole.

Based on the committee's knowledge of the operation of SCWO reactors of different sizes and models of their performance, the most corrosive environment is in the turbulent region at and below the feed nozzle, a region that extends approximately 5 reactor diameters down the reactor from the nozzles. For the reactor used in the Demo II test (6.065 inches in diameter and 63 inches long), this corrosion region would be within the top 30 inches of the reactor. Because corrosion was observed only in the top 1.5 inches, which did not have sufficient transpiration water flow, Foster Wheeler suggested that this reactor design would be effective with a properly constructed transpiring wall. In the tests following replacement of the reactor liner, no evidence of significant corrosion or salt buildup was found in the TW-SCWO reactor by post-test visual inspection and analysis of metals in the liquid effluent stream. This suggests that the transpiration water did protect the liner from corrosion and salt buildup.

During cleanup after test 2, a fine drill bit had been left in the inlet feed manifold, and a flow test partway through the VX campaign (test 4) showed an uneven flow from the feed ports. FW/EL/K believes the drill bit caused the erratic feed injection behavior, causing some of the feed to bypass the high-temperature SCWO reactor zone. Thus, some isopropyl methylphosphonic acid (IMPA) and tributylamine (TBA) were found in the initial 4-hour composite sample, and methylphosphonic acid (MPA) in the 4-, 24-, 34-, and 46-hour composite samples. The MPA concentrations ranged from 61 to 118.2 parts per million (ppm). These results indicate the TW-SCWO reactor design and operation are vulnerable to slight plugging of the feed nozzle.

The initial pressure control valve eroded during the workup run for the aluminum-rich GB campaign (test 3). When it was replaced with an abrasion-resistant valve, no erosion was observed during the aluminum-rich GB and VX campaigns (tests 3 and 4), which totaled 75 hours. During these aluminum-rich campaigns, the feed preheat heat exchanger did become plugged. This heat exchanger, which was a legacy from a prior program and was not designed for use with slurry, will not be used in the full-scale plant. Because the TW-SCWO reactor used in the Demo II tests was designed and built to process low-solids Navy wastes, some problems were encountered in treating feed hydrolysates with high concentrations of solids.

In the full-scale design, Foster Wheeler intends to use a TW-SCWO reactor that is 9 inches in diameter. This is only 50 percent larger in diameter and 2.25 times larger in cross-sectional area than the Demo II test reactor, so there will be only a small scale-up in size. However, there will be a significant scale-up in flow. The hydrolysate flow to the reactor in Demo II was 60 lb/hr, while the full-scale reactor will treat 2,100 lb/hr, which would be inconsistent with the small increase in cross-sectional area. This suggests that the ratio of hydrolysis to total feed will be increased by approximately a factor of 10 over the Demo II tests. FW/EL/K believes this problem can be overcome by proper design of the reactor and by changing the oxidant from air to oxygen.[1] The committee believes these modifications should be demonstrated during EDS testing.

Before the full-scale plant design is finalized, the committee also recommends demonstration tests for the following changes suggested by Foster Wheeler:

- using oxygen instead of air in the SCWO reactor
- increasing the temperature of the transpiration water to reduce thermal stresses during planned and unplanned shutdowns
- altering the flow circuitry of water in the transpiring wall
- using an evaporator/crystallizer instead of a flash separator after the reactor

A much more detailed discussion of the TW-SCWO Demo II testing can be found in *Assembled Chemical Weapons Assessment Program: Annual Report to Congress* (DoD, 2001) and *Foster Wheeler/Eco Logic/Kvaerner Draft Final PM-ACWA Demonstration II Test Program Technical Report* (FW/EL/K, 2000).

GAS-PHASE CHEMICAL REDUCTION SYSTEM

The following objectives were established for the GPCR™ system demonstration tests (DoD, 2001):

> - Validate the ability of the GPCR™ process to achieve 5X decontamination condition for metal parts and dunnage.
> - Demonstrate the effectiveness of the GPCR™ process to treat product gases generated during the treatment of metal parts and dunnage.
> - Validate the ability of the GPCR™ process to achieve a DRE of 99.9999 for HD and neat GB.
> - Characterize the gas, liquid, and solid process streams from the GPCR™ process for selected chemical constituents and physical parameters and the presence or absence of hazardous, toxic, agent, and CWC Schedule 2 compounds.
> - Demonstrate the ability of the GPCR™ process to produce a gas effluent that meets either EPA syngas or the hazardous waste combustion and thermal treatment regulations and requirements.
> - Determine the need for stabilization of residual dunnage solids based on the results of toxicity characteristic leachate procedure (TCLP).

One workup and three validation runs were planned for each feed to test the treatment of dunnage and chemical agents. The following materials were tested:

- trays containing activated carbon filter media
- wood spiked with 4,000 ppm pentachlorophenol (PCP)
- DPEs with 10 percent butyl rubber by weight to simulate gloves and boots
- fiberglass firing tubes
- neat GB
- M2A1 4.2-inch mortars spiked with simulated 30 percent HD heel

The GPCR™ process was able to achieve a 5X-level decontamination for carbon, PCP-spiked wood, DPE, firing tubes, and HD mortars. The first run with DPE was stopped prematurely because the reactor input line became plugged.

Reactor product gases were effectively treated in the two-stage scrubber system and product gas burner (PGB). However, process modifications may be necessary to control hydrogen sulfide and phosphine in the off-gas from the GPCR™ reactor. The liner in the PGB also showed evidence of corrosion during ACW Demo II tests.

In the test with partly filled 4.2-inch HD mortar shells, no agent was detected in either the scrubber solution or the scrubber filters. However, product gas sampling methods did not work as intended, so that DREs for the HD and GB runs could not be determined. Based only on the scrubber solution and filter results, FW/EL/K estimates a DRE of at least 99.9999 percent. However, this could not be verified. Earlier tests of the GPCR™ at Aberdeen (or Edgewood) in 1996 were able to destroy VX to a DRE of 99.999999 percent (NRC, 1999). Although the technology provider has analyzed the scrubber solution, the committee points out that the scrubber solution, which is pH-controlled caustic, is likely to hydrolyze any chemical agent that is absorbed. Therefore, agent would not be detected in the scrubber solution under any conditions.

The gas, liquid, and solid process streams from the GPCR™ process were analyzed for specified chemical constituents and physical parameters, as well as for the presence of hazardous, toxic, agent, and CWC Schedule 2 compounds. For the tests that included chemical agents, scrubber solution and scrubber filters were completely characterized. However, the gas sampling streams for the runs with agents could not be analyzed. During the test runs on GB and HD, Army personnel responsible for the use of the MiniCAMS (a type of air monitoring instrument) declined to use the unit on the product gas stream despite prior approval in the Test Plan, Safety Plan, and Site Review. This decision was based on the fact that the MiniCAMS had not been previously used for agent destruction streams (FW/EL/K, 2000). Testing of the GPCR™ product gas for agent was also inconclusive, because the tests showed that depot area air monitoring sys-

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[1] The problems anticipated in switching from air to oxygen were addressed in the NRC report *Using Supercritical Water Oxidation to Treat Hydrolysate from VX Neutralization* (NRC, 1998).

tem (DAAMS) tubes, which were designed for use on stack gas, were inadequate for use for the GPCR™ product gas.

A method of off-gas analysis and validation will therefore have to be developed before EDS tests are conducted. Because the Demo II gas samples could not be certified to be free of agent, they could not be taken off-site for complete analysis.

Except for the GB and HD campaigns, for which the data were incomplete, the stack off-gas did meet the hazardous waste combustion and thermal treatment regulations and requirements criteria for waste streams.

The GPCR™ product off-gas did not meet either the EPA comparable fuel exemptions or the synfuel characteristics (40 CFR 261.38) owing to unacceptable levels of benzene and polyaromatic hydrocarbons. The combustion of the product gas will therefore be regulated under RCRA. FW/EL/K also evaluated the stack gases from combustion of the GPCR™ product gas in the PGB. The PGB stack gas was found to meet the boiler and industrial furnace (BIF) emission standards (40 CFR 266.1). However, the EPA has recently promulgated new maximum achievable control technology (MACT) standards for hazardous waste combustion which are more stringent than BIF standards. FW/EL/K must determine whether the emissions from the PGB can meet the MACT standards, not just BIF rules.

Dioxins were detected in the GPCR™ off-gas only in tests with PCP-spiked wood, DPE suits, and butyl rubber. The levels were 0.0002 to 0.0008 ng/m^3, three orders of magnitude below the EPA criterion of 0.2 ng/m^3 for dioxin emissions from incinerators. In the neat GB test, the product off-gas contained 0.02 to 0.07 percent benzene and 0.01 to 0.06 percent phosphine.

A further objective was to determine the need for stabilization of residual dunnage solids based on toxicity characteristic leachate procedure (TCLP) results. Except for the DPE-solids, stabilization would not be necessary for the GPCR™ solid wastes. Stabilization of DPE-derived solids was necessary because in some tests the cadmium and lead criteria were not met by the dunnage treatment.

SAFETY CONCERNS

As described in the ACW I committee's previous report on the FW/EL/K design (NRC, 1999),

> The most significant worker safety issue will probably be maintenance of the hydrolysis vessels in DPE suits. These vessels have conveyor systems that operate in hot caustic solutions.

Since then, the hydrolysis process has been changed; the moving parts driving the baskets have been removed from the caustic bath and replaced with an overhead chain conveyor system. As a result, the maintenance associated with the hydrolysis process, which is performed by workers in DPE suits, is expected to be reduced, with a corresponding increase in worker safety.

The ACW I committee was also concerned that (NRC, 1999)

> The GPCR™ reactor operates in a hydrogen atmosphere and generates methane and other gaseous hydrocarbons that could burn or explode in the presence of air.

The Demo II tests showed that the safe operation of this high-temperature hydrogen process could be achieved in the relatively short time allowed for construction of the equipment, systemization, and training of Army operators. However, a preliminary hazards analysis must seriously evaluate the use of hydrogen in a closed area.

FW/EL/K has simplified the reverse-assembly/munitions-access processes. One of the changes involves grinding M28 propellant under a deluge of ambient-temperature caustic hydrolysis fluid. The ACW I committee had expressed a related concern (NRC, 1999):

> Friction, shear, or heat may result from the inadvertent introduction of metal, an excessive feed rate, or some other cause and could initiate the energetic material.

Demo I tests showed that inundating the shredder face with cooling water would prevent propellant ignition in the absence of metal parts (NRC, 2000). The proposed hydrolysis fluid or water deluge appears to address this concern. FW/EL/K has noted, however, that metal parts may occasionally enter the grinder, so the possibility of propellant ignition presented by the occasional metal part should be kept in mind as the design progresses. The committee believes that deluging with water would be sufficient and much less corrosive to the grinding equipment.

REEVALUATION OF STEPS REQUIRED FOR IMPLEMENTATION

The ACW I committee identified several steps to be addressed prior to implementation of this technology package (NRC, 1999). These are quoted below and reevaluated on the basis of the Demo II test results.

Overarching Comment

> Overall, the LMIDS appears to be capable of operating as proposed by the technology provider, but the process must be developed further, especially the interfaces between and integration of the process units. If the LMIDS were to proceed towards full-scale implementation, the next step should be to design, build, and operate a pilot-scale system that incorporates all of the unit operations into a fully functional, integrated process. Full-scale implementation will involve interfacing and integrating batch processes (the hydrolysis reactors and the thermal reduction batch processor) with continuous processes (the SCWO reactor, the thermal reduction continuous processor, and the GPCR™ reactor). These in-

terfaces must be tested at the demonstration stage to avoid implementation problems. Also, all problems with materials of construction and waste characterization will have to be solved before implementation. However, no problems have been identified that would prevent eventual full-scale implementation.

Pilot-Scale Evaluation for Hydrolysis of Energetics

1. Establish that the mechanical equipment used in the energetics hydrolysis vessels can tolerate the harsh conditions without excess maintenance.

Foster Wheeler has redesigned and simplified this process, but the recommendation is still valid.

2. Determine whether the hydrolysis of aluminum together with energetics presents any problems.

Hydrolysis of aluminum with energetics was not tested in Demo II. However, aluminum in the hydrolysate produced a slurry, which led to downstream fouling of the heat exchanger. Since then, FW/EL/K has changed the temperature and pH of the hydrolysate. This modification will have to be demonstrated for long-term operation with aluminum in combination with energetics. Another approach would be to reduce the amount of aluminum that enters the hydrolysis unit (by, for example, separating rocket fins).

Pilot-Scale Evaluation for SCWO

1. Show that the SCWO reactor platelet wall can be constructed.

Several units have been built and are in operation; therefore, this technology has been established. However, the Demo II tests revealed possible difficulties with the construction and operation of the TW-SCWO reactor.

2. Demonstrate that the SCWO reactor can be operated for sufficient periods of time without excessive clogging or corrosion.

Long-term tests with a TW-SCWO reactor designed specifically for this application will have to be done to demonstrate the operability of the TW-SCWO without excessive clogging and corrosion. The tests should also determine whether transient pressure surges or fluctuations could temporarily or permanently plug the transpiration holes. Demo II results show that, without the protection of transpiration water, the upper section of the platelet liner is subject to severe corrosion and salt deposition. Therefore, transpiration water flow must be maintained.

3. Fully characterize the SCWO gaseous effluent from mixed hydrolysates of agent and energetics.

This has effectively been done.

4. Establish that the continuous monitoring of the SCWO gaseous effluent ensures against unacceptable releases of hazardous materials.

This remains to be done and should be included at the EDS tests stage. Note, however, that all of the Demo II tests of the SCWO system have been done at flow rates and velocities that are significantly lower than, and a hydrolysate/total feed ratio different from, those of the proposed full-scale design. Therefore, in addition to establishing the long-term reliability of the SCWO system, it must be shown that corrosion and salt deposition will not be a problem at the design conditions. The higher flow velocities at the design conditions will result in a thinner wall boundary layer, which may change the effectiveness of the transpiring wall. Also, it needs to be established that satisfactory destruction efficiencies can be obtained at these higher flow rates.

Pilot-Scale Evaluation for GPCR™

1. Fully characterize the GPCR™ gaseous effluent and establish whether it can be used as a boiler fuel.

Demo II tests have shown that the GPCR™ off-gas does not meet the EPA syngas criterion. The stack gas from the PGB did meet the BIF emission requirements but may not meet the new EPA MACT requirement and therefore may be subject to RCRA regulations.

2. Ascertain whether the large quantity of soot generated in the thermal-reduction batch process will create any problems.

Based on the postwaste feed reactor cleanout (no solids recovered), there was little evidence that carbon dust made it out of the batch GPCR™ thermal reduction batch processor (TRBP) during the PCP-spiked wood test.

A comparison of prerun and postrun scrubber solutions showed that during the processing of firing tubes, suspended particulate levels in the scrubber solution did not increase. This suggests that particulate matter (carbon dust) from the breakdown of the firing tubes did not move from the batch TRBP to the scrubbers. The TRBP treatment of the firing tubes produced a solid residue that largely remained in the reactor. In the treatment of neat GB and mortars containing HD, no suspended solids were detected in the scrubber solution, indicating that appreciable amounts of carbon dust did not form in the reactor and exit with the product gas. Furthermore, in processing a mortar initially containing 810 grams of HD, the amount of residue after processing was too small for solids analysis.

REEVALUATION OF FINDINGS FROM ACW I REPORT

Finding LM-1. The disassembly methods proposed in the LMIDS are based largely on the baseline disassembly methods. The proposed modifications appear to be reasonable, but testing will be necessary to verify that performance, reliability, and production objectives can be met.

The disassembly process was not tested in Demo II, so this finding remains valid.

> **Finding LM-2.** Primary agent decomposition and detoxification is achieved using a strong caustic hydrolysis of bulk agent—a proven technology. Overall, the implementation of agent hydrolysis in the LMIDS is sound.

No additional test was recommended or required, so this finding remains valid.

> **Finding LM-3.** Primary decomposition and deactivation of energetics is also achieved using a strong caustic hydrolysis. This technology has been tested but is less mature than agent hydrolysis. The implementation of this technology in the LMIDS is reasonable but will require thorough testing at the pilot scale.

Energetics hydrolysis with caustic is the subject of a separate testing program being conducted during the EDS design phase of the ACWA program.

> **Finding LM-4.** The method of removing agent from metal parts—caustic solution jet washout followed by the movement of the parts in baskets through a caustic bath—is new and unproven. It is expected that this method can be made to work, but the effort and time required to come to acceptable performance goals may be longer than anticipated and may require alternate methods. Thus, it will be desirable to have alternate plans if the desired detoxification efficiencies are not achieved (e.g., increase the capacity of the GPCR™ unit to allow for more than the planned agent cleanup load).

> **Finding LM-5.** The hot-caustic environments in the initial hydrolysis vessels will pose severe challenges to the reliability and operability of the equipment inside these vessels, especially the basket transport mechanisms.

Removal of agent by caustic solution jet washout and movement of metal parts through a caustic bath in baskets were not tested in Demo II. FW/EL/K has proposed a new, simplified process so that only the baskets, not the conveyor system, are in contact with the corrosive hot caustic bath. Reliability of the overhead conveyor system in the presence of hot caustic vapor has not been demonstrated. The design change is likely to improve the reliability and reduce the maintenance requirement. However, this design should be tested before it is incorporated into a full-scale design.

> **Finding LM-6.** The SCWO process appears to be capable of completing the destruction of both agent and energetics in the hydrolysates. The key area of uncertainty in the technology provider's proposed application of SCWO is the use of its proprietary transpiring-wall tubular reactor. The demonstration of this technology will be essential to proving the efficacy of this crucial step in the agent/energetics destruction process.

The ability of the TW-SCWO reactor to destroy CWC Schedule 2 compounds in hydrolysates has been successfully demonstrated. The ability of the transpiring wall to eliminate salt plugging and corrosion has not been completely demonstrated. The committee believes that long-term reliability has not yet been demonstrated.

> **Finding LM-7.** The crystallization and evaporation operations have not been tested for this application. These conventional technologies, which are expected to work effectively, must still be tested.

These operations were not tested in Demo II, so this finding remains valid. Experience at the Tooele facility has shown that there may be better alternatives than evaporating brine streams, such as shipping off-site.

> **Finding LM-8**. The use of GPCR™ in an enclosed environment raises unique safety concerns because of the presence of hot hydrogen gas. Hydrogen is handled routinely (and safely) in the chemical industry, and the technology provider is aware of the hazards. Implementation of this technology will require a design that ensures that these hazards are thoroughly understood and mitigated.

This conclusion remains unchanged. However, it is important to note that these full-scale units have been operated by inexperienced Army personnel without an accident, rather than by experienced GPCR™ operators.

> **Finding LM-9.** The gas stream from SCWO is not subjected to hold-test-release. Instead, the gas is scrubbed, monitored, and passed through activated carbon. This treatment appears to be appropriate for the anticipated composition of the SCWO off-gases.

The results of Demo II and the characterization of all effluents from the TW-SCWO confirm this conclusion.

> **Finding LM-10.** Pilot-scale testing will be necessary to refine the component technologies and demonstrate that these technologies can be operated as an integrated system.

Further testing of the unit operations is planned in the EDS program. Integration issues are being addressed during EDS but will not be tested until the full-scale plant is constructed. Integration of the unit operations will be crucial to the successful implementation of this technology and, indeed, of all the technologies presently in demonstration and engineering design.

> **Finding LM-11.** The proposed use of the cleaned GPCR™ off-gas as a boiler fuel poses unique permitting challenges. Any demonstration must characterize this stream to ensure that permitting as a boiler fuel is possible. If this off-gas cannot be used as a boiler fuel, significant process modifications may be necessary.

The Demo II tests showed that the GPCR™ off-gas does not meet the EPA syngas criterion. Therefore, the boiler or other device in which this stack gas is burned may be subject to RCRA regulations.

> **Finding LM-12.** All of the findings in the NRC report, Using Supercritical Water to Treat Hydrolysate from VX Neutralization (NRC, 1998), apply to the LMIDS SCWO system.

This finding is unchanged. A 2001 letter report by the NRC reiterated that materials of construction and salt transport are among the issues that have to be resolved (NRC, 2001).

SUPPLEMENTAL FINDINGS

Finding DII FEK-1. The proposed full-scale TW-SCWO system has design and operating conditions significantly different from those tested in Demo II. These include the temperature of the transpiration water at the inlet; pH of the feed; turbulence in the reactor; and use of pure oxygen, not air, as the oxidant.

Finding DII FEK-2. The proposed full-scale design for the TW-SCWO system involves a scale-up in reactor cross-sectional area by a factor of 2.25 from the Demo II test unit and an increase in reactor throughput by a factor of 35. Performance under these full-scale design conditions has not been demonstrated.

Finding DII FEK-3. Aluminum present in the hydrolysates, which could lead to the formation of slurries and plugging, could be a problem. The proposed changes for mitigating this problem (e.g., changing operating conditions and/or removing aluminum during weapon disassembly) must be tested.

Finding DII FEK-4. Demo II tests confirmed that firing tubes and other solids could be treated to a 5X condition by the GPCR™ process.

Finding DII FEK-5. All waste streams have been or can be characterized sufficiently for engineering design to proceed.

Finding DII FEK-6. The current sampling and monitoring systems for agent in gaseous streams have not been certified or validated for use with the GPCR™ process off-gas.

Finding DII FEK-7. The product gas from the GPCR™ process does not meet the EPA syngas requirements because of high benzene and polyaromatic hydrocarbon content.

Finding DII FEK-8. While no agent was detected in the scrubbing solutions and scrubber filters, the ability of the GPCR™ process to destroy HD in mortars and neat GB could not be confirmed because sampling and analysis problems hampered the gathering of gas-phase data.

Finding DII FEK-9. Little evidence of soot formation was indicated when the GPCR™ unit was tested separately with PCP-spiked wood, HD mortars, M55 rocket firing tubes, and neat GB.

Finding DII FEK-10. The full-scale SCWO reactor design has not been tested and is different in size and in the flow rates of the feed streams from those used in the Demo II tests. The full-scale design treats hydrolysate at a rate per unit volume of reactor that is almost 10 times higher than that used during the Demo II tests. In addition, the ratio of the flow rates of all other streams to the flow rate of hydrolysate in the full-scale unit has decreased by approximately a factor of 10 from those used during the Demo II tests. These changes in hydrolysate processing per unit of reactor volume and the reduction of other feed streams relative to the hydrolysate may reduce the efficacy of the SCWO reactor and may be expected to exacerbate problems of corrosion and plugging.

Finding DII FEK-11. The experience of multiple shutdowns during Demo II testing of the TW-SCWO and the resulting thermal stresses and crack generation in the liner indicate a potential reliability issue, which must be significantly reduced or eliminated.

SUPPLEMENTAL RECOMMENDATIONS

Recommendation DII FEK-1. Since the hydrolysate/total feed ratio and flow velocity used in Demo II testing are so different from those of the proposed design, the TW-SCWO reactor must be tested at a hydrolysate/total feed ratio and flow velocities close to the proposed design conditions.

Recommendation DII FEK-2. Long-term testing of appropriately designed SCWO reactor liners under the new operating conditions for the proposed full-scale operation will be necessary to prove the reliability and effectiveness of the TW-SCWO unit.

Recommendation DII FEK-3. Long-term testing of the TW-SCWO should include feeds containing chlorine, phosphorus, and sulfur and be at residence times and flow velocities close to the proposed design conditions.

Recommendation DII FEK-4. The Army or the technology provider must develop analytical methods to determine the quantities of agent in the gas streams containing hydrogen.

4

Teledyne-Commodore Solvated Electron Technology Process

The Teledyne-Commodore process uses ammonia fluid jet cutting and washout for the disassembly of munitions and separation of agent, energetics, and metal parts. The agent and energetics are then destroyed by solvated electron technology (SET™), a process that reduces contaminants with a solution of metallic sodium in anhydrous liquid ammonia at ambient temperature and pressures of 110 to 167 pounds per square inch gauge (psig). The sodium ammonia reagent is prepared as it is used. The solid and liquid residuals from the SET™ process are first hydrolyzed with water to destroy the excess sodium. The hydrolysate is then oxidized with sodium persulfate or hydrogen peroxide to form environmentally more benign effluents. Metal parts are shredded and treated to 3X condition with SET™ solution in a tumbler. Dunnage is shredded and treated with SET™ solution in a rotary plow mixer. A schematic diagram of the process is shown in Figure 4-1.

The following unit operations were selected for Demo II testing:

- ammonia fluid jet cutting and washout
- SET™ destruction of energetics
- chemical oxidation of energetics hydrolysates
- SET™ destruction of agents
- chemical oxidation of agent hydrolysates
- shredding of metal parts and dunnage
- SET™ treatment of shredded metal parts and dunnage

The testing was terminated by the PMACWA after two incidents occurred: (1) energetic ignition of M28 propellant and (2) a sulfuric acid spill during equipment servicing for the demonstrations of SET™ with agents.[1] As a result of cost overruns and schedule constraints attributed to these incidents, the following tests were not conducted:

- SET™ destruction of energetics
- chemical oxidation of energetics hydrolysates
- SET™ destruction of agents
- chemical oxidation of agent hydrolysates

AMMONIA FLUID JET CUTTING AND WASHOUT

The objectives of the tests of fluid jet cutting and washout were as follows:

- Demonstrate the ability to prepare a suitable feed to the SET™ and oxidation reactors.
- Demonstrate the separation of the burster and propellant from the rockets.
- Demonstrate the accuracy and precision with which the fluid jet cutting system can position and cut the rockets using manual placement of the rockets.
- Determine the impact of operations on components in the SET™ chamber (e.g., integrity of the chamber seals).

The test plans called for validation runs on 15 inert M60 rockets that contained neither energetics nor agent and on three M61 rockets containing Composition B and M28 but no agent. The M60 tests were completed accurately and precisely. Visual examination showed no damage or degradation of chamber components.

No validation runs were completed on M61 rockets because the energetics ignited in the second workup (practice) run. At that point, all testing was stopped. A report of an investigation concluded that the most probable cause of the ignition was an exothermic reaction between ammonia vapor and M28 propellant. Ammonia vapor ignited after the propellant burn. Teledyne-Commodore subsequently deter-

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[1] Teledyne-Commodore installed a sulfuric acid scrubber to capture ammonia vapor. The spill was caused by the failure of a check valve in the scrubber system.

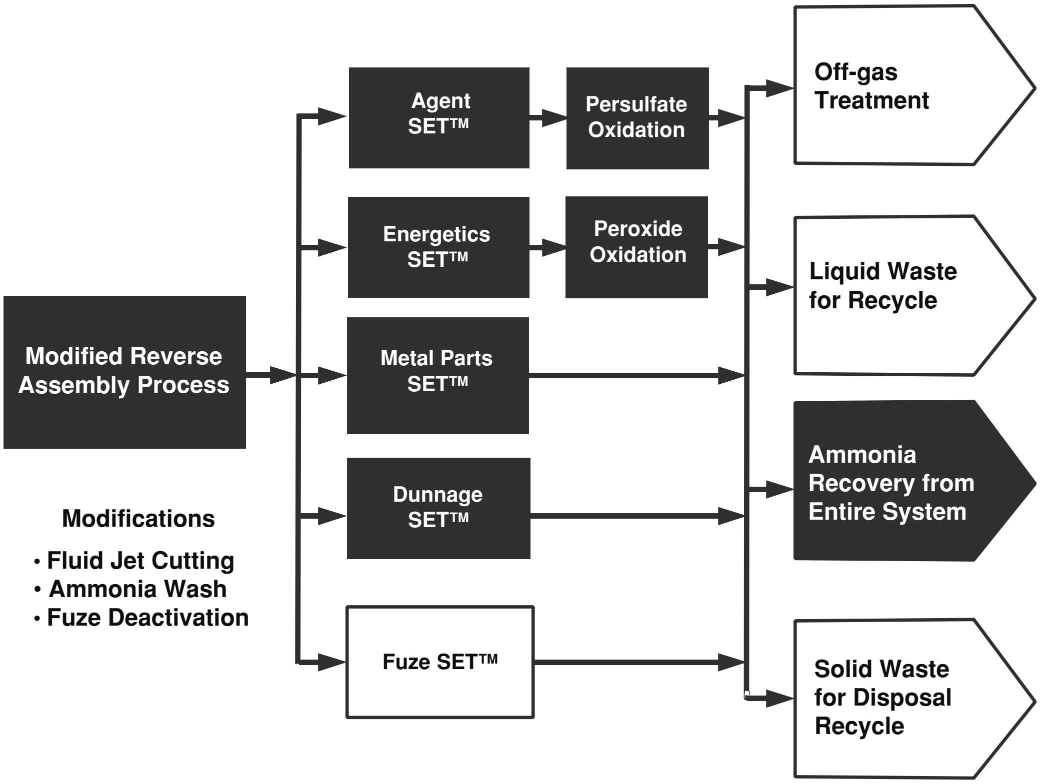

FIGURE 4-1 Schematic diagram of the Teledyne-Commodore SET™ process. Solid boxes represent unit operations for demonstration. SOURCE: PMACWA (2001).

mined that M28 propellant is chemically incompatible with ammonia vapor (Kumar, 2000).

The investigation report also documented another potential safety issue. Investigators found that Composition B dissolved in liquid ammonia had leaked through flanges into the valves and piping transferring the material from the ammonia fluid jet-cutting vessel to the SET™ reactor. The investigators were unable to disassemble the piping and valves during the investigation because dissembling parts contaminated with Composition B would have posed a hazard.

SHREDDING OF METAL PARTS AND DUNNAGE

The objectives of the shredding demonstrations were as follows:

- Validate that the shredder can adequately prepare the dunnage and metal parts for downstream processing in the SET™/dunnage reactor.
- Demonstrate the safe, effective handling and feeding of shredded dunnage and metal parts into the SET™/dunnage reactor.

Validation runs were conducted as planned on DPE/butyl bags (518 lb); wood pallets (52 lb) and pentachlorophenol; fiberglass firing tubes (54 lb); and M42A1 4.2-inch mortars (362 lb). The runs were carried out at a commercial facility, and all feed types were successfully size-reduced for subsequent processing in the SET™/dunnage reactor. Five pounds of each shredded material, spiked with simulant, were fed successfully into the SET™/dunnage reactor.

SET™ TREATMENT OF SHREDDED METAL PARTS AND DUNNAGE

The objectives of the tests of SET™ treatment of shredded metal parts and dunnage were as follows:

- Validate that the SET™/dunnage reactor could meet a 3X condition for agent simulants, metal parts, and dunnage.
- Relate the characterization of SET™/dunnage reactor off-gas to produce a total facility gas effluent that meets either the EPA syngas or BIF requirements.
- Characterize gas, liquid, and solid process streams from the SET™ process for selected chemical constituents and physical parameters and the presence or absence of hazardous and toxic compounds, including residual agent simulants.

Validation tests were conducted as planned on 5-lb aliquots of shredded DPE/butyl bags spiked with simulant, shredded wood pallets spiked with simulant and PCP, carbon spiked with simulant (shredding not required), shredded fiberglass firing tubes spiked with simulant, and shredded M42A1 4.2-inch mortars spiked with simulant.

All five dunnage feeds were processed as planned, and all necessary validation data were collected. However, the data did not validate that the SET™/dunnage reactor could meet a 3X condition for all feeds. The criterion for 3X is that agent concentration in the headspace above the treated solids does not exceed 3.0 µg per cubic meter of air for HD, 0.01 µg per cubic meter of air for VX, and 0.1 µg per cubic meter of air for GB. The sampling and analysis procedures used for the simulants (1,4-dichlorobutane for HD and GB and malathion for VX) yielded detection limits in the range of 100 to 200 µg per cubic meter of air, too high to validate 3X decontamination of dunnage materials (Teledyne-Commodore, 2000). The major test objective was not met. In any case, 1,4-dichlorobutane is probably not a very good simulant for mustard and GB.

REEVALUATION OF STEPS REQUIRED FOR IMPLEMENTATION

The steps required for implementation from the ACW I committee's report are reiterated below, along with a summary of the status as a result of the Demo II testing (NRC, 1999).

> 1. Verify products of SET™/hydrolysis of agents through experimentation.

This was not verified because testing was suspended.

> 2. Establish optimum conditions for SET™/hydrolysis of agents through laboratory tests, followed by pilot-plant demonstration.

This was not established because testing was suspended.

> 3. Identify the unknown precipitates of SET™ energetics reactions.

This was not identified because testing was suspended.

> 4. Establish optimum conditions for the oxidation of residuals from SET™/hydrolysis of both agents and energetics through laboratory tests, followed by pilot-plant demonstration.

This was not established because testing was suspended.

> 5. Test waste disposal methods.

This was not tested.

> 6. Conduct pilot tests of methods of decontaminating metal parts and dunnage.

The tests for this were inconclusive.

> 7. Revise the preliminary design for the hypothetical system, especially the interfaces between the unit processes and operations, and demonstrate the revised design at pilot-scale.

The data obtained from the demonstration tests of unit processes and operations were insufficient for meaningful redesign.

REEVALUATION OF FINDINGS FROM ACW I REPORT

> **Finding TC-1.** The use of ammonia jet cutting in the munitions disassembly process could solve some of the problems encountered in baseline disassembly. However, the process must be thoroughly tested to address reliability and maintenance issues.

The ignition that occurred during the workup run on M61 rockets calls into question the reliability of the system for the intended application.

> **Finding TC-2.** Conditions for SET™ destruction of agents have been reasonably well established but demonstrated only on a small scale.

> **Finding TC-3.** Conditions for SET™ destruction of energetics have not yet been determined. Moreover, energetics have not been completely deactivated in laboratory tests, which raises concerns about explosions or other upsets.

> **Finding TC-4.** The reaction chemistry is not yet fully understood for either SET™ destruction of agents or SET™ deactivation of energetics.

> **Finding TC-5.** The products of SET™/hydrolysis of agent and energetics have not been adequately characterized. Thus, the technology provider cannot be certain that all of the SET™/hydrolysis products can be oxidized by the persulfate step. Furthermore, the products of oxidation of the SET™/hydrolysis products have not been adequately characterized.

> **Finding TC-6.** The solid wastes produced by the overall process have not been characterized well enough to establish whether they are suitable for safe disposal by existing methods, such as landfill, or whether pretreatment methods (i.e., stabilization) to convert them to an acceptable form for disposal will be necessary.

> **Finding TC-7**. The use of cleaned off-gas as a boiler fuel poses unique permitting challenges. Any process demonstration must characterize this stream to ensure that this off-gas can be permitted as boiler fuel.

Because no agents or energetics were tested, findings TC-2, TC-3, TC-4, TC-5, TC-6, and TC-7 remain valid.

> **Finding TC-8.** The full scale system for hydrolysis of the SET™ products will differ significantly from the systems used in the reduced scale tests. Because further chemical reactions occur during hydrolysis, the components of the full scale system must be tested.

The Demo II test was designed to test some of the components of a full-scale system. The only components for which tests were completed, however, were the dunnage and metal shredders.

SUPPLEMENTAL FINDING

Finding DII TC-1. Demo II tests were delayed and could not be completed for the Teledyne-Commodore process because of incidents in which the immaturity of the process became apparent. For example, an exothermic reaction between ammonia vapor and M28 propellant led to an ignition incident. At another time, Composition B dissolved in liquid ammonia leaked through flanges into valves and piping that were intended to transfer the material from the ammonia fluid jet-cutting vessel to the SET™ reactor. These incidents revealed serious safety problems associated with the Teledyne-Commodore process.

5

Update of General Findings and Recommendations

Chapter 11 in *Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons* included 16 general findings and 7 general recommendations (NRC, 1999). Those findings and recommendations are quoted below and are followed by the current committee's evaluation of how the Demo II tests affect the status of the technologies. The findings in the ACW I supplemental report, *Evaluation of Demonstration Test Results of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: A Supplemental Review* (NRC, 2000), pertained to the three technologies tested during Demo I and are not relevant to the Demo II technologies.

REVIEW OF EARLIER FINDINGS AND RECOMMENDATIONS

> **General Finding 1**. The chemistries of all four of the primary technologies, (hydrolysis, SILVER II™, plasma arc, and SET™) as proposed, can decompose the chemical agents with destruction efficiencies of 99.9999 percent. However, each technology package raises other technical issues that must be resolved. One of the crucial issues is the identity and disposition of by-products.

The Army has produced large quantities of hydrolysate from GB, VX, and HD in which no agent can be detected, thereby confirming that DREs of 99.9999 percent have been achieved. The Demo II testing of the AEA SILVER II™ process has also been shown to achieve DREs of 99.9999 percent for VX, HD, and GB. The Teledyne-Commodore SET™ process was discontinued before progressing to tests with live agent. Complete destruction of agents by the SET™ technology has not been confirmed.

> **General Finding 2.** The technology base for the hydrolysis of energetic materials is not as mature as it is for chemical agents. Chemical methods of destroying energetics have only been considered recently. Therefore, there has been relatively little experience with the alkaline decomposition of ACWA-specific energetic materials (compared to experience with chemical agents). The following significant issues should be resolved to reduce uncertainties about the effectiveness and safety of using hydrolysis operations for destroying energetic materials:
>
> - the particle size reduction of energetics that must be achieved for proper operation
> - the solubility of energetics in specific alkaline solutions
> - process design of the unit operation and the identification of processing parameters (such as the degree of agitation and reactor residence time) necessary for complete hydrolysis
> - the characterization of actual products and by-products of hydrolysis as a function of the extent of reaction
> - the selection of chemical sensors and process control strategies to ensure that the unit operation following hydrolysis can accept the products of hydrolysis
> - development of a preventative maintenance program that minimizes the possibility of incidents during the cleanup of accumulated precipitates
>
> **General Finding 3**. The conditions under which aromatic nitro compounds, such as trinitrotoluene (TNT) or picric acid, will emulsify in the aqueous phase and not be completely hydrolyzed are not well understood. Therefore, this type of material could be present in the output stream from an energetic hydrolysis step.
>
> **General Finding 4**. The products of hydrolysis of some energetic materials have not been characterized well enough to support simultaneous hydrolysis of different kinds of energetic materials in the same batch reactor.
>
> **General Recommendation 5**. Whatever unit operation immediately follows the hydrolysis of energetic materials should be designed to accept emulsified aromatic nitro compounds, such as TNT or picric acid, as contaminants in the aqueous feed stream. (See General Finding 3.)
>
> **General Recommendation 6**. Simultaneous processing of different types of energetic materials should not be performed until there is substantial evidence that the intermedi-

ates formed from the hydrolysis of aromatic nitro compounds will not combine with M28 propellant additives or ordnance fuze components to form extremely sensitive explosives, such as lead picrate. (See General Finding 4.)

The PMACWA has established a technology base program to address the NRC's concerns about the hydrolysis of energetic materials. This program will be reviewed and evaluated in the EDS reports that address Tasks 2 and 3 in the ACW II committee's statement of task. However, as discussed earlier in Chapters 2 and 4, the committee believes that the destruction of energetics by both the SILVER II™ and the SET™ processes, which do not involve hydrolysis, remains immature. This finding is illustrated by the unexpected, and as yet unexplained, formation of insoluble intermediates in the anolyte vessel in the SILVER II™ processing of tetrytol.

General Finding 5. The primary chemical decomposition process in all of the technology packages produces environmentally unacceptable reaction products. Therefore, all of the packages are complicated processes that include subsequent treatment step(s) to modify these products.

General Finding 6. The waste streams of all of the ACWA technology packages could contain very small amounts of hazardous substances (besides any residual chemical agent). These substances were not fully characterized at the time of this report; therefore, all waste streams must be characterized to ensure that human health and the environment are protected. If more than one phase (gas, liquid, or solid) is present in a waste stream, each phase should be characterized separately.

Although effluent streams were extensively characterized during Demo II, the tests were of short duration and took place in undersized reactors. Consequently, the effluent streams were not characterized under actual or optimized conditions and may not be representative of the effluents that would be produced in full-scale operation.

General Finding 10. Testing, verification, and integration beyond the 1999 demonstration phase will be necessary because the scale-up of a process can present many unexpected challenges, and the ACWA demonstrations were limited in nature.

The PMACWA is planning an EDS testing program (EDS II) for both the Foster Wheeler/Eco Logic/Kvaerner and the AEA technology packages. The EDS II program, which has objectives similar to those of EDS I, will produce engineering design packages and further testing of the two technologies. As a result of the problems and delays during the initial testing and investigation of the Teledyne-Commodore technology package, the PMACWA has dropped SET™ from consideration.

The committee considers the Demo II tests as proof-of-concept tests of the demonstrated unit operations. The critical step of integrating them has not yet been addressed by the technology providers. Therefore, the following general findings and recommendations, all related to integration, were not affected by the Demo II tests.

General Finding 7. None of the proposed technology packages complies completely with the hold-test-release concept for all gaseous effluents (both process and ventilation effluents).

General Finding 8. Hold-test-release of gaseous effluents may not ensure against a release of agent or other hazardous material to the atmosphere. No evidence shows that hold-test-release provides a higher level of safety than current continuous monitoring methods for gaseous streams with low levels of contamination. Furthermore, none of the technologies provides for hold-test-release of effluents from ventilation systems that handle large volumes of gases from contaminated process areas.

General Finding 9. Solid salts will be hazardous waste, either because they are derived from hazardous waste or because they leach heavy metals above the levels allowed by the Resource Conservation and Recovery Act Toxicity Characteristic Leaching Procedure. Stabilization—mixing waste with a reagent or reagents to reduce the leachability of heavy metals—will probably be required before the salts can be sent to a landfill. The potentially high chloride and nitrate content of these salts will make the waste difficult to stabilize, and treatability studies will be necessary to determine a proper stabilization formula.

General Finding 11. Although a comprehensive quantitative risk assessment (QRA), health risk assessment (HRA), and ecological risk assessment (similar to assessments performed for the baseline process) cannot be completed at this stage of process development, these assessments will have to be performed and refined as process development continues.

General Finding 12. The "optimum" system for a particular chemical weapons storage depot might include a combination of unit operations from the technology packages considered in this report.

General Finding 13. Some of the ACWA technology providers propose that some effluent streams be used commercially. New or modified regulations may have to be developed to determine if these effluent streams can be recovered or reused.

General Finding 14. An extraordinary commitment of resources will be necessary to complete the destruction of the assembled chemical weapons stockpile in time to meet the current deadline using any of the ACWA technology packages. This would demand a concerted national effort. It is unlikely that any of the technology packages could meet this deadline.

General Finding 15. The Dialogue process for identifying an alternative technology is likely to reduce the level of public opposition to that technology. The committee believes that the Dialogue has been and continues to be a positive force for public acceptance of alternatives to incineration.

Although the Dialogue process requires a significant commitment of time and resources, it has been a critical component of the ACWA program to date.

General Finding 16. Although the committee did not have access to scientific data on the attributes of a technology that would be most acceptable to the public, input from members of the active publics and previous research indicate that technologies with the following characteristics are likely to stimulate less public opposition:

- minimal emissions, particularly gaseous
- continuous monitoring of effluents to verify that the process is operating as designed (process assurance measurement)
- provisions for representatives of the local community to observe and participate in the process assurance measurement

General Recommendation 1. If a decision is made to move forward with any of the ACWA technology packages, substantial additional testing, verification, and integration should be performed prior to full-scale implementation (see General Finding 10).

General Recommendation 2. The sampling and analysis programs at each phase of development should be carefully reviewed to ensure that the characterization of trace components is as comprehensive as possible to avoid surprises in the implementation of the selected technology (see General Finding 6).

General Recommendation 3. If a decision is made to move forward with any of these technology packages, health and safety evaluations should progress from qualitative assessments to more quantitative assessments as the process design matures. Quantitative (QRA), health (HRA), and ecological risk assessments should be conducted as soon as is practical. Early initiation of these assessments will allow findings to be implemented with minimal cost and schedule impact. (See General Finding 11.)

General Recommendation 4. Any of these technology packages, or any component of these technology packages, should be selected on a site-specific basis. (See General Finding 12.)

General Recommendation 7. The Department of Defense should continue to support the Dialogue throughout the current ACWA program and should seriously consider the participation of the Dialogue in follow-on programs.

SUPPLEMENTAL GENERAL FINDINGS

In the statement of task for this report, the committee was asked to determine if any of the technology packages chosen for demonstrations "have reached a technology readiness level sufficient to proceed with implementation of a pilot-scale." The ACW II committee's evaluations of the maturity of each unit operation are described in this report. Table 5-1 summarizes the committee's assessments. However, the AEA, Eco Logic, and General Atomics technology packages were chosen by the PMACWA to undergo engineering design studies for the destruction of the assembled chemical weapons at the Blue Grass Army depot. This decision was made by the PMACWA prior to the issuance of this NRC report. In view of the fact that testing in these areas is ongoing, the committee decided to cut off its fact-finding efforts for input to this report as of March 30, 2001. This cutoff was necessary in order to give the sponsor the information it needed in a timely fashion.

General Finding DII 1. The demonstration tests were not operated long enough to show reliability in long-term operation. The PMACWA's Demo II tests were required to be of the same duration as the Demo I tests. The technology providers had neither the time nor the resources for extensive systemization (preoperational testing) in Demo II. Consequently, these tests were simply proof-of-concept demonstrations that indicate whether or not a particular unit operation (with more development) might be applicable to the disposal of assembled chemical munitions.

General Finding DII 2. The AEA technology package is a very complex, immature chemical processing system. Several new unit operations required to address problems revealed in the Demo II tests will significantly increase the complexity of an integrated processing system and extend the time required for its development.

General Finding DII 3. The demonstrated components of the FW/EL/K technology package are ready to progress to the EDS phase. However, certain key units were not tested (or the results were inconclusive). Additional testing will be needed to verify the ability of the transpiring-wall technology to minimize corrosion; the testing should be carried out in parallel with development of an engineering design.

General Finding DII 4. Because of fire and safety problems, the basic process for the Teledyne-Commodore technology was not tested in Demo II. The Army decided against going forward because the Demo II goals could not be met in time. As a result, the committee had no technical basis on which to evaluate the process any further.

General Finding DII 5. As was true for Demo I, none of the unit operations tested in Demo II has been integrated into a complete system. The lack of integration is a major concern and a significant obstacle to full-scale implementation.

SUPPLEMENTAL GENERAL RECOMMENDATIONS

General Recommendation DII 1. Further development of the Teledyne-Commodore technology package for the destruction of assembled chemical weapons should not be pursued under the ACWA program.

TABLE 5-1 Summary Evaluation of the Maturity of Demo II Unit Operations and Processes

Technology Provider/Unit Operation or Process	Hydrolysates			Agent Munitions			Other
	VX/GB	HD	Energetics	VX/GB	HD	Energetics	
AEA							
SILVER II™[a]				C	C	C	
Solid/liquid waste treatment				C	C	C	
Gaseous waste treatment				D	D	D	
Foster Wheeler/Eco Logic/Kvaerner							
TW-SCWO	B	B	C				
GPCR™				B	B	B	B[b,c]
Teledyne-Commodore							
Ammonia fluid jet cutting and washout system				D	D	E	
SET™				D	D	D	C[b]
Persulfate oxidation (agent)				D	D	D	
Peroxide oxidation (energetics)				D	D	D	
Metals parts and dunnage shredding							A[b,c]

NOTE: Environmental and safety issues were considered in assigning maturity categorizations. Schedule and cost issues were not considered. The letter designations are defined as follows (a blank space indicates that categorization was not applicable for that material): A, demonstration provides sufficient information to justify moving forward to full-scale design with reasonable probability of success; B, demonstration provides sufficient information to justify moving forward to the pilot stage with reasonable probability of success; C, demonstration indicates that unit operation or process requires additional refinement and additional demonstration before moving forward to pilot stage; D, not demonstrated, and more R&D is required; and E, demonstrated unit operation or process is inappropriate for treatment.

[a] Includes integrated gas polishing system to support demonstration.

[b] Dunnage.

[c] Metal parts.

General Recommendation DII 2. Before the AEA technology proceeds to the EDS phase, extensive testing should be performed on the SILVER II™ process, including all the new unit operations that are being proposed to address the shortcomings identified in Demo II results.

General Recommendation DII 3. For the FW/EL/K technology package, additional testing should be performed in the EDS phase to complete GPCR™ off-gas characterization and demonstrate long-term operation of the modified TW-SWCO unit.

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Appendixes

Appendix A

Site Visits and Meetings

COMMITTEE MEETING 1, JUNE 8–9, 2000

Vitali's Restaurant
Edgewood, Maryland

NRC Participants

Committee chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Joan B. Berkowitz, David S. Kosson, Sheldon E. Isakoff, Frederick J. Krambeck, John A. Merson, William R. Rhyne, William R. Seeker, and Leo Weitzman. NRC staff members: Patricia P. Paulette, Bruce A. Braun, Harrison T. Pannella, and Jacqueline Campbell-Johnson.

Objectives

Complete administrative actions, including introductions and composition/balance discussions for committee members, and committee administrative support methodology; receive DoD briefings on the ACWA program concerning Demonstration II and status of Engineering Design Studies; tour and inspect the four test sites at Aberdeen Proving Ground (APG) or the APG Edgewood Area; develop specific committee assignments for future activities; discuss concepts for planned reports and strategy; determine location and date for the next committee meeting.

SITE VISIT 1, JUNE 9, 2000 (IN CONJUNCTION WITH MEETING 1)

Engineering Test Facilities
Edgewood, Maryland, and Aberdeen, Maryland

NRC Participants

Committee chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Joan B. Berkowitz, Sheldon E. Isakoff, Frederick J. Krambeck, John A. Merson, William R. Rhyne, William R. Seeker, and Leo Weitzman. NRC staff members: Patricia P. Paulette, Bruce A. Braun, Harrison T. Pannella, and Jacqueline Campbell-Johnson.

Objectives

Visit the following Demonstration II test units: AEA 12 kW SILVER II™ test unit for energetics; AEA 2kW SILVER II™ test unit for agent; and Eco Logic GPCR™ test unit.

Visit the following engineering design study test units: Parsons/Honeywell immobilized-cell bioreactor test unit.

SITE VISIT 2, JUNE 20–21, 2000

Engineering Test Facilities
Dugway Proving Ground and Deseret Chemical Depot, Utah

NRC Participants

Committee chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Willard C. Gekler, Sheldon E. Isakoff, Hank C. Jenkins-Smith, John A. Merson, Stanley I. Sandler, William R. Rhyne, and Leo Weitzman. NRC staff member: Harrison T. Pannella.

Objectives

Visit the following Demonstration II test units: Teledyne-Commodore SET™ test unit for energetics, SET™ test unit for agent, and fluid jet cutting test unit; Foster Wheeler SCWO test unit. Visit the following Engineering Design Study test units: General Atomics SCWO test unit and dunnage shredder/hydropulping system. Tour the baseline incineration-based Tooele Chemical Agent Disposal Facility and the adjacent chemical stockpile storage area.

COMMITTEE MEETING 2, AUGUST 8–9, 2000

National Academy of Sciences
Washington, D.C.

NRC Participants

Committee chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Joan B. Berkowitz, Ruth M. Doherty, Frederick J. Krambeck, David S. Kosson, John A. Merson, William R. Rhyne, Stanley I. Sandler, William R. Seeker, and Leo Weitzman. NRC staff members: Patricia P. Paulette, Bruce A. Braun, Harrison T. Pannella, Jacqueline Campbell-Johnson, and Gwen Roby.

Objectives

Receive briefings from the ACWA technical staff, the Citizens Advisory Technical Team, and technology providers (Foster Wheeler, Eco Logic, AEA Technology Engineering Services, Inc., Teledyne-Commodore, and Parsons/Honeywell). Discuss concept drafts of EDSs and Demo II reports; develop writing assignments, complete committee composition and balance, and finalize locations and dates for future committee meetings and site visits.

SITE VISIT 3, SEPTEMBER 27–29, 2000

Arthur D. Little, Inc.
Cambridge, Massachusetts

NRC Participants

Committee chair: Robert A. Beaudet. Committee member: Joan B. Berkowitz. NRC staff member: Patricia P. Paulette.

Objectives

Receive briefings by technology providers participating in the Demo II testing programs. Presentations concern the scope and methodology for each technology testing program, as well as initial testing results. Receive briefings by technology providers concerning the type of data and their format that will be provided to the PMACWA as a result of Demo II testing.

COMMITTEE MEETING 3, OCTOBER 19–20, 2000

J. Erik Jonsson Woods Hole Center of the National Academies
Woods Hole, Massachusetts

NRC Participants

Committee chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Joan B. Berkowitz, Ruth M. Doherty, Willard C. Gekler, Sheldon E. Isakoff, David S. Kosson, Frederick J. Krambeck, John A. Merson, William R. Rhyne, William R. Seeker, and Leo Weitzman. NRC staff members: Patricia P. Paulette, Bruce A. Braun, Harrison T. Pannella, and Chris Jones.

Objectives

Complete administrative actions, including introductions and discussion of committee composition and balance. Receive updates from the PMACWA technical team and associates concerning Demo II, Engineering Design, and energetics testing. Discuss initial findings and recommendations for the EDS Pueblo and Demo II reports.

SITE VISIT 4, OCTOBER 30, 2000

Holston Army Ammunition Plant
Kingston, Tennessee

NRC Participants

Committee members: Ruth M. Doherty and William R. Rhyne.

Objectives

Attend a review of the energetics hydrolysis testing planned for Holston Army Ammunition Plant. Tour facility and observe operations. Holston is a major site for hydrolysis testing of many different types of energetic materials.

COMMITTEE MEETING 4, DECEMBER 14–15, 2000

Grand Hyatt Washington Hotel
Washington, D.C.

NRC Participants

Committee chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Joan B. Berkowitz, Ruth M. Doherty, Willard C. Gekler, Sheldon E. Isakoff, Hank C. Jenkins-Smith, David S. Kosson, Frederick J. Krambeck, John A. Merson, William R. Rhyne, Stanley I. Sandler, William R.

Seeker, and Leo Weitzman. NRC staff members: Patricia P. Paulette, Harrison T. Pannella, and Jacqueline Campbell-Johnson.

Objectives

Report development. Review and revise current versions of EDS-Pueblo and Demo II reports. Devise path forward for first full message drafts of each report.

SITE VISIT 5, JANUARY 4, 2001

Holston Army Ammunition Plant
Kingston, Tennessee

NRC Participants

Committee members: Ruth M. Doherty, John A. Merson, and William R. Rhyne.

Objectives

Attend a review meeting on energetics hydrolysis testing at Holston and the results of this testing to date. Gather information on the scientific and engineering issues revealed during the test procedures.

COMMITTEE MEETING 5, FEBRUARY 8–9, 2001

National Academy of Sciences
Washington, D.C.

NRC Participants

Committee chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Joan B. Berkowitz, Ruth M. Doherty, Willard C. Gekler, David S. Kosson, Frederick J. Krambeck, John A. Merson, William R. Rhyne, Stanley I. Sandler, William R. Seeker, and Leo Weitzman. NRC staff members: Patricia P. Paulette, Bruce A. Braun, Harrison T. Pannella, and Jacqueline Campbell-Johnson.

Objectives

Receive briefings from the PMACWA technical staff concerning the status of EDS I, EDS II, and Demo II program activities and other presentations from the energetics hydrolysis testing group at Picatinny Arsenal. Review and revise reports; define steps to EDS I report concurrence draft; set goals for interim activities and for the next meeting.

SITE VISIT 6, JANUARY 25–26, 2001

ACWA Dialogue Meeting
Lexington, Kentucky

NRC Participants

Committee member: William R. Rhyne. NRC staff member: Patricia P. Paulette.

Objectives

Present an update of committee activities. Participate in and observe the Dialogue meeting.

SITE VISIT 7, MARCH 5–8, 2000

Arthur D. Little, Inc.
Cambridge, Massachusetts

NRC Participants

Committee members: Ruth M. Doherty, William R. Rhyne, and Stanley I. Sandler. NRC staff member: Patricia P. Paulette.

Objectives

Receive technical briefings from AEA and Foster Wheeler/Eco Logic/Kvaerner on their proposed EDS packages and testing schedules for EDS II. Receive briefings from ACWA technical team concerning EDS II schedules and reviews.

INFORMATION-GATHERING ACTIVITY, MARCH 11–14, 2001

National Association of Corrosion Engineers Symposium
Houston, Texas

NRC Participant

NRC Staff: Patricia P. Paulette.

Objectives

Attend National Association of Corrosion Engineers symposium on SCWO technology and materials of construction and corrosion reactions. Arrange to obtain scientific briefing packages and technical papers for distribution to committee members.

COMMITTEE MEETING 6, MARCH 26–27, 2001

Arnold and Mabel Beckman Center
National Academy of Sciences
Irvine, California

NRC Participants

Committee chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Joan B. Berkowitz, Ruth M. Doherty, Willard C. Gekler, Sheldon E. Isakoff, Frederick J. Krambeck, John A. Merson, William R. Rhyne, Stanley I. Sandler, William R. Seeker, and Leo Weitzman. NRC staff members: Bruce A. Braun, Patricia P. Paulette, Harrison T. Pannella, Gwen Roby, and William E. Campbell.

Objectives

Closed meeting for report development. Complete concurrence draft for EDS I Pueblo report and preconcurrence draft for Demo II report. Schedule next meeting and discuss items for the agenda.

Appendix B

Biographical Sketches of Committee Members

Robert A. Beaudet, chair, received his Ph.D. in physical chemistry from Harvard University in 1962. From 1961 to 1962, he was a U.S. Army officer and served at the Jet Propulsion Laboratory as a research scientist. He joined the faculty of the University of Southern California in 1962 as an assistant professor and was chair of the Chemistry Department from 1976 to 1979. He has also served on Department of Defense committees addressing chemical warfare agents in both offensive and defensive scenarios. He was chair of an Army Science Board committee that addressed chemical detection and trace-gas analysis and chair of an Air Force technical conference on chemical warfare decontamination and protection. He has served on two National Research Council (NRC) studies: chemical and biological sensor technologies and energetic materials and technologies. Most of his career has been devoted to research in molecular structure and molecular spectroscopy. Dr. Beaudet previously served as a member of the Board on Army Science and Technology (BAST) and as a BAST liaison to the Committee on Review and Evaluation of the Army Chemical Stockpile Disposal Program (Stockpile Committee), a standing NRC committee. He is currently a member of the NRC Committee on Review of the Non-Stockpile Chemical Materiel Disposal Program. Dr. Beaudet is the author or coauthor of more than 100 technical reports and papers.

Richard J. Ayen received his Ph.D. in chemical engineering from the University of Illinois. Dr. Ayen is a former vice president of technology for Waste Management, Inc., and is now an independent consultant. He has extensive experience in the evaluation and development of new technologies for the treatment of hazardous waste. Dr. Ayen managed all aspects of the Waste Management Clemson Technical Center, including treatability studies and technology demonstrations for hazardous and radioactive waste. He has published extensively in his fields of interest and is a member of the NRC Committee on Review of the Non-Stockpile Chemical Materiel Disposal Program.

Joan B. Berkowitz, who graduated from the University of Illinois with a Ph.D. in physical chemistry, is currently managing director of Farkas Berkowitz and Company. Her areas of expertise include environmental and hazardous waste management, available technologies for the cleanup of contaminated soils and groundwater, and physical and electrochemistry. She has contributed to several studies by the Environmental Protection Agency (EPA), been a consultant on remediation techniques, and assessed various destruction technologies. Dr. Berkowitz has written numerous publications on hazardous waste treatment and environmental subjects. She is currently a member of the NRC Committee on Review of the Non-Stockpile Chemical Materiel Disposal Program.

Ruth M. Doherty received a Ph.D. in physical chemistry from the University of Maryland. She is currently technical advisor for the Energetic Materials Research and Technology Department, Naval Surface Warfare Center, Indian Head, Maryland. Since 1983, she has coauthored about 60 publications on physical chemistry. In the past 6 years, Dr. Doherty has given 20 presentations on various aspects of the science and technology of explosives. In 1998–1999, she delivered a series of lectures on explosives technology for members of the Office of Naval Intelligence. For more than 15 years, she has been involved in research and development of energetics materials and explosives at the Naval Surface Warfare Center.

Willard C. Gekler graduated from the Colorado School of Mines with a B.S. in petroleum refining engineering and pursued graduate study in nuclear engineering at the University of California in Los Angeles. Mr. Gekler is currently an independent consultant working for his previous employer, EQE International, Inc. His extensive experience includes membership on the NRC ACWA committee and on the expert panel reviewing the quantitative risk assessments and safety analyses of hazardous materials handling, storage, and waste treatment systems for the Anniston and Umatilla chemical disposal facilities. His expertise is in hazard evaluation, quantitative risk analyses, reliability assessment, and database development for risk and reliability. Mr. Gekler is a certified reliability engineer and a member of the Society for Risk Analysis (SRA) and the American Nuclear Society. He is the author or coauthor of numerous publications.

Sheldon E. Isakoff, who received his Ph.D. in chemical engineering from Columbia University, is the retired director of the Engineering R&D Division of E.I. du Pont de Nemours and Company. His experience includes the management of technology, the direction of research and development, market assessment and development, process scale-up, commercial introduction, and leadership of personnel. His areas of expertise also include materials science and engineering and the development and application of new materials for industrial and consumer markets. Dr. Isakoff is a fellow and past president of the American Institute of Chemical Engineering and a former director of its materials engineering and sciences division. Dr. Isakoff was elected to the National Academy of Engineering in 1980 and has served on several NRC committees.

Hank C. Jenkins-Smith is a professor of Public Policy at the George H. W. Bush School of Government and Public Service at Texas A&M University in College Station, Texas. He holds the Joe R. and Teresa Lozano Long Chair of Business and Government at the Bush School. He was previously a professor of political science and director of the Institute for Public Policy at the University of New Mexico. Professor Jenkins-Smith's areas of research include science and technology policy, environmental policy, public perceptions of environmental and technical risks, and national security policy. Professor Jenkins-Smith has written books on the public policy process and policy analysis and has served on a number of committees for the National Research Council.

David S. Kosson 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. He is chairman and professor of the Department of Civil and Environmental Engineering and professor of chemical engineering at Vanderbilt University and a former 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. Dr. Kosson served on the NRC Committee on Review and Evaluation of the Army Stockpile Disposal Program for 7 years, the final 2 years as chair. As a member of the NRC Committee on Alternative Chemical Demilitarization Technologies and the Panel on Review and Evaluation of Alternative Chemical Disposal Technologies, he contributed to the Army's decision to use alternative methods of destruction at both the Aberdeen and Newport facilities. Dr. Kosson is well known for his expertise in bioremediation.

Frederick J. Krambeck received his Ph.D in chemical engineering from the City University of New York. He is a senior consultant for ExxonMobil Research and Engineering Company. His expertise includes research and development (R&D) in petroleum refining, including process and reactor design and development, chemical reaction engineering, on-line and off-line optimization, modeling, and R&D project management. He is also experienced in technology strategy considerations for greenhouse gas stabilization. Dr. Krambeck was elected to the National Academy of Engineering in 1999 and is a fellow and member of the Board of Directors of the American Institute of Chemical Engineers. He has authored or coauthored over 25 patents and 40 publications.

John A. Merson received a B.S. and M.S. in chemical engineering from the University of New Mexico and a Ph.D. in chemical engineering from Arizona State University. His areas of expertise include research, development, and application of energetic materials and components in the nuclear weapons stockpile. Dr. Merson is the department manager of the Explosive Subsystems and Materials Department at Sandia National Laboratories, which designs, develops, and characterizes explosive, propellant, and pyrotechnic components and subsystems to meet specific needs. Dr. Merson is a member of the American Institute of Chemical Engineers.

William R. Rhyne received a B.S. in nuclear engineering from the University of Tennessee and an M.S. and D.Sc. in nuclear engineering from the University of Virginia and is cofounder and director of H&R Technical Associates, Inc. Dr. Rhyne has extensive experience in risk and safety analysis associated with nuclear and chemical processes and with the transport of hazardous nuclear materials and chemicals. From 1984 to 1987, he was the project manager and principal investigator for a probabilistic accident analysis of transporting obsolete chemical munitions. Dr. Rhyne is the author or coauthor of more than 40 publications and reports on nuclear and chemical safety and risk analysis, including *Hazardous Materials Transportation Risk Analysis: Quantitative Approaches for Truck and Train.* He is a member of the NRC Transportation Research Board Hazardous Materials Committee, the Society for Risk Assessment, the American Nuclear Society, and the American Institute of Chemical Engineers.

Stanley I. Sandler, who received his Ph.D. in chemical engineering from the University of Minnesota, is currently the Henry Belin du Pont Chair and director of the Center for

Molecular and Engineering Thermodynamics at the University of Delaware. His extensive research interests include applied thermodynamics and phase equilibrium, environmental engineering, and separations and purification. Dr. Sandler is a recipient of the Warren K. Lewis Award from the American Institute of Chemical Engineers and the Inaugural E.A. Mason Memorial Lecturer Award from Brown University. He is a member of the National Academy of Engineering and has published more than 250 technical articles in recognized journals and conference proceedings.

William R. Seeker received his Ph.D. in engineering (nuclear and chemical) from Kansas State University. He is senior vice president and member of the Board of Directors of Energy and Environmental Research Corporation, a wholly owned subsidiary of General Electric Company. He has extensive experience in the use of thermal treatment technologies, environmental control systems for managing hazardous waste, and air pollution control. He is a member of the Executive Committee of the Environmental Protection Agency Science Advisory Board and the author of more than 150 technical papers on various aspects of technology and the environment.

Leo Weitzman received his Ph.D. in chemical engineering from Purdue University. He is a consultant with 28 years of experience in the development, design, permitting, and operation of equipment and facilities for the treatment of hazardous wastes and remediation debris. Dr. Weitzman has extensive experience in the disposal of hazardous waste and contaminated materials by thermal treatment, chemical reaction, solvent extraction, biological treatment, and stabilization. He has published more than 40 technical papers.