Analysis of Engineering Design Studies for Demilitarization of Assembled Chemical Weapons at Blue Grass Army Depot

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

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ANALYSIS OF ENGINEERING DESIGN STUDIES FOR DEMILITARIZATION OF ASSEMBLED CHEMICAL WEAPONS AT BLUE GRASS ARMY DEPOT

Committee on Review and Evaluation of Alternative Technologies for
Demilitarization of Assembled Chemical Weapons: Phase II

Board on Army Science and Technology

Division on Engineering and Physical Sciences

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Preface

The United States has been destroying its chemical munitions for well over a decade. Initially, the U.S. Army, with 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 have opposed incineration on the grounds that it is impossible to determine the exact nature of the effluents escaping from the stacks. Although the Army has continued to pursue incineration at four of the eight storage sites in the continental United States, in response to growing public opposition to incineration in Maryland and Indiana and a 1996 report by the NRC, *Review and Evaluation of Alternative Chemical Disposal Technologies*, the Army is developing alternative processes to neutralize chemical agents by hydrolysis. These processes will be used to destroy the VX nerve agent at Newport, Indiana, and the mustard agent at Aberdeen, Maryland, both of which are stored in bulk one-ton containers.

In 1996, persuaded by the public opposition in Lexington, Kentucky, and Pueblo, Colorado, Congress enacted Public Law 104-201, which instructed the 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." The Army established a Program Manager for Assembled Chemical Weapons Assessment (PMACWA) to respond to Congress. In Public Law 104-208, PMACWA was required to "identify and demonstrate not less than two alternatives to the baseline incineration process for the demilitarization of assembled chemical munitions." Following an elaborate selection process and the demonstration of six technologies, PMACWA selected three as candidates for destroying the weapons at Blue Grass Army Depot. The three technologies—AEA SILVER II™, Eco Logic, and General Atomics Total Solution—have since progressed to the engineering design phase of DoD's Assembled Chemical Weapons Assessment (ACWA) program. This report evaluates the engineering design studies for these three candidates.

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 issue before the committee was not whether incineration is an adequate technology but, given that some citizens are strongly opposed to incineration, whether other disposal methods acceptable to the stakeholders could be used. A second committee, the Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: Phase II (ACW II Committee), was established in the spring of

2000 for the engineering design phase of the ACWA program.

This report provides an independent technical evaluation of the engineering-design packages of the three candidate processes being considered for use at the Blue Grass Army Depot. A separate chapter is devoted to each technology package that addresses all aspects of demilitarization, from disassembly of the weapons to disposal of the waste streams. Specific recommendations related to the technology are found in these chapters but are not repeated in the Executive Summary. The committee's evaluation is expected to contribute to DoD's record of decision (ROD) for the selection of a technology for the Lexington Blue Grass site. The ROD was scheduled to be released in September 2002. Therefore, to be of value in the selection process, this report had to be published by August 2002. Unfortunately, not all of the tests associated with the three packages were completed at the time that data gathering for this report was terminated to meet the mid-July deadline.

This report documents the ACW II Committee's completion of its final task. Since 1997, the ACW I and ACW II Committees have produced four full reports and one letter report (NRC, 1999, 2000a, 2001a, 2001b, 2001c). Usually, NRC study committees are constituted to produce only a single report during their tenure. The aim of the ACW I and ACW II Committees has been to provide constructive advice to PMACWA. Questions that committee members asked at meetings attended by the ACWA team and its support contractors have supplemented the advice contained in the reports.

The environment in which the ACW II Committee works has changed since the events of September 11, 2001. Destruction of the stockpile has become more urgent, and this has made the general findings in this report more pertinent than ever. The committee confirms its belief that technologies using hydrolysis to destroy both agent and energetics are now mature, safe, straightforward, and effective. Moreover, the products from the hydrolysis no longer exhibit the acute toxicity of the agents and could be treated at commercial toxic or hazardous waste facilities if the states permit it. A precedent was established at Aberdeen Proving Ground: The mustard in bulk containers is being destroyed by hydrolysis, using mostly the facilities that already exist on site and sending the products to a commercial treatment facility.

I wish to express my gratitude to the members of the ACW II Committee, all of whom served as volunteers and most of whom served with me on the ACW I Committee. They have all given unselfishly of their time and knowledge. These technology packages are complex and voluminous, and it required extensive work to properly evaluate them. The committee members' areas of expertise include chemical processing, biological remediation, environmental regulations and permitting, energetic materials, public involvement, and public acceptance. Each member attended plenary meetings, visited the headquarters of technology providers and test sites, observed design-review sessions, and studied the extensive literature, including engineering charts and diagrams, given by the technology providers. Fortunately for me, the members are all brighter and more experienced in chemical processing than am I, a physical chemist.

The committee recognizes and appreciates the extensive support of the Army ACWA team and the committee interactions with stakeholders and the Dialogue group, particularly the four members of the Dialogue known as the Citizens Advisory Technical Team (CATT). Members of the CATT attended all open meetings of the committee and shared information and their views with us. I believe our relationship with the sponsor, PMACWA, and his team and support contractors has been effective and constructive and that the committee has been given the best available information to conduct this evaluation of the three technologies.

The committee also appreciates the openness and cordiality of the representatives of the technology providers. They and the Army provided it with early drafts of test reports and other documentation to facilitate the writing of this report while work was still in progress.

A study like this always requires extensive logistic support, and the committee is indebted to the NRC staff for their assistance. I would like to acknowledge particularly the close working relationship I had with the NRC study director for this study, Patricia P. Paulette. Working as a team in leading this study, she and I spoke on the phone daily and e-mailed each other incessantly. Invaluable contributions were also made by Harrison T. Pannella, who took extensive notes at all of our meetings, edited draft text for the report, and provided suggestions for organizing the report. In addition, Gwen Roby provided the logistic support that freed us to concentrate on our task. Assistance was also provided by James C. Myska. The report was edited by Liz Fikre of the Division on Engineering and Physical Sciences. I am also indebted to colleagues in the Chemistry Department at the University of Southern California, who willingly substituted for me in 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

Acknowledgment of Reviewers

This report has been reviewed by individuals chosen for their diverse perspectives and technical expertise, in accordance with procedures approved by the National Research Council's Report Review Committee. The purpose of this independent review is to provide candid and critical comments that will assist the 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:

Ronald Ballinger, Massachusetts Institute of Technology,
Steven Konkel, Eastern Kentucky University,
Richard S. Magee, Carmagen Consulting,
Walter May (NAE), Consultant, Urbana, Illinois,
Digby McDonald, Pennsylvania State University,
Ray McGuire, Consultant, Livermore, California,
George Parshall (NAS), Chemical Science (retired), and
Bruce E. Watkins, Lawrence Livermore National Laboratory.

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 Hyla S. Napadensky (NAE), Napadersky Energetics, Inc. (retired). Appointed by the National Research Council, she 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, Abbreviations, and Chemical Symbols

ACAMS	automatic continuous air monitoring system
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
ACWA	Assembled Chemical Weapons Assessment
Ag^{2+}	silver ion having a valence of 2
AgCl	silver chloride
a-HAX	solution containing potassium hydroxide and humic acid
AHSV	agent hydrolysate surge vessel
ANR	agent neutralization reactor
AQS	agent quantification system
ARDEC	Armament Research, Development and Engineering Center
BIF	boiler and industrial furnace
BPCS	basic process control system
BWM	burster washout machine
CAMDS	chemical agent munitions disposal system
CATOX	catalytic oxidation
CEES	chloroethyl ethyl sulfide
CEMS	continuous emission monitoring system
CO	carbon monoxide
CO_2	carbon dioxide
COINS	continuous indexing neutralization system
Composition B	an energetic material that contains (nominally) 59.5 percent RDX, 39.5 percent TNT, and 1.0 percent wax
CSDP	Chemical Stockpile Disposal Program
CST	continuous steam treater
CWC	Chemical Weapons Convention
DAAMS	depot area air monitoring system
Demo I	Demonstration I (demonstration testing of the initial three technologies selected for the first phase of ACWA technology testing)

Demo II	Demonstration II (demonstration testing of the second set 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
DSHS	dunnage shredding and handling system
DTS	dunnage treatment system
ECR	explosion containment room
EDP	engineering design package
EDS	engineering design study
EPA	Environmental Protection Agency
EPDM	ethylene propylene dimer
EPDR	energetics/propellant deactivation reactor
ERD	energetics rotary deactivator
ERH	energetics rotary hydrolyzer
ESS	emergency shutdown system
EST	engineering-scale test
GATS	General Atomics Total Solution (technology package)
GB	a nerve agent
GC/MS	gas chromatography/mass spectrometry
GPCR	gas-phase chemical reduction
H	undistilled mustard agent
H_2	hydrogen
HAAP	Holston Army Ammunition Plant
HD	distilled mustard agent
HDC	heated discharge conveyor
HEPA	high-efficiency particulate air (filter)
HF	hydrofluoric acid
HMX	cyclotetramethylene-tetranitramine (an energetic material)
HNO_3	nitric acid
HPLC	high-performance liquid chromatography
HRA	health risk assessment
HT	a mixture of H and T mustard agents
HVAC	heating, ventilating, and air-conditioning
ICB	immobilized-cell bioreactor
ICI	Imperial Chemical Industries
IITRI	Illinois Institute of Technology Research Institute
IMPA	isopropyl methylphosphonic acid
IRS	impurities removal system
JACADS	Johnston Atoll Chemical Agent Disposal System
KOH	potassium hydroxide
kPa	kilopascal (unit of pressure; 6.895 kPa equals 1 pound per square inch)
kPag	kilopascals gauge

kW	kilowatt
LANL	Los Alamos National Laboratory
LMIDS	Lockheed Martin Integrated Demilitarization System
M	molar concentration
M28	energetic material used for propulsion of certain assembled chemical weapons
MACT	maximum achievable control technology
MAV	modified ammunition van
MDB	munitions demilitarization building
MDM	munitions demilitarization machine
MEO	mediated electrochemical oxidation
MPA	methylphosphonic acid
MPa	megapascals
MPT	metal parts treater
MSB	munitions storage building
N_2	nitrogen
N_2O	nitrous oxide
NEPA	National Environmental Policy Act
NO_x	nitrogen oxides
NRC	National Research Council
NSWC	Naval Surface Warfare Center
O_2	oxygen
OB/OD	open burn/open detonation
PA	picric acid
PCP	pentachlorophenol
PCR	propellant collection reactor
PFA	perfluoroalkoxy
PGB	product gas burner
PHA	preliminary hazards analysis
PID	proportional integral differential controller
PLC	programmable logic control
PMACWA	Program Manager for Assembled Chemical Weapons Assessment
PMD	projectile mortar demilitarization (machine)
POTW	publicly owned treatment works
ppm	parts per million
PRH	projectile rotary hydrolyzer
PRR	propellant removal room
psi	pounds per square inch
psig	pounds per square inch gauge
PTFE	polytetrafluoroethylene (Teflon)
PVDF	polyvinylidene fluoride
PWS	projectile washout system
QRA	quantitative risk assessment
R3	resource, recovery, and recycling (process)
R&D	research and development

RAAP	Radford Army Ammunition Plant
RCRA	Resource Conservation and Recovery Act
RDM	rocket dismantling machine
RDX	cyclotrimethylenetrinitramine (an energetic material)
RFP	request for proposals
ROD	record of decision
RWM	rotary washout machine
scfm	standard cubic feet per minute
SCWO	supercritical water oxidation
SET™	solvated electron technology
SILVER II™	electrochemical oxidation using Ag^{2+} in nitric acid
SO_x	sulfur oxides
SO_2	sulfur dioxide
SVOC	semivolatile organic compound
TACOM	Tank-Automotive and Armaments Command
TBA	tributylamine
TCLP	toxicity characteristic leaching procedure
THC	total hydrocarbons
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
UPA	unpack area
VOC	volatile organic compound
VX	a nerve agent
WHEAT	water hydrolysis of explosives and agent technology
WMDM	WHEAT multipurpose demilitarization machine
WPMD	WHEAT projectile/mortar disassembly (machine)
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, 8-hour, time-weighted average limit for worker exposure. The level for mustard agent is 3.0 mg/m^3 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	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.

Executive Summary

The Program Manager for Assembled Chemical Weapons Assessment (PMACWA) of the Department of Defense (DoD) asked the National Research Council (NRC) to assess the engineering design studies (EDSs) developed by AEA Technologies, Eco Logic, and General Atomics for a chemical demilitarization facility to completely dispose of the assembled chemical weapons stored at the Blue Grass Army Depot in Richmond, Kentucky. The results of the committee's scientific and technical assessment of the technology packages are presented in this report. This evaluation will support the selection by DoD of a technology for destruction of the assembled chemical weapons at Blue Grass. The record of decision (ROD), which finalizes the technology choice, is expected early in 2003. The committee evaluated the engineering design packages (EDPs) proposed by the technology providers, as well as results of experimental studies that were performed to support the designs of unit operations in the EDP. A significant part of this testing program involved expanding the technology base for the hydrolysis of energetic materials associated with assembled chemical weapons. (In its original report (NRC, 1999), the ACW I Committee had expressed concern about the hydrolysis process for destroying energetics.)

The present study was conducted as the experimental tests were still in progress. Some supporting unit operations tests were not completed in time for the committee to incorporate the results into its evaluation. In those cases, the committee identified and discussed potential problems associated with the operations. Based on its expertise and aggressive data-gathering activities, the committee was able to conduct a comprehensive review of the test data that had been completed as well as the overall designs in the EDPs.

BACKGROUND

The U.S. Army is in the process of destroying the country's stockpile of aging chemical weapons, stored at eight locations in the continental United States and on Johnston Atoll in the Pacific Ocean. The deadline for completing the destruction of these weapons, as specified by the Chemical Weapons Convention (CWC) international treaty, is April 29, 2007. Originally, the Army selected incineration as the preferred baseline destruction technology, and it currently operates two incineration facilities—one on Johnston Atoll and one at the Deseret Chemical Depot near Tooele, Utah. The Johnston Atoll Chemical Agent Disposal System (JACADS) completed destruction of the stockpile on Johnston Island in late 2000, and closure of the facility is under way.[1] Similar baseline incineration system facilities were planned for all of the remaining storage sites. However, incineration has met with public and political opposition. In response to this opposition, neutralization processes (based on the hydrolysis of chemical agent using either water or sodium hydroxide solution) were developed to destroy the chemical agents stored in bulk containers at Aberdeen, Maryland, and Newport, Indiana. For the five remaining sites in the continental United States, where munitions containing both chemical agent and energetic materials (i.e., assembled chemical weapons) are stored, incineration continued to be the planned approach for destruction. At three of these sites—Umatilla, Oregon; Pine Bluff, Arkansas; and Anniston, Alabama—baseline incineration systems are being constructed and will soon be ready for processing the chemical weapons.

In late 1996, in the face of public opposition, Congress become involved and enacted Public Law 104-201. This law instructed 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."

Another law, Public Law 104-208, required the Program Manager for Assembled Chemical Weapons Assessment (PMACWA) to "identify and demonstrate not less than two alternatives to the baseline incineration process for the demilitarization of assembled chemical munitions." In addition, the law prohibited any obligation of funds for the construction of incineration facilities at the two remaining storage sites where no disposal facilities had yet been built—Lexington/Blue Grass, Kentucky, and Pueblo, Colorado. This prohibition was to remain in effect until the demonstrations were completed and Congress had assessed the results submitted to it by DoD.

As a result of Public Laws 104-201 and 104-208, DoD created the Assembled Chemical Weapons Assessment (ACWA) program to select and evaluate technologies that would be appropriate for destroying the stockpiles at Pueblo Chemical Depot and Blue Grass Army Depot (NRC, 1999). Seven technology packages were considered in the initial round of the selection process, but one was rapidly rejected. Three of the six remaining underwent the first round of demonstration testing (Demo I). Two of the technologies tested in Demo I were selected as candidates for the destruction of chemical weapons at the Pueblo Chemical Depot: General Atomics' Total Solution (GATS) process and Parsons/Honeywell's Water Hydrolysis of Explosives and Agent Technology (WHEAT) process. The PMACWA subsequently initiated EDSs for the two technologies that had successfully completed Demo I.

Congress also directed the PMACWA to demonstrate the remaining three technology packages that had not been selected for Demo I: AEA's SILVER II™ process using mediated electrochemical oxidation; Eco Logic's process using hydrolysis followed by transpiring-wall supercritical water oxidation and gas-phase chemical reduction (GPCR™); and Teledyne Commodore's solvated electron technology (SET) using sodium in anhydrous ammonia followed by persulfate oxidation. After the Demo II results were in, PMACWA selected the AEA and the Eco Logic technology packages to proceed to the engineering design studies stage (EDS II). Accordingly, the AEA and the Eco Logic technology packages, along with GATS, have become candidates for the destruction of the chemical weapons at the Blue Grass Army Depot in Richmond, Kentucky.[2]

The purpose of the EDS phase is threefold: (1) to support the development of a request for proposal (RFP) for a full-scale facility; (2) to support the certification decision of the Under Secretary of Defense for Acquisition and Technology, as directed by Public Law 105-261; and (3) to support documentation required for the National Environmental Policy Act (NEPA) and the data required for a permit under the Resource Conservation and Recovery Act (RCRA). Each EDS comprises two parts: an EDP and engineering tests and studies to generate required data that were not obtained during the earlier demonstration test phase.

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[1] The stockpile on Johnston Island comprised 2,031 tons, or 6.4 percent, of the original 31,496 tons of chemical nerve and blister (mustard) agents in the U.S. stockpile.

[2] The Parsons/Honeywell WHEAT process was deemed suitable for disposing of the mustard agent munitions stored at Pueblo Chemical Depot, but not for those at Blue Grass Army Depot. This is because the biotreatment component of the Parsons technology is not effective for the treatment of the hydrolysates from the organophosphorus nerve agents that are present at Blue Grass.

ROLE OF THE NATIONAL RESEARCH COUNCIL

In 1997, in response to Public Law 104-201, PMACWA asked the National Research Council (NRC) to evaluate each of the seven technologies that had passed DoD's initial screening. The Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons (ACW I Committee) did this and published its report in August 1999 (NRC, 1999). A supplemental review, requested by PMACWA to evaluate the tests for the three technologies selected for Demo I, was published in February 2000 (NRC, 2000a). Two of the technologies, General Atomics and Parsons/Honeywell, were judged ready to proceed to an engineering design phase. After it had completed the supplemental report, the ACW I Committee was dissolved. Subsequently, under the continuing mandate from Congress, the PMACWA requested that the NRC form a second committee (the ACW II Committee) to evaluate the engineering design packages and related tests for alternative technology facilities at Pueblo and Blue Grass and to examine and evaluate the Demo II tests of the three additional technologies that had not been tested in Demo I.

STATEMENT OF TASK

The present report is the committee's response to the third and final task in its overall statement of task. The report will be produced in time to contribute to the record of decision (ROD) by the Office of the Secretary of Defense on the selection of a technology for the Blue Grass site.[3]

> Task 3
> For the third task, the NRC will assess the ACWA EDS phase in which General Atomics, Foster Wheeler/Eco Logic/Kvaerner, and AEA 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. It is expected that the testing will be completed at or around September 30, 2001. Based on receipt of the appropriate information, including: (a) the PMACWA-approved EDS Plans, (b) the EDS test reports produced by the technology providers, (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 proposed engineering design packages for a chemical demilitarization facility design for disposing of both nerve and mustard munitions
> - produce a report for delivery to the PMACWA by April 5 provided the engineering design package is received by November 15, 2001.

DESCRIPTION OF THE TECHNOLOGY PACKAGES

The assembled chemical weapons stored at Blue Grass Army Depot contain either mustard or nerve agents; most of them are associated with energetic materials (Table ES-1). The technology packages consist of multiple unit operations that work in sequence or concurrently to carry out all aspects of the chemical weapons destruction. The EDP prepared for each technology is designed to be a total solution—that is, it is to treat all of the materials associated with disposal of assembled chemical weapons, energetic materials, metal parts (including munitions bodies), dunnage (e.g., wooden pallets and packing boxes used to store munitions), and nonprocess wastes (e.g., plastic demilitarization protective ensemble (DPE) suits; the carbon from DPE suit filters and from plant heating, ventilating, and air-conditioning (HVAC) filters; and miscellaneous plant wastes). Each EDP includes engineering drawings and documentation, a preliminary hazards analysis, and life-cycle costs and schedules. Short descriptions of the three alternative technologies considered for implementation at Blue Grass Army Depot are given below. More detailed descriptions of the unit operations for each technology are given in Chapters 3, 4, and 5.

AEA Technology Process

Figure ES-1 is a block diagram of the AEA SILVER II™ technology process. The following major operations are included:

- The chemical agent, energetic materials, and metal parts are separated using a modified version of the Army's baseline system disassembly process. Munitions are punched, drained of agent, then opened and emptied of energetics materials.
- Chemical agent is mixed with concentrated nitric acid and silver nitrate. Ag^{2+} generated by the electrochemical cells quickly oxidizes the agent,

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[3] During the course of the ACWA testing program, delays were experienced and receipt of the results slipped into the first quarter of 2002. The NRC contract was therefore modified and a new date (August 15, 2002) was established for completion of the report.

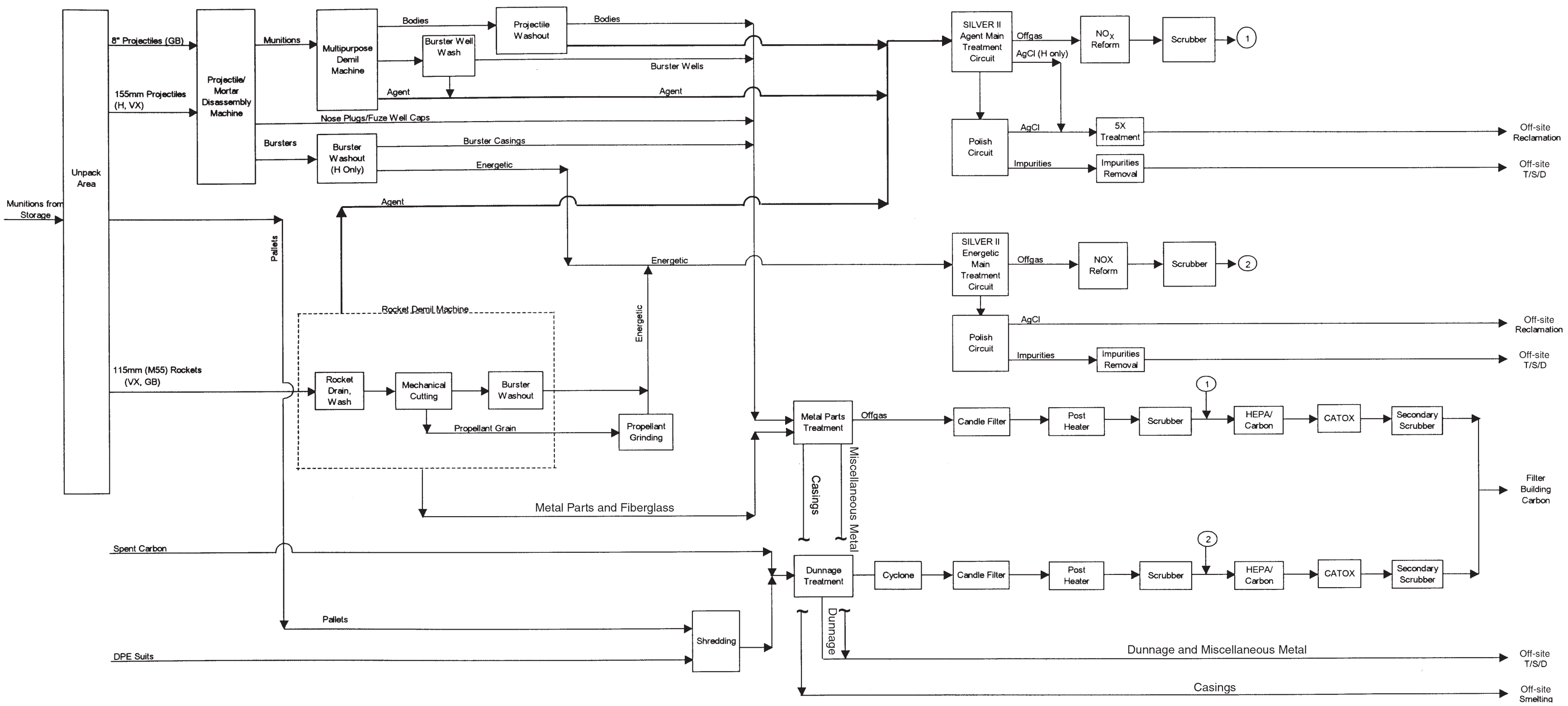

FIGURE ES-1 AEA SILVER II™ demilitarization process. SOURCE: AEA (2001a).

TABLE ES-1 Description of the Chemical Weapons in the Blue Grass Army Depot Stockpile

Item	Fill	Quantity	Agent per Munition (lb)	Total Agent (tons)	Energetics[a]	Energetics Weight per Munition (lb)	Total Energetics Weight (tons)
155-mm projectile, M110	H	15,492	11.7	90.63	Tetrytol	0.41	2.9
8-inch projectile, M426	GB	3,977	14.4	28.83	None		
115-mm rocket, M55	GB	51,716	10.7	276.68	Composition B/	3.2	74.46
					M28 propellant	19.1	448.99
115-mm rocket warhead, M56	GB	24	10.7	0.13	Composition B	3.2	0.035
155-mm projectile, M121/A1	VX	12,816	6	38.45	None		
115-mm rocket, M55	VX	17,733	10.1	88.67	Composition B/	3.2	25.53
					M28 propellant/	19.1	153.95
115-mm rocket warhead, M56	VX	6	10.1	0.03	Composition B	3.2	0.0086

[a] Tetrytol contains 70 percent tetryl and 30 percent TNT. Composition B contains 60 percent RDX, 39 percent TNT, and 1 percent wax.

SOURCE: Adapted from U.S. Army (1997b).

forming carbon dioxide, nitrogen oxides, water, and carbon monoxide.

- Energetics are size-reduced and mixed with concentrated nitric acid and silver nitrate to form a slurry. The slurry is mixed with Ag^{2+} from the electrochemical cells to oxidize the energetic material, forming carbon dioxide, nitrogen oxides, water, inorganic salts, and carbon monoxide.
- Metal munition parts are washed with dilute nitric acid and sent to a metal parts treater along with undissolved fuzes. Any agent remaining on the metal parts and the fuzes is destroyed by thermal decomposition. The resultant metal parts are decontaminated to a 5X level.
- Dunnage and DPE suits are decontaminated to a 5X level in a continuous steam treater.
- Gaseous effluents from the electrochemical, dunnage, and metal parts treatment processes are treated in an emission control system that uses thermal decomposition followed by catalytic oxidation, subsequent scrubbing, and carbon filtering. All treated gaseous effluent is discharged through the HVAC carbon filters for the munitions demilitarization building.

On the basis of earlier results from Demo II testing, PMACWA asked AEA to perform EDS testing and studies on the following:

- Continuous destruction of agent simulant—dimethyl methylphosphonate (DMMP)—in a 12-kW test unit.
- Continuous destruction of energetic material (M28 and Composition B) in a 12-kW test unit.
- Assessment of burster washout methods for projectile and rocket bursters.
- Assessment of safe energetic concentrations for slurries containing energetic materials from the Blue Grass stockpile.
- Propellant size reduction by grinding, followed by some type of high-shear process.
- Particle size reduction and slurry mixing tests (for propellant and burster energetics) to identify a suitable technology for these functions; high-shear vortex mixers were selected and used in agent and energetics destruction testing.
- Testing of hydrocyclones for control of particle size in the feed streams to both agent and energetics electrochemical cells.
- Silver chloride separation and economic recovery of silver for recycling back to the process.
- Nitric acid evaporation from liquors of the electrochemical polishing circuit.
- Characterization and identification of fluoride removal methods for use when processing nerve agent GB.
- Confirmation of corrosion performance of the materials selected for process piping, vessels, and other components; the selected materials were polytetrafluoroethylene (PTFE) and perfluoroalkoxy (PFA) linings.

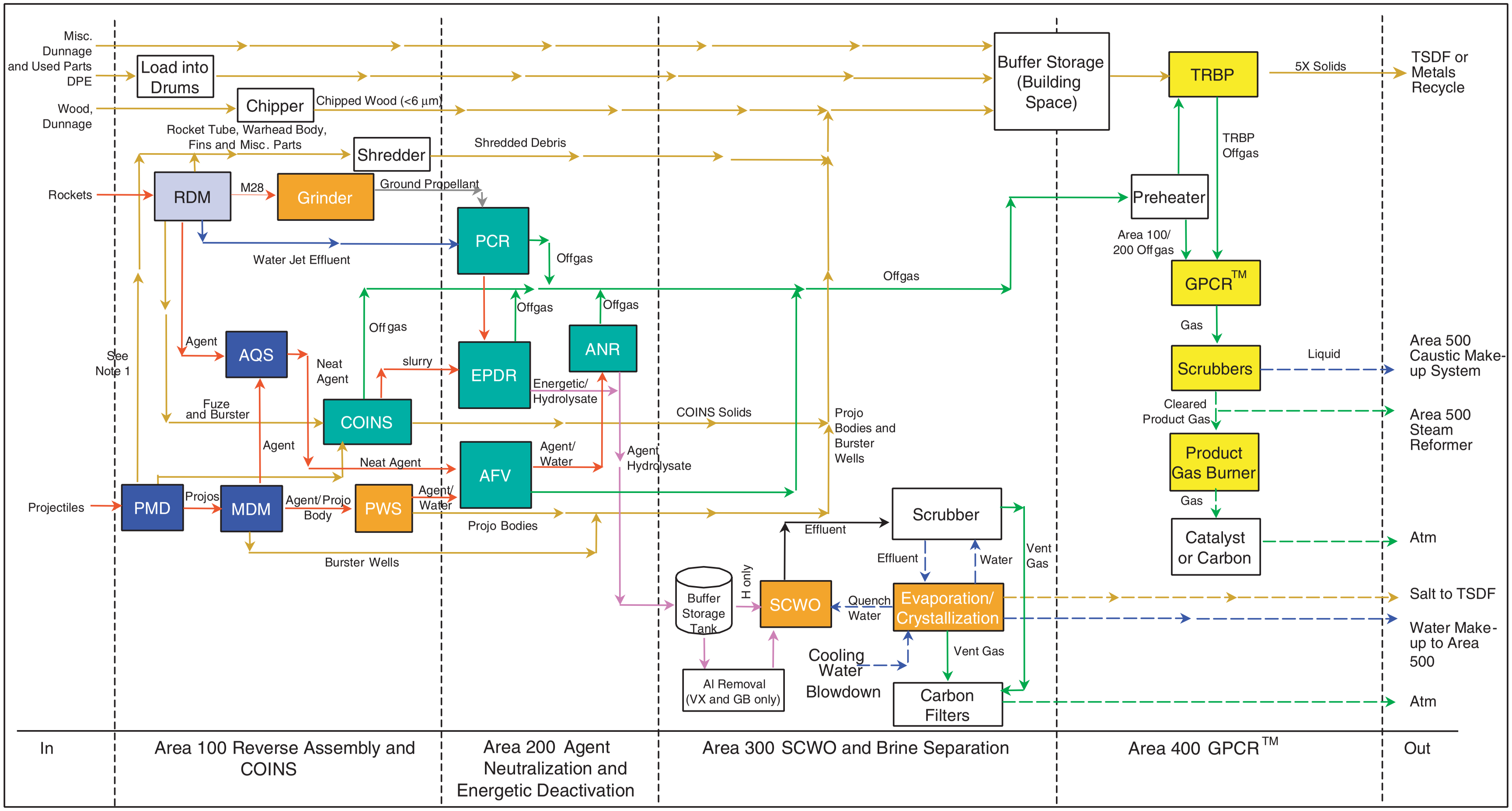

FIGURE ES-2 Schematic flow diagram of the Eco Logic technology package. SOURCE: Eco Logic (2001a).

Eco Logic Technology Process

Figure ES-2 is a block diagram of the Eco Logic technology process. The primary treatment destroys the agent and the energetic materials by hydrolysis with caustic or water. However, the hydrolysis products (hydrolysates) must be further treated prior to final disposal. For this secondary step, Eco Logic proposes to use a transpiring-wall supercritical water oxidation (SCWO) reactor design. The following major operations are included:

- The chemical agent, energetic materials, and metal parts are separated using a modified version of the Army's baseline system disassembly process. Rockets are punched, drained of agent, and disassembled. Projectiles are disassembled first and then drained of agent.
- Chemical agent and energetic materials are decomposed in three separate hydrolysis systems.
- The hydrolysates are further treated in a transpiring-wall SCWO system in which organic compounds are destroyed.
- Metal parts and dunnage are decontaminated to a 5X level, and gaseous effluents from the hydrolysis processes are treated in the gas-phase chemical reduction (GPCR™) system.

The earlier Demo II tests generally confirmed the performance and efficacy of the GPCR™ process for the waste streams tested. However, problems with the agent detection method hampered a full evaluation of the process, and some concerns were raised about the selection of materials of construction when processing agent. In addition, the impact of processing energetics within the thermal reduction batch processor (TRBP) was not understood, and an effective method for grinding M28 propellant was not clearly demonstrated. Accordingly, a test program was implemented to investigate the following areas:

- development of analytical methods specific to the GPCR™ process
- the ability of the TRBP to process residual energetics from the munitions disassembly process
- corrosion testing of metals as materials of construction
- testing of the suitability of various elastomers for use in seals
- testing of equipment for grinding M28 propellant
- long-term testing of the transpiring-wall SCWO system

Tests of the first five areas were completed, and the results were taken into account in the drafting of this report. The long-term testing of the transpiring-wall SCWO system developed by Foster Wheeler was ongoing at Dugway Proving Ground (Utah), and this report incorporates only the early results from these tests.

General Atomics Technology Process

Figure ES-3 is a block diagram of the General Atomics Technology Solution (GATS). The primary treatment destroys the agent and the energetic materials by hydrolysis with caustic or water. The hydrolysis products (hydrolysates) require further treatment before final disposal. For this secondary step, General Atomics proposes to use a vertical cylindrical configuration SCWO reactor. The following major operations are included:

- A modified baseline disassembly process is used; however, cryofracture is used to open the projectile bodies to access the agent. The bodies are cooled to the temperature of liquid nitrogen and fractured. Then the metal parts are separated from the agent.
- The agent-contaminated metal parts and other munition components are treated in a rotary hydrolyzer that rinses the parts with caustic solution and hydrolyzes any remaining chemical agent.
- Energetic materials are treated in a second rotary hydrolyzer in which the energetics are destroyed.
- The chemical agents and partially hydrolyzed agent and energetics from the rotary hydrolyzers are further treated with caustic in batch reactors until all the agent and energetics are destroyed.
- The dunnage is shredded and slurried with energetics hydrolysate.
- All the resulting hydrolysates and the slurried dunnage are treated by SCWO to produce environmentally benign products.
- All the metal parts are treated in a heated discharge conveyor to achieve a 5X condition.
- System offgases are processed through carbon filters.

The GATS process underwent testing and develop-

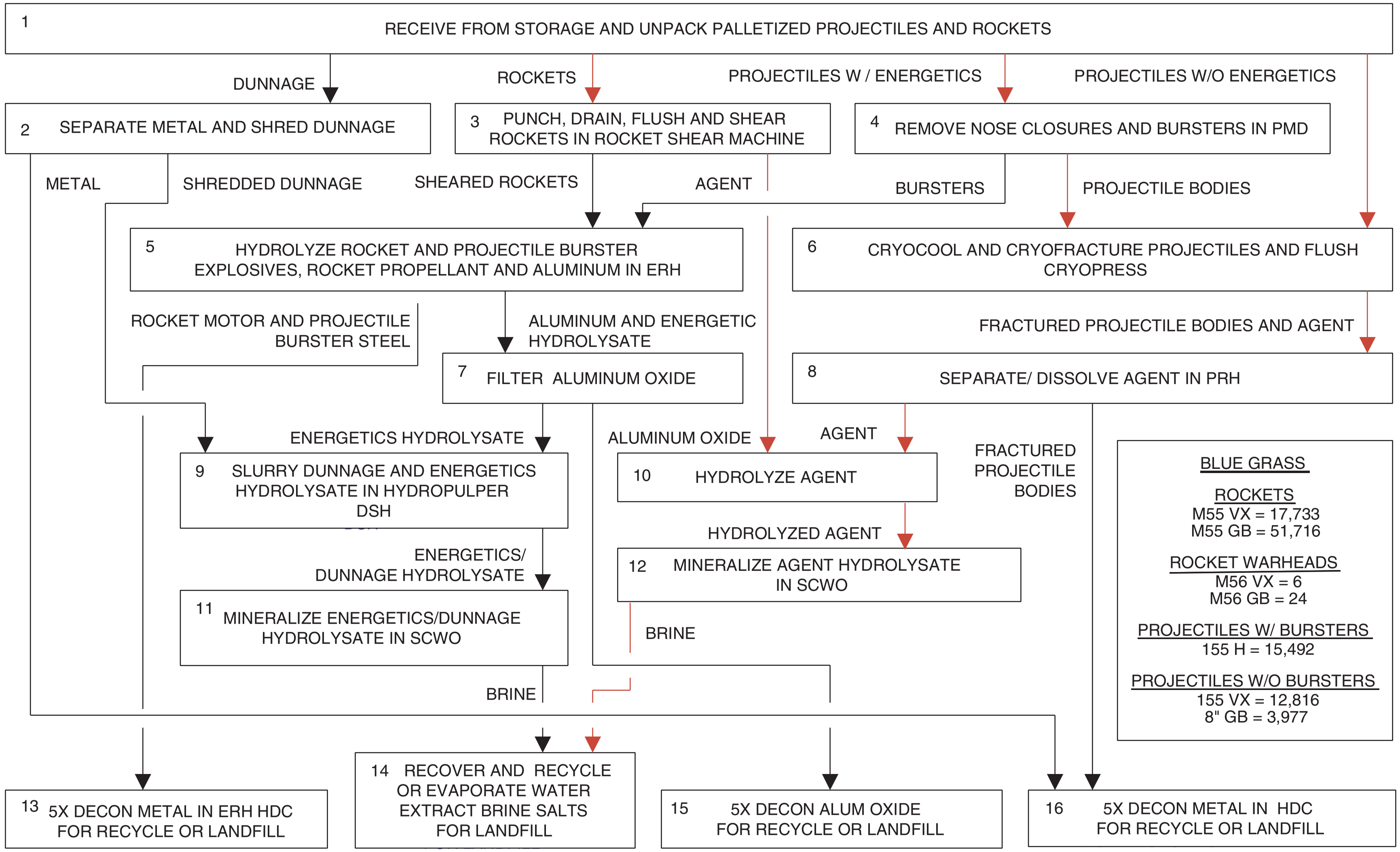

FIGURE ES-3 GATS Blue Grass block flow diagram. SOURCE: General Atomics (2001a).

ment for approximately 3 years, reaching completion in January 2002. The unit operations tested were the dunnage shredder hydrolysis system (DSHS), the energetics rotary hydrolyzer (ERH), and the SCWO reactor.

For this report, the committee concentrated on evaluating the following critical components of the GATS process design that had been identified as potential concerns in earlier NRC reports:

- the long-term reliability of the SCWO system for processing secondary waste feeds from the disposal processing of Blue Grass stockpile agents and munitions
- the advantages and disadvantages of cryofracture over the baseline technology as a means of accessing the agent in the munitions
- the ability of the projectile rotary hydrolyzer (PRH) and the ERH to process their respective feed materials in a reasonable time and with acceptable safety and reliability

Methodology

The committee formed working groups to perform in-depth evaluations of each technology package. As part of their efforts, the groups visited the EDS test sites at Aberdeen Proving Ground, Maryland; Dugway Proving Ground, Utah; and Deseret Chemical Depot, Utah. Committee members held review meetings and also attended PMACWA status-review meetings, which were held periodically. The technology providers and PMACWA staff provided draft copies of reports as they were generated. The final EDPs were released in December 2001. Results of most EDS tests and studies were also provided either before or during December 2001. Data gathering concluded on February 1, 2002; however, the committee continued to monitor final reports and updates on testing as it prepared this report.

In evaluating the general efficacy of the design plans for a chemical demilitarization facility suited to the stockpile at the Blue Grass Army Depot and the readiness of each technology to go forward to the next level, pilot plant testing, the committee relied on its knowledge of the proposed systems, available test results, aggressive data-collection activities, and a thorough review of the engineering design plans and the past experience of the committee members.

GENERAL FINDINGS

General findings on the EDS phase of the ACWA program for the hydrolysis of energetic materials and the three technology packages evaluated in this report appear below. The general findings must be considered in light of the fact that a few of the ACWA EDS tests were not completed in time for the committee to obtain final test results and still meet publication deadlines, and that some process steps remain to be demonstrated on a pilot scale. Also, following its statement of task, the committee limited its evaluations to separate assessments of the total solutions proposed by AEA, Eco Logic, and General Atomics for destroying the assembled chemical weapons at Blue Grass Army Depot. That is, the committee has neither compared the proposed alternatives with one another nor compared them with the Army's baseline incineration system.

Specific findings and recommendations for each technology package, and for the PMACWA-sponsored investigations on hydrolysis of energetic materials, appear at the end of the related chapter. The energetics hydrolysis test program has been completed, and the results can be used to meet the engineering requirements for construction of a disposal facility at Blue Grass Army Depot. Issues concerning hydrolysis of neat tetryl, optimum granulation sizes, more complete characterization of hydrolysis products from aromatic nitro compounds, and optimum process control strategies for full-scale operations were among the issues investigated.

General Finding 1. The reverse assembly of munitions, followed by water or caustic hydrolysis of nerve or mustard agents and associated energetic materials, is a mature, safe, and effective method for initial treatment of the chemical weapons stored at Blue Grass Army Depot. It is ready for immediate implementation for the neutralization of energetics and agents. However, the resulting hazardous streams must be treated further before they are released to the environment.

This finding is based on the test results presented earlier in this report for the EDS hydrolysis of energetic materials and on the effectiveness of the modified baseline reverse assembly and agent hydrolysis processes discussed in this and previous NRC reports (NRC, 1999, 2001a). Because hydrolysis destroys agents and active energetics, the resulting hydrolysates pose a lesser hazard than the assembled chemical weapons.[4] This finding is independent of any subsequent secondary treatment steps that will be necessary to render the hydrolysates environmentally acceptable. That is, agent and energetics could be hydrolyzed even before the secondary treatment process is selected. However, further treatment is necessary to reduce hazardous waste concentrations to a level sufficiently low to allow for final disposal. Additional treatments will also be required for dunnage and the metals parts generated in the reverse disassembly process.

General Finding (Blue Grass) 2. Several of the unit operations of the technology packages have process streams that are not unique to the chemical weapons stockpile. These streams, which include dunnage, brines from water recovery, and hydrolysates, could potentially be treated at existing off-site treatment, storage, and disposal facilities. Off-site treatment of these wastes would simplify the overall technology and facilitate process integration by eliminating the need for further development of individual process steps in the secondary treatment processes and their integration into a whole system. It might also simplify design requirements to meet safety concerns.

All of the process streams that could be treated off-site have compositions similar to waste streams routinely treated by commercial industrial waste treatment facilities. Thus, they could be transported by standard commercial conveyance to commercial facilities that are appropriately permitted to receive the waste.

General Finding (Blue Grass) 3. Based on the results of the PMACWA-sponsored test program on the hydrolysis of energetic materials, the committee believes that hydrolysis can be used safely, effectively, and on the scale necessary for the destruction of the energetic materials contained in assembled chemical weapons at Blue Grass, provided that lead-containing propellants are not processed with tetrytol until the possibility of lead picrate formation can be eliminated.

The test plan that was executed at Holston Army Ammunition Plant (HAAP) demonstrated that the hydrolysis process for the energetic materials of interest successfully destroyed more than 99.7 percent of the energetics treated. Over 9,500 pounds of energetic materials were destroyed while encountering only the types of problems normally expected during the implementation of a new facility and process. The suite of tests performed at Los Alamos National Laboratory (LANL) and at HAAP covered a range of reaction temperatures and caustic concentrations. Under all these conditions, the destruction of energetics was carried out safely and effectively.

Effective destruction of energetic materials during the test program is interpreted by the committee to mean that the products resulting from destruction no longer pose an explosion or detonation hazard. The committee notes, however, that the hydrolysate produced and the gaseous decomposition products are not benign. Gases such as ammonia and oxides of nitrogen are evolved, and cyanide or other toxic substances may be present in the hydrolysate. However, a variety of conventional methods for treating the offgases are available.

The fate of lead during energetics hydrolysis in the presence of picrate is not understood. Until more information becomes available on the speciation of lead and the solubility of lead picrate under the conditions of hydrolysis, the possibility of solid lead picrate formation cannot be dismissed. As a precautionary measure, lead-based propellants should be processed separately from tetryl and tetrytol, eliminating the possibility of lead picrate formation.

General Finding (Blue Grass) 4. Based on the results of EDS testing and the engineering design package, the committee believes that the AEA SILVER II™ total solution technology package could eventually destroy the assembled chemical weapons stored at the Blue Grass Army Depot. However, the immaturity of a significant number of processes in this electrochemical destruction technology could threaten PMACWA's achievement of its objectives for weapons destruction. Considerable additional development and demonstration must be completed before this technology can proceed to a full-scale facility. There is also concern that the ever-increasing complexity and frequent configuration changes that have occurred in the AEA technology package to date are indicative of the immaturity of

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[4] All tests of agent destruction were performed with field-grade chemical agents produced at approximately the same time as the agents loaded into the chemical weapons. Thus, any complications arising from stabilizers, other additives, or degradation products were accounted for during testing.

the process. The approach employed by AEA has been to solve problems as they arise by incorporating additional steps that are modifications of previously demonstrated technologies. The technology provider infers the success of these new steps based on previous demonstrations in other applications but has not generated the data to show that the modifications will result in a safe, effective, and reliable integrated system for assembled chemical weapons destruction.

The AEA SILVER II™ technology process does accomplish the following:

- It destroys chemical agent to a 99.9999 percent DRE and energetic materials to a 99.999 DRE by mediated electrochemical oxidation, provided that the following safeguards are observed:
  —hydrocyclones are used on all slurry feed streams,
  —high-shear vortex mixers are used,
  —piping design minimizes dead legs, and all system equipment and piping use trace heating to prevent precipitation of energetic intermediates, and
  —a second set of electrochemical cells is used to polish the anolyte to the required destruction level.
- It produces acceptable slurry compositions for processing burster energetics and rocket propellants.
- It safely treats process offgases using a combination of thermal treatment, catalytic oxidation, activated carbon filters, and treatment of the munitions demilitarization building (MDB) HVAC system ventilation air through activated carbon filter media prior to release.

However, the committee notes that the effectiveness of some process steps has not been demonstrated:

- Removal of energetics from rockets using the new tube-cutting technology in a modified baseline RDM and achievement of agent-free energetics for processing by SILVER II™ electrochemical cells in a Category C area.
- Integrated operation of the primary and polishing anolyte circuits with a common catholyte circuit.
- Effective control of the flow of slurry mixture to a large number of parallel flow paths. (Currently, flow control has been demonstrated on only three parallel paths, whereas the full-scale application would have up to 432 parallel flow paths in the catholyte feed circuit.)
- Reliable operation of the electrochemical cell membrane, particularly for agent-containing slurries. (Currently, cell pressures and chemistry must be carefully controlled, and it is unclear if the membranes will work well in prolonged exposure to organophosphate-containing slurries.)
- Demonstration of a method to control the movement of fluorine and assure that all elements of the system exposed to fluorine-containing liquids and vapor streams can operate without excessive corrosion and maintenance.
- Decontamination of metal parts and fuzes to a 5X condition in the metal parts treater (MPT).
- Decontamination of dunnage and DPE suits to a 5X condition in the proposed, but as yet untested, enclosed-auger version of the dunnage treatment system (DTS) and successful management of dioxins and furans in the offgas from this process.

General Finding (Blue Grass) 5. Stable operation of the ELI Eco Logic/Foster Wheeler SCWO system at the design conditions has not yet been demonstrated. The SCWO system for treatment of hydrolysates in EDS II testing exhibited frequent spiking in hydrocarbon and carbon monoxide concentrations in the offgas. This issue must be resolved before implementing the Eco Logic process at Blue Grass. If it is resolved, the committee believes that the Eco Logic package could provide an effective and safe means for destroying the assembled chemical weapons. However, the following design features still require validation:

- Design of the overhead conveyor system for the caustic bath should be tested to demonstrate satisfactory reliability.
- Removal of aluminum from the feed to the SCWO reactor. At this time, Eco Logic has not proposed an aluminum removal technology.
- Smoke abatement from the thermal reduction batch processor (TRBP) smoking rooms and the measurement and management of carbon monoxide and other products of incomplete combustion generated in these rooms. (These processes were not adequately addressed in the EDP.)

The Eco Logic technology does accomplish the following:

- It disassembles munitions by a modified baseline reverse-assembly technology.
- It destroys chemical agent to a 99.9999 percent destruction and removal efficiency (DRE) and energetic materials to a 99.999 DRE.
- It effectively treats solid and gaseous residues in a gas-phase chemical reduction (GPCR™) system and decontaminates solids to a 5X condition.
- It destroys by SCWO the hydrolysates and slurries that result from upstream processing.
- It adequately treats the low volumes of offgases produced in the process, including those from the SCWO reactors, through catalytic oxidation and activated carbon adsorption systems.

The committee also points out the following:

- Eco Logic's munitions reverse-assembly process starts with the baseline system technology, but modifications have been made. The rocket dismantling machine (RDM) operation is complex and differs in several aspects from the baseline operation. Further development of this process is needed.
- A testing program, described in Chapter 4, showed that the M28 propellant grinding could be done safely. However, additional testing with a larger, modified grinder is required to demonstrate any design modifications and to determine grinder and motor sizes for the full-scale plant.
- The Demo II tests generally confirmed the performance and efficacy of the GPCR™ process for the treatment of the waste streams from the process. However, problems with the agent detection method hampered a full evaluation of the process, and some concerns were raised about the selection of materials of construction for the processing agent.

EDS testing has provided additional design data, but the results have not yet been used in operating systems.

General Finding (Blue Grass) 6. Based on the results of the EDS testing and the EDP, the committee believes that the General Atomics technology package is an effective and safe method for the destruction of assembled chemical weapons at the Blue Grass Army Depot. The GATS SCWO system appears to have reached a level of maturity where construction and testing of a full-scale reactor to treat agent hydrolysate is the next logical step. Tests of the SCWO system have shown that the SCWO reactor requires frequent scheduled maintenance; however, the level of maintenance estimated by General Atomics for the Blue Grass application is manageable by well-trained operators and maintenance personnel. The committee still is concerned about possible problems associated with the extent of scale-up that will be needed for the SCWO reactor. Another concern is the proposed operation of the full-scale ERH and PRH in a continuous rather than batch mode and the maintenance difficulties that would result.

The General Atomics technology does accomplish the following:

- It disassembles munitions by a modified baseline disassembly that removes the agent from the projectile bodies by cryofracture.
- It destroys chemical agents to a 99.9999 percent DRE by hydrolysis.
- It destroys fuzes by processing first in the ERH and then in the HDC.
- It destroys energetic materials by hydrolysis.
- It provides effective 5X-level decontamination for munition bodies using an electrically heated HDC.
- It destroys by SCWO the hydrolysates and slurries that result from this processing.
- It adequately treats the offgases produced in the process, including those from the SCWO reactors, through a CATOX unit and activated carbon adsorption systems.

It is expected that weapons processing at Blue Grass will require no SCWO liner changes for the SCWO reactors treating energetics hydrolysates and dunnage slurried in energetics hydrolysates. For each of the two operating reactors used to treat agent hydrolysates, the technology provider has scheduled six liner changes that are estimated to be necessary for treatment of GB hydrolysate (i.e., 12 liner changes total for the GB campaign) over the 16-month operating life of the plant. During periods of steady operation with GB hydrolysate, based on six liner changes per reactor, each liner will be replaced after 110 hours of operation. It appears likely that liner change-out procedures in the full-scale system will encounter more problems than were encountered in the smaller test reactors used to date.

Although no evidence was found of material hanging up in the ERH during the batch testing conducted to date, this is, in the committee's opinion, a distinct possibility during the longer operating runs of a full-scale system in a continuous mode. The ERH is 50 feet long and the PRH is 40 feet long in the Blue Grass design. Should some form of blockage or other problem occur inside cylinders of this length, maintenance would be extremely difficult, especially considering that energetics and/or agent might be present. The tests demonstrated that the ERH and PRH appear to work well in a batch mode. Replacing the continuous-flow hydrolyzers proposed for Blue Grass with larger versions of the batch hydrolyzers used during the Demo I and EDS testing would appear to eliminate this risk and simplify system operation.

General Finding (Blue Grass) 7. As the ACW I Committee observed, the unit operations in any of the three technology packages have never been operated as total integrated processes (NRC, 1999). As a consequence, a prolonged period of systemization will be necessary to resolve integration issues for the selected technology as they arise, even for apparently straightforward unit operations.

1

Introduction

At the request of Congress, the Army is pursuing alternatives to incineration for the destruction of assembled chemical weapons at Pueblo Chemical Depot in Pueblo, Colorado, and at Blue Grass Army Depot in Richland, Kentucky. The Army's Program Manager for Assembled Chemical Weapons Assessment (PMACWA) is pursuing two technology options—Parsons/Honeywell's Water Hydrolysis of Explosives and Agent Technology (WHEAT) and General Atomics Total Solution (GATS)—for Pueblo and three options (AEA SILVER II™, the Eco Logic process, and GATS) for Blue Grass. The four technologies have been carried forward to an engineering design phase that also includes experimental demonstration testing.

In the spring of 2000, at the request of PMACWA, the National Research Council (NRC) assembled the Committee on Review and Evaluation of Alternative Technologies for Demilitarization of Assembled Chemical Weapons: Phase II (ACW II Committee) to evaluate and assess the alternative technologies for the two sites. This report contains the ACW II Committee's assessment of the engineering design packages (EDPs) and associated testing for the three alternative technologies being considered for Blue Grass Army Depot. This first chapter will briefly describe the program, the NRC's role in the program, and the characteristics of the chemical weapons stockpile and chemical agents stored at Blue Grass Army Depot.

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 to the baseline incineration system developed by the Army for the demilitarization of assembled chemical weapons and that it conduct demonstration tests for at least two of them. The laws included the following stipulations:

- All funds for the construction of baseline incineration facilities at 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 never had been associated with the baseline incineration program.
- DoD should "coordinate" its activities with the NRC.

In December 1996, DoD appointed the deputy to the commander of the Soldier and Biological Chemical Command to be the 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 (U.S. Army, 1997a). The term "total solution" means that a candidate technology should address all aspects of chemical weapons disposal, from disassembly and separation of the agent and energetics to disposal of the product streams. 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 in 1999 (Burns and Roe; General Atomics; and Parsons-Allied Signal). Details 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, on operations that had not been previously used for 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 (now designated Demo I) 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, which mandated 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.

In response, the PMACWA authorized engineering design studies (EDSs) 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 EDSs for Parsons/Honeywell and General Atomics were configured for application of these technology packages at the Pueblo Chemical Depot in Pueblo, Colorado. These studies are designated the EDS I phase of the ACWA program. Of the two technologies, only the General Atomics Total Solution (GATS) is among the three alternative technologies being considered for use at the Blue Grass Army Depot in Richmond, Kentucky, in the EDS II phase of the program, which is the subject of this report.[1]

The purposes of the EDS for each technology were 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 a request for proposal (RFP) for a pilot facility; and (3) support the documentation requirements for compliance with the National Environmental Policy Act (NEPA) and the Resource Conservation and Recovery Act (RCRA) permit application. Each EDS required the development of an EDP and experimental tests to acquire necessary 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 explored under the ACWA program." PMACWA was requested to proceed under the same guidelines as contained in Public Law 104-208 and to continue use of the Dialogue process and Citizens' Advisory Technical Team and their consultants.

The PMACWA then initiated a second demonstration program, referred to as Demo II, to demonstrate the three technologies that had not been selected for Demo I. The Demo II tests were performed between March and July 2000 by three technology providers: (1) AEA Technologies, (2) Eco Logic, and (3) Teledyne-Commodore. These three technologies are among those listed in Table 1-1. If on the basis of

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[1] The Parsons/Honeywell WHEAT process was deemed suitable as a candidate for disposing of the mustard agent munitions stored at Pueblo Chemical Depot, but its biotreatment component would not be effective for the treatment of the hydrolysates from the organophosphorus nerve agents stored at Blue Grass.

TABLE 1-1 The Original Seven Candidate Technologies Under Consideration for the ACWA Program

Technology Provider[a]	Access to Munitions	Treatment of Agent	Treatment of Energetics	Treatment of Metal Parts	Treatment of Dunnage
AEA Technology	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	Modified reverse assembly (multiple lines, compact layout, new drain and wash).	Hydrolysis; SCWO; gas-phase chemical reduction (GPCR™).	Hydrolysis, SCWO, GPCR™.	Hydrolysis; GPCR™ to 5X.	Hydrolysis; GPCR™ to 5X.
Parsons/ Honeywell	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 Foster Wheeler, a former member of the Lockheed Martin team along with ELI Eco Logic, as the prime contractor. ELI Eco Logic is the technology provider for the gas-phase chemical reduction (GPCR™) system, and Foster Wheeler is the technology provider for the transpiring-wall supercritical water oxidation (SCWO) system. El Dorado Engineering is the technology provider for the reverse-assembly equipment design and the materials-handling equipment design. Kvaerner is the provider of the hydrolysis design and performs plantwide process integration services.

[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.

Demo II testing these technology packages were deemed viable options for destroying assembled chemical weapons, they would also be considered for implementation at the Blue Grass Army Depot and could progress to the EDP.

Two of these technologies, AEA's SILVER II™ process and the Eco Logic process,[2] were carried forward to the EDS II phase of the ACWA program along with the GATS process.[3] Evaluations of these three technologies for implementation at the Blue Grass Army Depot and an update of the status of PMACWA-sponsored tests on hydrolysis of energetic materials are the subject of this report.[4]

AEA Technologies, Eco Logic, and General Atomics were awarded contracts to develop EDPs and to perform further required demonstration testing for disposal of the chemical weapons at Blue Grass Army Depot. The initial (preliminary) EDPs were submitted in July 2001, and drafts of the final EDPs were submitted to PMACWA in October 2001.

ROLE OF THE NATIONAL RESEARCH COUNCIL

When the ACWA program was initiated in late 1996, the PMACWA requested that the NRC independently evaluate alternative technologies and submit a report by September 1, 1999, a month before the DoD's *Supplemental Report to Congress* was due (DoD, 1999). After agreeing with DOD 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. In accordance with the statement of task for the study, the committee did not recommend a best technology or compare any of the technologies with the baseline incineration system in use at some storage sites. Members of the committee visited the demonstration test sites for the three technologies selected for Demo I before the unit operations were systemized in January 1999, but data-gathering activities had to be terminated on March 15, 1999, before the results of the Demo I tests had been received, to allow producing 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 Demo I tests. The committee was asked to determine if and how the results affected the findings and recommendations in the original report, as well as to reevaluate the steps for implementation it had indicated were required (NRC, 1999). In March 2000, the committee published a supplemental report (NRC, 2000a) 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 ACW I 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 Chemical Depot (EDS I); and (3) an evaluation of the EDSs for Blue Grass Army Depot (EDS II). In August 2001, the ACW II Committee published a report evaluating the engineering design packages developed by General Atomics and Parsons/Honeywell (formerly Parsons-Allied Signal) and related studies for the Pueblo Chemical Depot in fulfillment of the second of the three tasks (NRC, 2001a). In October 2001, a second report evaluating the Demonstration II results was published in response to the first task (NRC, 2001b). The present report, in response to the third task, evaluates the three EDSs that are candidates for destroying the assembled chemical munitions at the Blue Grass Army Depot.

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[2] In the EDS II phase of the ACWA program, Eco Logic became the prime contractor for a team that also included Kvaerner Process Systems Inc., Foster Wheeler, and El Dorado Engineering.

[3] The Teledyne-Commodore technology did not successfully complete the Demo II testing.

[4] The testing program on energetics hydrolysis that PMACWA sponsored to improve the technology base for this means of disposal was initially reviewed in Analysis of Engineering Design Studies for Demilitarization of Assembled Chemical Weapons at Pueblo Chemical Depot (NRC, 2001a).

DESCRIPTION OF THE STOCKPILE AT BLUE GRASS ARMY DEPOT

Agents

The unitary[5] chemical agents in U.S. stockpiles are nerve agents (GB and VX)[6] and three related forms of blister (mustard) agent (H, HD, and HT). The original stockpile contained 31,496 tons of unitary agents, which were stored in a variety of munitions and containers under ambient conditions, where they exist primarily as liquids. Because the stockpile at Johnston Atoll in the Pacific has been completely destroyed by incineration and the stockpile at Deseret Chemical Depot, in Utah, has been partially incinerated, as of October 3, 2001, the amount in storage was approximately 24,098 tons.

Table 1-2 lists some of the physical properties of the chemical agents GB, VX, and HD. The nerve agent VX is a nonvolatile liquid that has a high boiling point and adheres to surfaces for days or weeks, whereas the nerve agent GB (sarin) is a liquid with a volatility similar to that of water and therefore evaporates relatively quickly. These two nerve agents are highly toxic and lethal in both liquid and vapor forms. They are ingested by respiration or skin contact and can kill in a matter of minutes by interfering with respiratory and nervous system functions. In pure form, these nerve agents are practically colorless and odorless. GB is relatively nonpersistent in the environment, whereas VX can persist in the environment, especially under dry conditions.

The blister agents are skin vesicants that evaporate very slowly, although somewhat faster than VX.[7] The active ingredient in all these blister agents is bischloroethyl sulfide, $(ClCH_2CH_2)_2S$. HD, called distilled mustard, is nominally pure mustard. H, often called Levenstein mustard, is approximately 70 percent mustard and 30 percent impurities that tend to be polysulfides such as $(ClCH_2CH_2)_2S_n$, where n = 2, 3. HT contains 67 percent mustard and 22 percent T, $(ClCH_2CH_2SCH_2CH_2)_2O$, and minor components. Mustard has a garliclike odor and is hazardous on contact and as a vapor. Mustard agent is only slightly soluble in water and is very persistent in the environment.

The stockpile at Blue Grass Army Depot contains munitions with nerve agents VX and GB and the blister agent H (see Table 1-3). Originally, references supplied to the committee indicated that the projectiles at Blue Grass contained distilled mustard, HD (U.S. Army, 1996). However, information supplied to the committee by PMACWA in March 2002 indicated that the M110 155-mm projectiles actually contain H and not HD (U.S. Army, 1997b). All ACWA testing with mustard agent during the demonstration and EDSs was performed using HD because very little H was available to produce the large quantities of hydrolysate required for the tests. Testing with H is planned to verify that compositional differences do not alter the results of the tests performed to date with HD.

Containers and Munitions

Unitary chemical agents are stored in spray tanks, bulk storage (ton) containers, and a variety of munitions, including land mines, rockets, bombs, and artillery and mortar projectiles. Some munitions may contain a fuze, booster, burster, and/or propellant. These components are referred to collectively as energetic materials or energetics. The term “assembled chemical weapon” (ACW) is used to describe munitions that contain chemical agent and may or may not contain energetic materials in an assembled configuration. Rockets, mortars, and land mines are typically stored with their energetic components in place; projectiles may or may not contain bursters or fuzes. The ACWA program (and this report) is focused on alternative means of destroying these four types of munitions. Additional details on the weapons stored at Blue Grass Army Depot are provided in Appendix A.

M55 rockets pose the greatest storage risk because they contain approximately 20 pounds of M28 propellant, a double-base propellant composed of nitroglycerine, nitrocellulose, plasticizers, a burn-rate modifier, and a stabilizer. The exact composition is given in Table 1-4. The propellant slowly decomposes exother-

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[5] The term “unitary” indicates a single chemical loaded in munitions or stored as a lethal material. More recently, “binary” munitions have been produced in which two relatively safe chemicals are loaded into separate compartments to be mixed to form a lethal agent after the munition is fired or released. The components of binary munitions are stockpiled in separate states. They are not included in the present CSDP, but they are being destroyed in a separate program.

[6] Nerve agents are organophosphonate compounds. They contain phosphorus double-bonded to an oxygen atom and single-bonded to a carbon atom. GB is O-isopropyl methylphosphonofluoridate. VX is O-ethyl-S [2-(diisopropyl amino) ethyl]-methylphosphonothiolate.

[7] Names such as mustard gas, sulfur mustard, and yperite have also been applied to this agent. The term “mustard gas” is often used, but the chemical is a liquid at ambient temperature.

TABLE 1-2 Physical Properties of Chemical Warfare Agents

Agent Characteristic	Nerve GB	Nerve VX	Blister[a] HD
Chemical formula	$C_4H_{10}FO_2P$	$C_{11}H_{26}NO_2PS$	$C_4H_8Cl_2S$
Molecular weight	140.10	267.38	159.08
Boiling point (°C)	158	298	217
Freezing point (°C)	–56	<–51	14.45
Vapor pressure (mm Hg)	2.9 at 25°C	0.0009 at 25°C	0.072 at 20°C
Volatility (mg/m^3)	22,000 at 25°C	12.6 at 25°C	75 at 0°C (solid) 610 at 20°C (liquid)
Surface tension (dynes/cm)	26.5 at 20°C	32.0 at 20°C	43.2 at 20°C
Viscosity (cS)	1.28 at 25°C	12.256 at 20°C	3.95 at 20°C
Liquid density at 20°C (g/cm^3)	1.0887	1.0083	1.2685
Solubility (g/100 g of distilled water)	100; soluble in organic solvents	5 at 25°C; best solvents are dilute mineral acids	0.092 at 22°C; soluble in acetone, CCl_4, $CHCl_3$, tetrachloroethane, ethyl benzoate, ether
Heat of vaporization			
(Btu/lb)	149	125	190
(cal/g)	82.8	69.4	94
Heat of combustion			
(Btu/lb)	10,000	15,000	8,100
(cal/g)	5,600	8,300	4,500

[a] The blister agents are labeled H, HD, and HT. The active ingredient, mustard, in all these blister agents is bis(2-chloroethyl) sulfide, $(ClCH_2CH_2)_2S$. HD, called distilled mustard, is nominally pure mustard. H, often called Levenstein mustard, is approximately 70 percent mustard and 30 per cent impurities, which tend to be polysulfides such as $(ClCH_2CH_2)_2S_n$, where $n \geq 2$. HT contains 67 per cent mustard and 22 percent T, $(ClCH_2CH_2SCH_2CH_2)_2O$, and minor components.

SOURCE: Buchanan et al. (1999); Penski (1994); Samuel et al. (1983).

TABLE 1-3 Description of the Chemical Weapons in the Blue Grass Army Depot Stockpile

Item	Fill	Quantity	Agent per Munition (lb)	Total Agent (tons)	Energetics[a]	Energetics Weight per Munition (lb)	Total Energetics Weight (tons)
155-mm projectile, M110	H	15,492	11.7	90.63	Tetrytol	0.41	2.9
8-inch projectile, M426	GB	3,977	14.4	28.83	None		
115-mm rocket, M55	GB	51,716	10.7	276.68	Composition B/	3.2	74.46
					M28 propellant	19.1	448.99
115-mm rocket warhead, M56	GB	24	10.7	0.13	Composition B	3.2	0.035
155-mm projectile, M121/A1	VX	12,816	6	38.45	None		
115-mm rocket, M55	VX	17,733	10.1	88.67	Composition B/	3.2	25.53
					M28 propellant	19.1	153.95
115-mm rocket warhead, M56	VX	6	10.1	0.03	Composition B	3.2	0.0086

[a] Tetrytol contains 70 percent tetryl and 30 percent TNT. Composition B contains 60 percent RDX, 39 percent TNT, and 1 percent wax.

SOURCE: Adapted from U.S. Army (1997b).

TABLE 1-4 Composition of M28 Propellant

Component	Percentage
Nitrocellulose (12.6% N)	60.1
Nitroglycerine	23.8
Triacetin	9.9
Dimethylphthalate	2.6
2-nitrodiphenylamine (2-NDPA)	1.7
Lead stearate	1.9

SOURCE: Bonnett (2000).

mically, forming nitrogen dioxide, which in turn can catalyze more rapid decomposition. The stabilizer reacts with the nitrogen dioxide and prevents the autocatalytic action. However, because these munitions have been stored since the 1950s and 1960s, and because the stabilizer concentration continues to slowly diminish in each rocket, the risk of a catastrophic event increases with time. The rockets also have an aluminum body with welded joints that are susceptible to corrosion by GB, creating a situation that has resulted in agent leakage.

Routine inspections of the stockpile storage igloos occasionally reveal the presence of leaking rockets or other munitions. When leakers are detected, they are overpacked and subsequently stored and handled separately.

STATEMENT OF TASK

The complete statement of task for the ACW II Committee is given below. This report addresses only Task 3.[8]

> 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.)
>
> *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, Foster Wheeler/Eco Logic/Kvaerner, and AEA 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. It is expected that the testing will be completed at or around September 30, 2001. Based on receipt of the appropriate information, including: (a) the PMACWA-approved EDS Plans, (b) the EDS test reports produced by the technology providers, (c) PMACWA's

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[8] During the course of the ACWA testing program, delays were experienced and receipt of the results slipped into the first quarter of 2002. The NRC contract was therefore modified and a new date (August 15, 2002) was established for completion of the report.

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 proposed engineering design packages for a chemical demilitarization facility design for disposing of both nerve and mustard munitions
- produce a report for delivery to the PMACWA by April 5, 2002 provided the engineering design package is received by November 15, 2001.

SCOPE AND APPROACH OF THIS STUDY

As mentioned above, this report is the ACW II Committee's response to Task 3 of its statement of task (i.e., review and evaluate EDP drawings, documentation, and tests developed for the destruction of chemical weapons at the Blue Grass Army Depot). This report had to be produced in time to contribute to the record of decision (ROD) by the Office of the Secretary of Defense on a technology selection for the Blue Grass site, which at the time of this writing was scheduled for early 2003, following satisfaction of NEPA procedures and requirements. As with previous NRC reports on the ACWA program, the committee did not compare the technologies with one another or with the Army's baseline incineration system.

It is important to note that although the designs in the EDPs were developed for processing the H agent that is stored at Blue Grass, all testing to date has been with HD. This assessment of technology performance is therefore based on test data for HD. PMACWA was planning to perform tests with H when this report was being prepared. The committee notes that differences in chemical composition between H and HD may affect the efficacy of the technologies.

Because some experimental tests in support of the EDPs were not completed or available when this report was being prepared, the committee was not able to review or evaluate the full set of final test results. However, committee members did attend status-review sessions organized by PMACWA and were given access to all available draft reports by the technology providers. To meet the publication date of August 2002, the committee had to submit its consensus draft in April 2002 for editing and preparation for peer review, which forced it to terminate its data-gathering phase by early February 2002. Throughout the preparation of this report, the ACW II Committee continued to monitor what was happening in the ACWA Dialogue group and to participate in activities where information was exchanged between members of the committee and the Dialogue.

ORGANIZATION OF THIS REPORT

Chapter 2 discusses the results of hydrolysis studies on energetic materials, sponsored by PMACWA in response to the ACW I Committee's original recommendations. Chapters 3, 4, and 5 evaluate each of the three candidate technology engineering packages. The results of testing completed in support of the EDPs and available to the ACW II Committee at the time of this report are also reviewed in each chapter. Chapter 6 summarizes the committee's evaluation of the three technology packages and presents some new general findings. Appendix A describes munitions stored at the Blue Grass Army Depot. Appendix B lists committee meetings, site visits, and information-gathering activities. Appendix C contains biographies of the committee members.

2

Hydrolysis Tests of Energetic Materials

Both the General Atomics and the Eco Logic technology packages use caustic (base) hydrolysis as the initial treatment to destroy the energetic materials recovered from chemical weapons (see Table 2-1). Secondary treatment of the resulting hydrolysate is accomplished by supercritical water oxidation (SCWO). The chemistry of caustic hydrolysis takes advantage of the susceptibility of the functional groups commonly found in energetic materials to attack by hydroxide ion, yielding products that are essentially nonenergetic. Caustic hydrolysis decomposes energetic materials to organic and inorganic salts, soluble organic compounds, and various gaseous effluents. Partial hydrolysis of some energetic materials, particularly materials with aromatic ring systems, may lead to ill-defined oligomeric materials with low solubility in either water or organic solvents.

An understanding of the chemistry for the hydrolysis of energetic materials is essential to the design and operation of a chemical agent demilitarization facility, where high levels of engineering controls are necessary to ensure the safe disposal of both the chemical agents and the energetic materials and the prevention of process upsets throughout the facility.

In its 1999 report, the ACW I Committee expressed concern that the technology base for the hydrolysis of energetic materials was not as mature as that for the hydrolysis of chemical agents, that chemical methods for destroying energetics had only recently been considered, and that the chemistry of the process was not well understood (NRC, 1999). Among the specific issues to be addressed were these:

- the particle size reduction that must be achieved for proper operation

TABLE 2-1 Nominal Composition of Energetic Materials Used in Chemical Munitions

Energetics Material	Composition
Tetryl	2,4,6-trinitrophenylmethylnitramine
Tetrytol	70% tetryl/30% TNT
Composition B	60% RDX/39% TNT/1% wax
Composition B-4	60% RDX/39.5% TNT/0.5% calcium silicate
M28 propellant	60.0% nitrocellulose/23.8% nitroglycerin/ 9.9% triacetin/2.6% dimethyl phthalate/ 2.0% lead stearate/ 1.7% 2-nitrodiphenylamine
M8 propellant	52.15% nitrocellulose/43% nitroglycerin/ 3% diethyl phthalate/1.25% potassium nitrate/0.6% ethyl centralite
M1 propellant	84% nitrocellulose/9% dinitrotoluene/ 5% dibutyl phthalate/1% diphenylamine/ 1% lead carbonate

SOURCE: Bonnett (2000).

- the solubility of energetics in specific alkaline solutions
- the 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 byproducts of hydrolysis as a function of the extent of reaction

Similar concerns were reiterated by the ACW I Committee in a supplemental report evaluating the results of testing conducted during the Demonstration I phase of the ACWA program (NRC, 2000a). In response to these concerns, PMACWA initiated a multilaboratory test program to identify the chemical and engineering parameters for the efficient, safe alkaline hydrolysis of the energetic materials found in assembled chemical weapons. The project plan and preliminary results of this program were reported in Chapter 2 of the ACW II Committee's 2001 report on the EDS I phase of the ACWA program for an alternative technology facility at Pueblo Chemical Depot (NRC, 2001a). In this chapter, the ACW II Committee briefly describes the hydrolysis treatment process for energetic materials, reviews PMACWA's program for engineering design testing of energetics hydrolysis, and assesses the results of the test program.

OVERVIEW OF THE TEST PROGRAM

In response to the challenges listed above, PMACWA devised a test plan to address these issues. This EDS testing began in December 2000, and the tests were completed at the end of March 2001. The U.S. Army Tank-Automotive and Armaments Command (TACOM) Armament Research, Development and Engineering Center (ARDEC) at Picatinny Arsenal, New Jersey, was responsible for coordinating the program.

The Picatinny test plan requirements document described objectives, planned testing, and team member responsibilities in the EDS test program for hydrolysis of energetic materials (Bonnett, 2000). However, the objectives evolved during the execution of the program, and the final retrospective statement of the objectives, dated June 18, 2001 (after the completion of the testing), is given in Appendix N of the final report on the EDS testing program for energetics hydrolysis (Bonnett and Elmasri, 2001). As stated in Appendix N, the objectives were as follows:

> - address concerns identified by the National Research Council (NRC) and processing issues that surfaced at Radford AAP [Army Ammunition Plant] and PANTEX, Inc., during the manufacturing of the various hydrolysates used to support the previous demonstration testing;
> - determine the optimum process operating parameters to support scale-up of the hydrolysis process and the definitization of the Engineering Design Package (EDP) for the pilot phase for the Pueblo Chemical Agent Disposal Facility scheduled in August of 2001 and for the Blue Grass Chemical Agent Disposal Facility scheduled in August 2002;
> - define a hydrolysis process that is safe and environmentally compliant, and that will efficiently produce hydrolysates of energetic materials recovered from the various chemical munitions during the disassembly process.
> - produce hydrolysates that will be ready for post-treatment processing using such technologies as SCWO, bio-reactor, etc.

The participants in the program and their principal responsibilities were as follows:

- TACOM-ARDEC was responsible for program management and incorporation of bench-scale results into processes at Holston Army Ammunition Plant (HAAP) to demonstrate hydrolysis operations.
- HAAP and BAE Systems were responsible for design, installation and operation of a hydrolysis reactor system. The design of the hydrolysis system was done by BAE Systems, in consultation with TACOM-ARDEC.
- Los Alamos National Laboratory (LANL) performed bench-scale testing in support of the engineering-scale hydrolysis reactor system.

Testing at the Holston Army Ammunition Plant

The reactor used for the tests at HAAP has the following specifications (Bonnett and Elmasri, 2001):

- a 2,000-gallon, glass-lined, jacketed reactor vessel
- a recirculation loop, unheated and uninsulated, that reenters the reactor vessel at the bottom
- a dual-flight, Hastelloy C, variable-speed agitator in the reactor vessel
- a condenser/scrubber for offgases
- meters to monitor pH, temperature, and flow

The energetic feeder is a single-screw, loss-in-weight feeder with a 500-lb/hr capacity. The control system uses programmable logic control (PLC) with remote and local control capabilities. Energetic material was fed into the reactor as a dry solid and screened through a 1.0-inch × 1.0-inch mesh as it was added to the feeder hopper to prevent large particles from entering the reactor.

Six different energetic materials and one mixture of materials were processed in the study (Bonnett and Elmasri, 2001):

- Composition B
- Composition B-4
- tetrytol
- M1 propellant
- M28 propellant in granular form
- M8 sheet propellant
- M28 propellant/Composition B-4 (86/14 weight-percent ratio, based on amounts in an M55 rocket)

Runs 1 through 5, denoted "commissioning runs," were used to verify that the systems for operating the reactor and for data collection during the subsequent energetics hydrolysis tests were functioning as intended. A summary of the conditions used in the commissioning runs is given in Table 2-2. The feed used for these tests was Composition B, a material that is readily available at HAAP and very similar in composition to Composition B-4 (see Table 2-1). The working volume in the reactor for all the commissioning runs was about 1,700 gallons, and the reaction temperature was maintained at 87°C for the duration of the hydrolysis. The feed was added continuously through an Acrison loss-in-weight feeder.

The test matrix for the energetics hydrolysis tests in runs 6 through 21 is given in Table 2-3 (Bonnett and Elmasri, 2001). For these runs, the reactor operated at 87°C with 700 gallons of caustic. Each run lasted 12 hours, comprising a 4-hour addition phase (only 3 hours for tetrytol) followed by an 8-hour postaddition processing phase. Throughout the duration of the runs, airflow at a rate of 40 standard cubic feet per minute (scfm) was maintained through the reactor headspace. No material balance calculations were performed. For reasons discussed below, run 14 was extended by 12 hours to give a total run duration of 24 hours. The tests involving Composition B-4, M1 propellant, M28 propellant, and the mixture of Composition B-4 and M28 propellants all used a 50-lb/hr feed rate during the first hour, 100 lb/hr during the second hour, 150 lb/hr during the third hour, and 200 lb/hr during the fourth hour, for a total of 500 pounds of energetic material per run. However, for the tetrytol runs, the addition phase lasted only 3 hours and the total mass was only 350 pounds per run, because of the limited availability of tetrytol. The feed rate profile for tetrytol was 50 lb/hr during the first hour, 100 lb/hr during the second hour, and 200 lb/hr during the third hour.

The progress of the hydrolysis reaction was monitored by periodic sampling and analysis of the hydrolysate and by continuous monitoring of the gases evolved. Sixteen 42-ml samples of the hydrolysate were collected during each run. The sampling system attached to the recirculation loop comprised an insulated cabinet with an ice or dry ice bath in which the

TABLE 2-2 Conditions Used in the Commissioning Runs at HAAP

Test Run No.	Total Feed (lb)	Feed Rate (lb/hr)	Caustic Concentration (wt %)	Comments
1	200	Erratic	12	Software and configuration problems encountered with loss-in-weight feeder.
2	550	492 nom. (erratic)	6	Developed strategy for dealing with foaming.
3	252	125	20	Strategy for eliminating foam refined.
4	500	125	14	Starting caustic solution was hydrolysate from run 3 to which additional NaOH had been added to increase the concentration to 14%.
5	500	500	9.6	Starting caustic solution was hydrolysate from run 4.

SOURCE: Bonnett and Elmasri (2001).

TABLE 2-3 Test Matrix for the Army Hydrolysis Study at HAAP

Test Run No.	Energetics Feed	Caustic Concentration (wt %)	Agitation Speed (rpm)
6	Composition B-4	12	70
7	Composition B-4	18	70
8	Composition B-4	25	70
9	M1 propellant	11.3	70
10	M1 propellant	20	70
11	M1 propellant	25	70
14	M1 propellant	20	70
12	M28 propellant (unleaded)	11.3	70
13	M28 propellant (unleaded)	20	70
15	Tetrytol	12	80
16	Tetrytol	21	80
17	Tetrytol	26	80
18	M8 sheet propellant	12	80
19	M8 sheet propellant	21	80
20	M8 sheet propellant	26	80
21	M28 propellant (leaded)/ Composition B-4, 86/14 wt%	20	80

SOURCE: Bonnett and Elmasri (2001).

sample bottles were chilled so that the hydrolysate samples would be cooled as soon as the samples were collected. Each sample bottle was charged with 30 ml of 6 N sulfuric acid prior to the start of the run. The combination of the cooling and the neutralization of the caustic by the sulfuric acid quenched any further reaction. At the end of each run, a 4- or 8-L sample of the hydrolysate was collected from the recirculation loop.

Evolved gases were monitored continuously for O_2, CO_2, NO_x, CO, and total hydrocarbons (THC) by a continuous emission monitoring system (CEMS). Batch samples of the offgases were also taken and analyzed for NH_3, HCN, residual energetic materials, volatile organic compounds, and N_2. The batch samples were collected over the entire duration of each run, and the single value reported represents the average concentration for the whole run.

At the conclusion of each test run, the interior surfaces of the reactor were visually inspected and samples of residue collected and analyzed.

Bench-Scale Tests at Los Alamos National Laboratory

Researchers at Los Alamos National Laboratory (LANL) studied the hydrolysis reactions at bench scale by analyzing the hydrolysate and gaseous effluents generated by processing of the following energetic materials: Composition B-4 tetrytol, M1 propellant, M8 propellant, and M28 propellant, and mixtures of M28 propellant with Composition B-4 and M28 propellant with tetrytol. Two concentrations, 12 and 20 percent, of caustic were used in the experiments for all materials except the M8 propellant, which was studied using only 20 percent caustic.

The experiments were carried out in a 2-L Parr reactor fitted with an internal cooling coil to cool the reaction mixture if necessary and to prevent runaway reactions. The temperature was controlled by a proportional-integral-differential (PID) controller. All experiments started at ambient temperature, which was raised to the desired reaction temperature, 93°C, in a nearly linear ramp over about 15 minutes for the propellants or 25 and 35 minutes for Composition B-4 and tetrytol, respectively.

The Composition B-4 was provided to LANL in the form of large flakes and was used as received. The tetrytol was in large to medium-size chunks. The M1 propellant and the M28 propellant were provided and used as small grains. The M8 propellant was provided in stacks of three sheets weighing 30-35 g each and sewn together with cotton thread. The sheets were not separated before being manually fed into the reactor.

Gas evolution was monitored by online mass spectrometry. Liquid samples were removed during the experimental runs through a sampling port in the reactor. The samples were immediately diluted by a factor of 10 with cold water to quench the reaction and were kept refrigerated until they were analyzed. Ion chromatography was used to determine the concentrations of nitrite, nitrate, formate, and acetate. Total organic carbon, total inorganic carbon (TOC/TIC), and ammonia, were determined. Differential scanning calorimetry was used to determine whether there were any products in the hydrolysate liquid or the solid residue at the end of the reaction that would decompose exothermically. Hydrolysates from Composition B-4 and tetrytol were also subjected to high-performance liquid chromatography (HPLC) analysis to determine whether any of the starting energetic compounds remained at the end of the hydrolysis.

RESULTS OF THE TEST PROGRAM AT THE HOLSTON ARMY AMMUNITION PLANT

Sixteen hydrolysis runs were successfully completed at HAAP in the course of this study (see Table 2-3). For every energetic material subjected to hydrolysis by itself (i.e., not mixed with another energetic material), the energetic components were reduced to products that no longer posed an explosion hazard. Although the operational parameters, such as base concentration and agitation rate, were varied only minimally, the tests demonstrated that the destruction of the energetic materials could be carried out at a rate consistent with that of other unit operations in the applicable technology packages and under conditions that permitted control of the rate of heat release of the hydrolysis reaction.

Process Controls

One of the major concerns in processing energetics when an exothermic reaction occurs is the reliable control of temperature to prevent runaway reactions. Under the conditions employed at HAAP, the temperature of the reactor varied by only a few degrees from the 87°C set point for all the energetic materials processed. The formation of foam on the surface of the caustic solution during the processing of Composition B-4 was initially a concern, because the foam could insulate the surface of the liquid, preventing heat from escaping and possibly leading to uncontrollable self-heating. However, the spray system used to break up the foam was effective, and the processing of Composition B-4 proceeded uneventfully.

The feeding of dry energetic material into the reactor involved the greatest hazard in the whole process. Standard precautions employed in the handling of dry energetic powders (grounding and bonding of metal surfaces, minimization of stresses exerted on the energetic material) were followed. No incidents occurred during the handling of any of the dry energetics. It should be noted that none of the technology packages using hydrolysis for the destruction of energetics includes handling them as loose dry powders. Instead they are handled as slurries.

Gaseous Products of Hydrolysis

Samples of the gaseous reaction products were taken through a sampling line upstream of the condenser and scrubber through which the offgases were treated prior to release to the atmosphere. The major gaseous products found during all the hydrolysis runs were NH_3, N_2O, CO, and hydrocarbons, which were below flammable limits. Quantities of the gases differed depending on the material being hydrolyzed. The evolution of N_2O occurred primarily during the addition of the feed to the reactor and decreased during the postaddition reaction phase. Ammonia evolved throughout the duration of the run, with the amount evolved increasing after the completion of the addition phase.

Composition B-4 Runs

The hydrolysis of Composition B-4 produced N_2O and CO rapidly upon addition of the energetic feed. For all three runs involving Composition B-4 (runs 6, 7, and 8), the maximum concentration of CO in the offgas was 400-450 ppm, peaking near the end of the addition phase. By the end of the runs, the concentration dropped below 50 ppm for runs 6 and 7 and below 100 ppm for run 8. The evolution of N_2O paralleled the evolution of CO. It peaked at a concentration of about 20,000 to 25,000 ppm and dropped off to less than 5,000 ppm at the end of the run. Ammonia gas was measured only in the batch samples. The concentration ranged from 12.9 to 23.3 g/m^3.

Small amounts of the energetic constituents of Composition B-4 were detected in the offgas stream during the addition phase. RDX and HMX (an impurity in RDX) were measured at a few hundreds and a few tens of micrograms per cubic meter, respectively. TNT, which is more volatile than RDX and HMX, was detected at a few milligrams per cubic meter. Minor amounts of other energetic impurities found in TNT, such as 2,4- and 2,6-dinitrotoluene, were also detected. This was attributed to the entrainment of some of the dust from the energetic material in the offgas stream. No energetic compounds were detected in the offgas at the end of the runs.

M1 Propellant Runs

Three concentrations (11.3, 20, and 25 weight percent) of caustic were used in the hydrolysis runs of M1 propellant. In runs 9, 10, and 11, the THC level in the offgas continued to increase during the postaddition reaction phase, rising to 1,600-2,500 ppm by the end of the 12-hour runs. The runs at base concentrations of 20 and 25 percent showed lower hydrocarbon evolution than the one at 11.3 percent concentration. The runs in 20 and 25 percent caustic showed THC levels of 1,600 ppm in the offgas at the end of the run, whereas the run at 11.3 percent showed 2,500 ppm. Run 14, a replicate of run 10, was carried out for 24 hours instead of the standard 12 hours. It produced THC results very similar to those of run 10 for the first 12 hours. Near the end of the first 12 hours, the THC concentration began to decrease and had reached a level of about 800 ppm as the duration of the run approached 24 hours. Just before termination of the run, the agitation rate was increased and a sudden spike appeared in the THC level, reaching a maximum of about 2,200 ppm. The final report for the project states that these data supported the hypothesis that there is a significant amount of dissolved gas in the hydrolysate (Bonnett and Elmasri, 2001).

Several compounds were identified in the offgas stream from M1 propellant that probably arose from the solvents used in processing the propellant. Acetone (~50-90 ppm), ethyl ether (~30-130 ppm), and toluene (~30 ppm) were all detected in runs 9, 10, and 11. Cyclohexanone was reported for all these runs at 70-120 ppm. No batch analyses of the offgases from run 14 were reported.

There was also a dependence on caustic concentration in the evolution of N_2O in the runs. The runs at 20 and 25 percent caustic produced higher levels of N_2O during the addition phase, peaking at about 800-900 ppm, then tailing off to less than 100 ppm at the end of the run. For the run at 11.3 percent caustic, the level of N_2O did not exceed 500 ppm at any time and decreased slowly after the addition phase to about 100 ppm by the end of the run.

M28 Propellant Runs

M28 propellant contains about 24 percent nitroglycerin. Nitroglycerin is a liquid at ambient temperature and pressure, with a significant vapor pressure at the reactor temperature. During the addition phase of the hydrolysis of M28 propellant, about 7,000 ppm of nitroglycerin was detected in the offgases, but shortly after completion of the addition phase, the level dropped to about 400 ppm and remained nearly constant for the duration of the run.

Acetone and toluene were detected in the offgases at levels comparable to those in the M1 propellant offgas. No diethyl ether was reported for M28 propellant. A few ppm of 2-butanone (methyl ethyl ketone), another common solvent, were detected.

Ammonia evolved over the course of the reaction, starting at an average level of about 100 mg/m^3 during the addition phase and rising to about 1,000 mg/m^3 during the postaddition reaction. Some N_2O was generated, although at levels an order of magnitude lower than for Composition B-4. Oxides of nitrogen were detected at a few parts per million during both the addition and reaction phases.

Tetrytol Runs

Tetrytol, like Composition B-4, contains a matrix of TNT in which a second ingredient is incorporated. In tetrytol, the second component is tetryl (2,4,6-trinitrophenylmethylnitramine). The volume and content of the offgases differ markedly from those of Composition B-4. Whereas Composition B-4 generates significant quantities of ammonia, tetrytol generates two to three orders of magnitude less ammonia. Composition B-4 produced N_2O in the range of 15,000 to 20,000 ppm during the addition phase, whereas tetrytol produced only one-tenth that amount. However, the THCs were 10 times higher for tetrytol than for Composition B-4. Neither explosive produced more than a few parts per million of NO_x.

As was the case with Composition B-4, some energetic materials were detected in the offgases during the addition phase, but none of these persisted throughout the run. No energetic materials were found in the offgases during the postaddition reaction phase.

M8 Propellant Runs

The main constituents of the offgases from the M8 propellant hydrolysis were CO, N_2O, THCs, and NH_3. Both CO and N_2O were generated mainly during the addition phase, reaching levels of 150-250 and 4,000 ppm, respectively. The THCs rose slowly during the addition phase, reaching a peak about 1 or 2 hours after completion of the addition, then declining slightly from a peak of about 700-800 ppm to an end-of-run level of a little over 500 ppm. The ammonia level averaged 500 ppm during the addition phase and then rose to an average level of about 1,800 ppm for the reaction phase.

Mixed M28 Propellant and Composition B-4 Run

The mixed M28 propellant and Composition B-4 produced offgases that were the combined results of the separate treatment of the two energetic materials. The evolution of N_2O was high during the addition phase (peak of ~12,000 ppm), as was characteristic of Composition B-4, and the evolution of THCs maintained a level of about 150 ppm during the entire postaddition reaction phase. Low levels of NO_x were observed. Ammonia was evolved at levels lower than those produced by Composition B-4 and higher than those produced by M28. The only oddity in the offgas data for the mixed M28 and Composition B-4 run was the presence of tetryl during the addition phase of the hydrolysis. This suggests that there was contamination at some point, possibly in the feed system, because tetrytol was the last energetic processed through the Acrison loss-in-weight feeder in the three runs just preceding the mixed M28 and Composition B-4 run.

Hydrolysate Analyses

Since the hydrolysis of energetic materials in the Eco Logic and General Atomics technology packages (see Chapters 4 and 5) produces hydrolysates that would be subjected to some subsequent treatment (e.g., SCWO), the main concern is the continued presence of an energetic species in the hydrolysate that might constitute an explosion hazard. The data collected from midrun samples are not reliable because the results are scattered, and it is not clear whether the suspended solids were included in the analysis. The most that can be determined from these data is that the concentration of energetic material is decreasing with time during the processing phase, as expected.

The end-of-run hydrolysate analyses provide more useful data on the presence of energetic species, because they are based on a larger homogeneous sample. The end-of-run hydrolysate analyses indicate that, in half of the cases, the level of energetic material had been reduced below the detection limit. The exceptions are listed in Table 2-4. None of these low levels of energetic material pose any explosion hazard, because even slurries with 10 to 30 percent of strong explosives such as RDX or HMX are not detonable. The explosion hazard of the hydrolysates containing only a few parts per million of any of the compounds in Table 2-4 is nil (EDE, 2001a).

TABLE 2-4 Residual Energetic Material in End-of-Run Hydrolysate Samples

Test Run No.	Caustic Strength (wt%)	Energetic Feed	Energetics in Hydrolysate	
			Compound	mg/L
6	12	Composition B-4	TNT	4.89
7	18	Composition B-4	TNT	4.89
16	21	Tetrytol	Picric acid[a]	73.1
17	26	Tetrytol	Picric acid[a]	39.1
18	12	M8 propellant	Nitroglycerin	11.2
19	21	M8 propellant	Nitroglycerin	0.40
21	20	M28 propellant/ Composition B-4	TNT HMX	29.8 24.1

[a] In alkaline solution, this will be present in the dissociated form, as picrate anion.

SOURCE: Bonnett and Elmasri (2001).

The main identifiable carbon-containing compounds in the hydrolysates of all the runs were acetate and formate salts. The fraction of the total organic carbon in the hydrolysate that was accounted for by these two species ranged from about 10 percent for tetrytol to about 40 percent for the M28 propellant. Thus, a considerable amount of the carbon in solution remains unidentified. Because the hydrolysate will undergo further processing, this is not necessarily a problem, provided the explosion hazard has been removed in the hydrolysis. The concentration of organic carbon in the hydrolysate is about 2 percent (16.375 g/L in run 12), and slurries of high explosives such as RDX are not detonable at concentrations below about 30 percent (see above), so the assumption that the hydrolysate would not be detonable even if all the organic carbon in the hydrolysate were present as an explosive remains valid.

The presence of cyanide in the hydrolysate does raise some concern. The levels of cyanide in the end-of-run analyses ranged from 29 mg/L (M1 propellant, 11.3 percent caustic) to 705 mg/L (tetrytol, 26 percent caustic). While the solution is alkaline, the cyanide will remain in solution as aqueous cyanide ion. However, if the solution were made neutral or acidic, which would reduce the hazards associated with handling caustic solutions, the cyanide would be converted to hydrocyanic acid (HCN), which would present a toxic gas hazard (DoD, 1999).

M8 Sheet Propellant

The hydrolysis tests at HAAP of M8 propellant proceeded as expected, with the typical gaseous and condensed products being formed as noted previously and with the M8 being completely consumed in the course of the hydrolysis. A problem was encountered, however, because the layered sheets of the propellant are sewn together with cotton thread. The cotton threads did not decompose or disintegrate and became entangled on the lower agitator and shaft. Thus, they could clog pumps, pipes, and valves. However, the committee expects that simple engineering solutions are available to prevent this from happening. For the tests at HAAP, the sheets were not treated in any way to disassemble them or reduce the size of the feed introduced into the reactor. Instead, the sheets were added to the caustic solution manually, bypassing the loss-in-weight feeder.

RESULTS OF THE TEST PROGRAM AT LOS ALAMOS NATIONAL LABORATORY

The studies at LANL were designed to provide complementary data on the hydrolysis reaction of energetic materials under similar conditions to those employed at HAAP. Gas production was monitored by mass spectroscopy over the course of the reaction, but only N_2, N_2O, NH_3, and NO were reported. No measurements were made of total hydrocarbons (THC) in the offgas.

Composition B-4 and Tetrytol Hydrolysis

Some experiments on the hydrolysis of Composition B-4 were reported prior to this program (see Appendix C in Bonnett and Elmasri, 2001). Composition B-4 at 10 percent concentration was treated with 3 M (12 weight percent) caustic at 90°C in a total solution of 50 ml. However, the temperature was not well controlled and oscillated over about 10°C with a periodicity of approximately 10 minutes. The results were consistent with the observations made at HAAP:

- The main organic products were acetate and formate, with formate being present at about 5 to 10 times the concentration of acetate.
- Most of the carbon in solution was in the form of organic compounds, not inorganic.
- The main nitrogen species in solution was nitrite, with a minor amount of nitrate.

The experiments undertaken at LANL were carried out in a 2-L Parr reactor charged with 1 kg of caustic solution and 10 percent energetics at 93°C (versus 87°C at HAAP). In contrast to the experiments at HAAP, the energetic material was introduced into the caustic at ambient temperature before heating. The heating from ambient to 93°C typically took about 40 minutes. The reaction temperature was maintained with vigorous stirring set at 720 rpm until the total reaction time reached 100 min.

Hydrolysis of Composition B-4 and tetrytol under these conditions using 12 or 20 percent caustic destroyed the energetic materials. Analysis of the Composition B-4 and tetrytol hydrolysate by high-performance liquid chromatography (HPLC) and differential scanning calorimetry did not detect picric acid or any other known energetic material. However, because detection limits for the method were not reported, it is not possible to determine whether amounts of picrate or other energetics below the detection limits might have been present in these small samples (Bonnett and Elmasri, 2001).

In the LANL experiments, all of the energetic material was introduced into the vessel at the beginning of the run, when the caustic was still at ambient temperature; in the HAAP runs, the caustic was heated to the reaction temperature before the energetic feed was introduced. Therefore, the rate of destruction from the LANL data is not comparable to that from the HAAP data. It appears that the evolution of gas commenced at about 65-70°C for both Composition B-4 in 20 percent caustic and tetrytol in 12 percent caustic. No temperature data were presented for Composition B-4 in 15 percent caustic, and tetrytol appeared to begin generating gas at a somewhat higher temperature (~80°C) in 20 percent caustic.

Propellant Hydrolysis

The hydrolyses of all the propellants tested (M1, M8, and M28) formed some solid residue, the analysis of which was not pursued. However, differential scanning calorimetry analysis showed no significant exotherms in the thermograms of the residues from any of the propellants.

The evolution of gas from the propellants began at a somewhat higher temperature than for Composition B-4 and tetrytol. The first observation of N_2O did not occur for M1 propellant until the reaction temperature reached about 90°C. The other propellants began evolving gas at around 80°C.

Rayon Cloth Treatment with Caustic

At the request of the PMACWA, LANL also looked at the degradation of the rayon cloth used to contain the M1 propellant charges. Swatches of the cloth were heated in caustic solutions (6, 12, and 20 weight percent NaOH) at 93°C for 340 minutes. Although the swatches were damaged by the treatment, they were not consumed nor did they disintegrate. The final weights were not determined, because some crystalline material adhered to the swatches and could not be removed by repeated rinsing with water. This result is consistent with the results at HAAP. However, the committee believes that simple engineering solutions are available to address the problem posed by the persistence of the rayon bags.

The Possible Formation of Lead Picrate

The possible formation of lead picrate when nitroaromatic compounds such as TNT and tetryl are hydrolyzed in the presence of lead has been an ongoing concern of the two NRC ACW committees (NRC, 2000a, 2001a, 2001b). The perceived problem is that solid lead picrate, precipitated from solution and dried, might detonate. For this to happen, the following conditions must be met: (1) both lead (II) ions and picrate ions must be present in the solution, (2) the lead picrate must precipitate because its solubility product is exceeded, and (3) the solid must be present in a condition that is detonable with a stimulus that can be achieved at some point during the processing cycle. The processing cycle includes the hydrolysis, storage, and subsequent treatment of the hydrolysate (if the solid is transferred along with the hydrolysate to the post-hydrolysis treatment step) and, when appropriate, cleanup of the hydrolysate reactor and system.

It has been shown that picrate ion is formed during the hydrolysis of tetrytol. As shown in Table 2-5, picrate was observed in the midrun analyses for all the hydrolysis runs of tetrytol and in the midrun analyses for the mixed M28/Composition B-4 run. For two of the three tetrytol runs, there was a low level of picrate remaining at the end of the run, but no picric acid was reported in any of the hydrolysate samples from any of the Composition B-4 runs that preceded the tetrytol runs. From the midrun concentrations of picric acid in the tetrytol runs, it appears that the formation of picrate decreases with increasing base concentration. This observation, and the nonzero values of picric acid in the end-of-run hydrolysate samples for runs 16 and 17, would lead one to expect to find some picrate remaining in the hydrolysate at the end of run 15. However, none was reported. The level of picric acid reported during the postaddition phase of run 15 varied between 10 and 30 mg/L, yet none is reported in the end-of-run analysis of the hydrolysate.

The presence of picrate in run 21 (M28/Composition B-4) might be attributable to contamination. The offgas analysis during the addition phase of the mixed energetic indicated some tetryl in the offgas. This may have been due to contamination of the feed system, since tetrytol was processed in the runs that used the loss-in-weight feeder immediately preceding the mixed energetic run. (The M8 sheet propellant was hydrolyzed in runs 18, 19, and 20, between the tetrytol and the mixed M28/Composition B runs, but the M8 sheets were fed into the reactor manually, bypassing the loss-in-weight feeder.) If some tetrytol was inadvertently introduced into the reaction vessel from a contaminated feeder, it might have led to the formation of a small amount of picrate. No picrate was reported in the end-of-run analysis of the hydrolysate from run 21.

The results from the work done at LANL (see below) failed to find any picrate in the hydrolysates from either Composition B-4 or tetrytol. However, because the detection limit for the method used was not reported, it is not possible to determine whether or not the LANL results are consistent with those reported in the Holston study (Bonnett and Elmasri, 2001).

The two studies, taken together, suggest that tetryl, not TNT, leads to the formation of picrate, since neither the LANL results nor the Holston results showed the formation of picrate in the absence of tetryl. If this is correct, the formation of lead picrate cannot occur when Composition B and M28 propellant are coprocessed, provided no tetryl contamination is present. The solubility of lead picrate in the reaction medium under consideration is not known. Because lead in solution at high pH is predominantly in the form of plumbite ion ($HPbO_2^-$), the concentration of free Pb^{2+} ions should be very low, and the solubility product of lead picrate would have to be exceedingly small for

TABLE 2-5 Picrate Formation in the Hydrolysis of Energetics

Test Run No.	Energetics Feed	Caustic Concentration (wt%)	Picrate Concentration (mg/L) Max Midrun	End-of-Run
15	Tetrytol	12	~85	None reported
16	Tetrytol	21	~40	73.1
17	Tetrytol	26	~20	39.1
21	M28/ Composition B-4	20	~6	None reported

SOURCE: Bonnett and Elmasri (2001).

any lead picrate to precipitate. Even if some lead picrate were to be formed, the risk of an explosion from initiation of the lead picrate would be small. If good engineering practices are followed and the hydrolysis reactor is designed to eliminate dead-legs in the piping and other places where solids might accumulate, any solid that is formed would be distributed through the reactor and would be dispersed in the surrounding aqueous phase. The main hazard due to the formation of any dry lead picrate would occur if the reactor were allowed to dry out (thus removing the water that desensitizes the lead picrate) or during disassembly of the apparatus.[1]

Although precipitation of lead picrate is not likely to be a hazard during normal operation of a well-designed hydrolysis reactor, some data are still unavailable. During the hydrolysis runs at Holston, the material balance for lead was extremely poor. None of the runs of M1 propellant, each of which contained a little less than 4 pounds of lead in the feed, indicated any lead in the end-of-run hydrolysate analysis. Also, no lead was identified in the solids remaining at the end of any of the hydrolysis reactions. The inability to account for the lead dissolved or suspended in solution at high pH is troubling. At a minimum, the lack of data leaves open the possibility that lead picrate could be formed until such time as the speciation of the lead can be explained or the precipitation of lead can be fully dismissed on the basis of the solubility of lead picrate at these pHs.

An added complication in the destruction of the chemical weapons at Blue Grass is the imprecision with which the composition of the munitions is known. The original ACWA RFP stipulated that the burster charges in the 155-mm rocket warheads contained Composition B (U.S. Army, 1997a). However, the MIDAS database, which is maintained by the Army to describe the entire munitions stockpile, states that those bursters may contain either Composition B or tetrytol (U.S. Army, 1997b). Surveillance data from the Army indicate that the bursters in the Blue Grass stockpile contain only Composition B, even though certain munitions lots in the overall stockpile were manufactured with both Composition B and tetrytol. The type of burster is controlled by lot at manufacture and can be deduced from the lot number information. If a comprehensive inspection of the stockpile at Blue Grass reveals that some of the bursters contain tetryl, then it will be necessary for the technology providers to employ disassembly processes that keep the rocket propellant segregated from the burster explosive.

SUMMARY ASSESSMENT

The EDS energetics hydrolysis test program was instituted to address the ACW I Committee's concerns about the hydrolysis of energetic materials. The test program has now been completed, and the results indicate that the hydrolysis of the energetic materials present at Blue Grass can be conducted safely. The program addressed all of the findings and recommendations of the ACW I Committee (NRC, 1999, 2000a) with the exception of testing on the hydrolysis of energetics contaminated by agent. The committee believes that such testing is no longer necessary because the EDS program has clearly indicated that both agents and energetics are destroyed by caustic. The probability of any interaction between the agent and the energetics that would prevent complete destruction is unlikely. Moreover, the committee again notes that integration concerns such as the safe handling and destruction of agent-contaminated energetics should be addressed as soon as practicable to minimize delays during systemization of the disposal facility (NRC, 2001a).

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[1] Although the transport of *dry* lead picrate is forbidden by the Department of Transportation (DOT) (49 CFR 172.101), the addition of water desensitizes it. No hazard classification is available from DOT for wetted lead picrate, but silver picrate, which is similar in sensitivity, may not be shipped when dry but is classified as a flammable solid (Hazard Class/Division 4.1) when it is wetted with water by at least 30 percent by mass.

FINDINGS AND RECOMMENDATIONS

Findings

Finding (Blue Grass) EH-1. Alkaline hydrolysis is an effective and safe method for destroying the energetic materials present in the weapons at Blue Grass Army Depot. There appear to be no insurmountable obstacles to using this technology to destroy the energetics in assembled chemical weapons.

Finding (Blue Grass) EH-2. Results from the energetics hydrolysis test program have shown that hydrolysis rates are consistent with the proposed throughput rates necessary to meet the current disposal destruction schedule for the Blue Grass stockpile.

Finding (Blue Grass) EH-3. The presence of environmentally hazardous compounds in the offgases of the hydrolysis reactor requires that an emission control system capable of removing these products be employed in any technology package that uses caustic hydrolysis for the destruction of energetic materials.

Finding (Blue Grass) EH-4. Cyanide was present in the alkaline hydrolysate from all the energetic materials tested at HAAP. If the hydrolysate is neutralized or made acidic, HCN will be liberated.

Finding (Blue Grass) EH-5. The cotton threads used to hold sheets of M8 propellant together and the rayon bags used to hold M1 propellant grains are not destroyed by the caustic under the conditions that were used in this study. This may cause clogging or entanglement in pipes, pumps, or valves.

Finding (Blue Grass) EH-6. Analysis of the hydrolysates from lead-containing energetic materials failed to account for most of the lead present in the feed.

Recommendations

Recommendation (Blue Grass) EH-1. Tetrytol should not be processed simultaneously with any lead-containing energetic material until such time as it can be confirmed that lead picrate will not precipitate from the hydrolysate. The solubility of lead picrate in the reaction medium should be determined as a function of temperature, and the lead in the reaction should be accounted for in a mass balance.

Recommendation (Blue Grass) EH-2. Steps should be included in the processing of M1 propellant and M8 sheet propellant to prevent the persistent rayon bags or cotton threads from clogging or jamming the hydrolysis equipment.

3

AEA SILVER II™ Technology Package

DESCRIPTION OF THE PROCESS

The AEA Technology (AEA) SILVER II™ technology is based on the highly oxidizing nature of Ag^{2+} ions, which are generated by passing an electric current through a solution of silver nitrate in nitric acid in an electrochemical cell similar to those used in commercial electrochemical processes. The electrochemical reactions used in the SILVER II™ technology belong to a class of chemical processes collectively known as mediated electrochemical oxidation (MEO). MEO processes have been offered as an alternative to conventional incineration for destroying hazardous wastes. Because they are also relatively new in development and application, experience with these processes is limited (Chiba et al., 1995).

Detailed descriptions of AEA's original total system solution and its unit operations were provided in the original ACW I Committee's report (NRC, 1999) and the ACW II Committee's report on Demo II testing (NRC, 2001b). This report provides an update reflecting major changes in the process identified by the technology provider team (consisting of AEA Technology and CH2MHILL) based on prior testing results and on EDS II tests and studies.

Figure 3-1 shows a block diagram of the overall process for the current 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. Energetic materials are removed, reduced in size, and prepared as a slurry in water before further treatment. The agent and energetics are then destroyed in separate electrochemical processing units. Metal parts and fuzes are thermally decontaminated to a 5X condition in a metals parts treater (MPT). Dunnage and DPE suit material are decontaminated to a 5X condition by a dunnage treatment system (DTS) that is similar but not identical to the continuous steam treater (CST) in the Parsons/Honeywell WHEAT[1] technology package proposed for disposing of the mustard agent munitions at Pueblo Chemical Depot (NRC, 2001a). Silver, water, nitric acid, and NO_x are recovered, converted to reagents, and reused in the process. Other solid, liquid, and gaseous effluents from various process units are collected, separated, treated, and tested to prepare them for safe discharge from the plant. Table 3-1 summarizes key plant performance requirements used in developing the SILVER II™ technology package.

The following sections recap briefly the sequence of process steps beginning with munitions disassembly and ending with treatment of various waste streams for final disposal. They are derived from the complete description of the AEA technology proposed in the EDP

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[1] Water hydrolysis of explosives and agent technology.

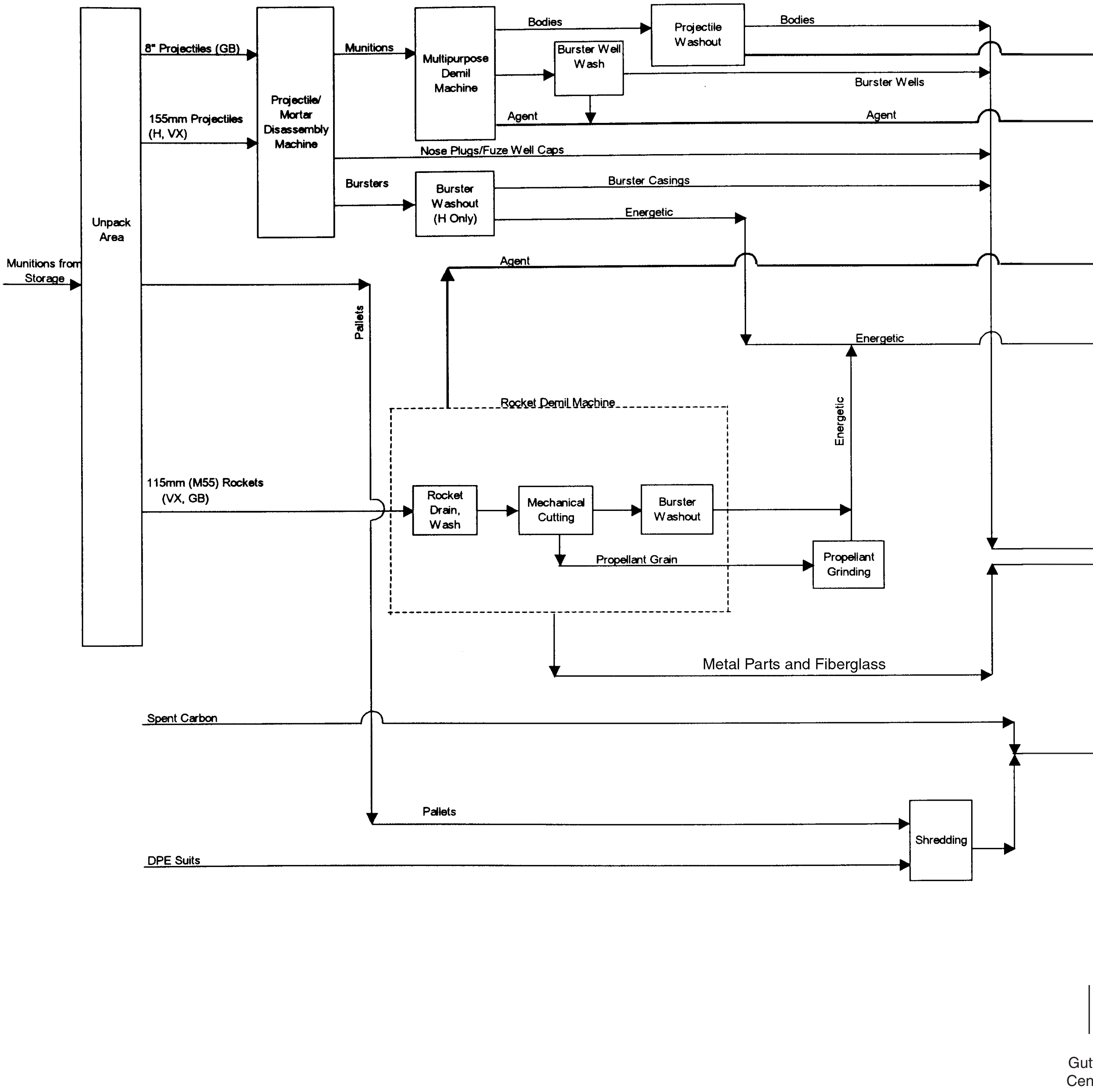

FIGURE 3-1 AEA SILVER II™ demilitarization process. SOURCE: AEA (2001a).

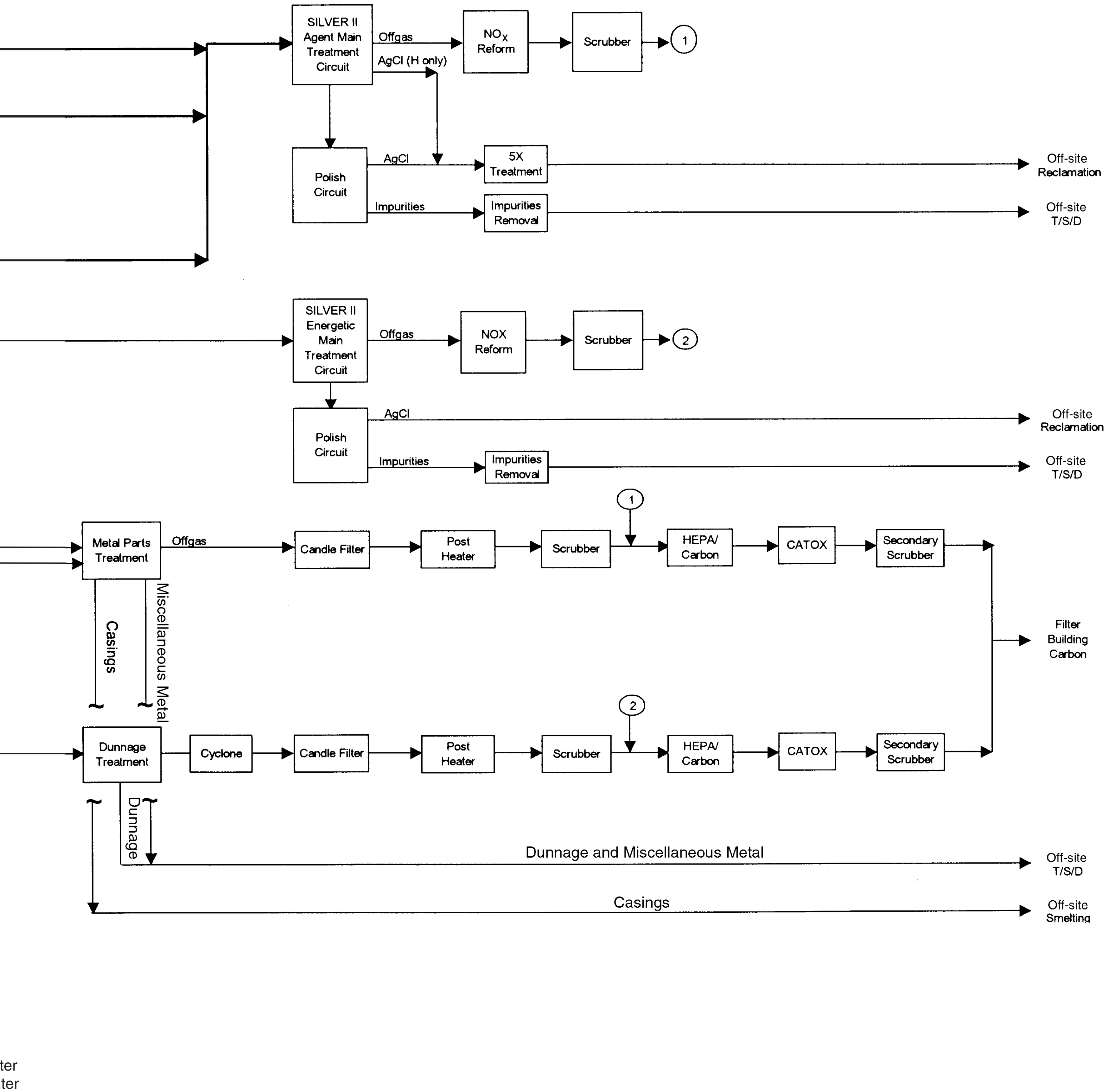
SILVER II Agent Main Treatment Circuit
Offgas
AgCl (H only)
NO_X Reform
Scrubber
1
Polish Circuit
AgCl
5X Treatment
Off-site Reclamation
Impurities
Impurities Removal
Off-site T/S/D
SILVER II Energetic Main Treatment Circuit
Offgas
NOX Reform
Scrubber
2
Polish Circuit
AgCl
Off-site Reclamation
Impurities
Impurities Removal
Off-site T/S/D
Metal Parts Treatment
Offgas
Candle Filter
Post Heater
Scrubber
1
HEPA/ Carbon
CATOX
Secondary Scrubber
Casings
Miscellaneous Metal
Filter Building Carbon
Dunnage Treatment
Cyclone
Candle Filter
Post Heater
Scrubber
2
HEPA/ Carbon
CATOX
Secondary Scrubber
Dunnage
Dunnage and Miscellaneous Metal
Off-site T/S/D
Casings
Off-site Smelting
ter
ter

TABLE 3-1 Key Plant Performance Requirements for SILVER II™ Technology at Blue Grass Army Depot

Parameter	Quantity
Total processing period	2 years (730 days)
Peak energetics throughput (lb/hr)	287.8 of M28 propellant (equivalent to 12.8 M55 rockets/hr)
Peak agent throughput (lb/hr)	166.7 of mustard agent (equivalent to 14.3 155-mm projectiles/hr)
Destruction efficiency (%)	
Agent feed	99.9999
Energetics feed	99.999
Electrolyte composition	
Anolyte nitric acid	8 M
Anolyte silver	0.5 M
Anolyte steady-state TOC	3,000 mg/L in agent and energetics, main or primary circuit, 3,000 to <1 ppm in agent polishing or secondary circuit, 3,000 to <10 ppm in energetics polishing or secondary circuit
Catholyte nitric acid	6 M
Catholyte silver	1 M

SOURCE: Adapted from AEA (2001a).

submitted to PMACWA for a full-scale pilot plant at Blue Grass Army Depot (AEA, 2001a), and they highlight changes from earlier reports of the ACW I and II Committees.

Disassembly of Munitions

Munitions Unpacking

Unpacking of the munitions is performed in the unpack area (UPA), which is the same as that in the Army's baseline incineration system design (NRC, 1999).

Projectile Processing

The AEA technology package uses the baseline system projectile mortar demilitarization (PMD) machine with modification for burster and agent washout (AEA, 2001a). After removal of the nose closure, fuze adaptor, and fuze cup, the burster tube is removed from the agent cavity and transferred to the burster washout machine (BWM), which uses high-pressure water to remove the energetic material from the burster tube. Water is added to the resulting slurry as it is drained to achieve an energetic concentration of 20 weight percent. The slurry is then pumped through an inline static macerator and into a buffer storage tank for the SILVER II™ energetics destruction system.

The agent is then drained from the projectile and the cavity washed out with a water spray. The amount of drained agent is measured and sent to the buffer storage tank for the SILVER II™ agent destruction system. The burster tube and other projectile metal parts are placed in a bin for transport to the MPT for 5X decontamination.

Rocket Processing Line

The rocket dismantling machine (RDM) proposed by AEA is based on the baseline system design but has significant modifications, including these:

- a modified punch-and-drain station having a hollow top punch to allow water or low-pressure steam to wash out gelled or viscous agent
- a tube-cutting machine to cut the rocket in its shipping tube into four segments after removing agent
- a water jet washout station to wash energetic materials out of the burster tube
- a mechanical extractor to remove the propellant grain in one piece for subsequent size reduction in a propellant grinder (AEA, 2001a)

Tests to demonstrate the operability of these modifications would be done at the vendor's plant prior to shipment or during systematization of a constructed facility using the SILVER II™ technology.

Each rocket in its shipping and firing tube is manually removed from its storage pallet in the UPA and transferred to a metering device to ensure that it is oriented with the warhead assembly heading first into the explosion containment vestibule (ECV). The metering device feeds each rocket into the ECV and from the ECV into the RDM. Two clamps on the RDM engage and grip the rocket tube near the fuze and near the connection between the bottom of the warhead and the top of the rocket motor, as in the baseline configuration. Once gripped in place, the rocket is punched once on top and twice on the bottom, and the agent drains through the two bottom punches to the agent weigh tank. Hot water or low-pressure steam is then passed into the agent cavity through the hollow top punch to wash out additional agent and minimize agent loading in the metal parts treater (MPT). The agent that drains from the cavity through the two bottom punches is pumped to the agent weigh tank and then to the agent buffer storage tanks before it is fed to the agent SILVER II™ processing system. Rinse water or steam/condensate and residual agent go directly to a buffer storage tank for the SILVER II™ process (AEA, 2001a, 2001b).

When processing GB rockets, the agent weigh tank control system provides a signal indicating the quantity of agent drained from the warhead. If less than a certain minimum percentage of the agent has been drained, the GB is assumed to be gelled or crystallized inside the warhead. Then, low-pressure, saturated steam (15 psig and less than 249.5°F) is fed through the punch head to rinse out the residual agent. When processing VX rockets, after draining the agent, low-pressure water is fed through the punch head to rinse residual agent out of the warhead.

After the agent is washed out, the rocket is rotated 90 degrees about its longitudinal axis to minimize leakage of liquid remaining in the cavity. The rocket is then advanced to the rocket cutting station, where it is clamped into the rocket rotator head and then rotated about its longitudinal axis. The first of three tube cutters cuts through the firing tube and rocket body and separates the fuze from the rocket, exposing the top of the burster well cavity. The severed fuze and associated firing tube section are dropped through a chute and conveyed to a bin for transport to the MPT. A power lance with high-pressure (~15,000 psig) water flowing from multiple nozzles is used to wash the explosive from the burster tube as the remaining section of the rocket in its firing tube is rotated. The washed-out energetic material is pumped through an inline macerator to form a slurry and collected in a tank and mixed with additional water to reduce the energetics content to 20 weight percent. The slurry is then sent to the energetics buffer storage for the SILVER II™ process.

Additional tube cutters then cut the warhead and the fin assembly from the motor casing. After both of these cuts have been made, the motor casing is moved to another station, where the attached antiresonance rod assembly is removed from the propellant grain. The antiresonance rod assembly and igniter are then transferred into a metal parts bin for transport to the MPT, and the motor casing section is moved to another station, where the rolled edge on the casing is expanded. A pusher assembly then forces the propellant grain from the motor casing, and the grain drops into the propellant grinder hopper. The warhead, fin assembly, and motor casing, along with associated firing tube segments, are dumped into a metal bin via the collection chute and conveyed to the MPT for 5X decontamination.

Preparation of Energetic Materials for the SILVER II™ Process

Slurries of energetic materials from two sources are treated in the SILVER II™ process: the explosive in the rocket and projectile burster tubes (which may be either Composition B or tetrytol) and the ground-up M28 propellant grain in the rocket motors.

The propellant grain is transferred through two interlocked blast gates into the grinder, where it is ground up under water to produce a slurry with particles having a maximum dimension of $^1/_4$ inch. The slurry is discharged through a pipeline in a manner that limits the content of the pipe to no greater than 20 weight percent propellant. The energetics slurry then flows into the energetics slurry feed tank (AEA, 2001a).

The uniformity of the energetics slurry, which may at times be combined propellant and explosive, is assured by using a high-shear vortex mixer and a conventional stirrer in the energetic slurry feed tank. The high-shear vortex mixer completes the size reduction of the energetics from burster washout and propellant grinding in preparation for SILVER II™ treatment. The stirrer ensures that all the slurry in the tank passes through the mixer head. The feed tank and slurry piping are trace-heated to prevent cold spots and precipitation or plate-out of energetics materials (AEA, 2001a).

The energetics slurry is transferred from the energetics slurry feed tank into the anolyte feed tank through a hydrocyclone. The concentration of the energetics in the hydrocyclone underflow is nominally 40 weight percent. This underflow drains into the anolyte tank. The overflow, depleted in energetics, goes to a second hydrocyclone. The underflow from the second hydrocyclone, still containing substantial quantities of energetics, returns to the energetics slurry feed tank. The overflow from the second hydrocyclone goes to the propellant grinder, where it makes up part of the water needed to slurry the incoming propellant grains.

Preparation of Agent for SILVER II™ Treatment

The recovered agent from the projectile and rocket processing lines is pumped to the agent feed system, consisting of a tank and a pump (not shown in Figure 3-1). Dilute nitric acid (HNO_3) from the NO_x reformer is added to the agent tank depending on the water volumes. (Water additions and other reagent additions achieve an 8 M HNO_3 concentration and a 0.5 M $AgNO_3$ concentration for the anolyte feed stream.) An agitator is provided to ensure homogeneity of the contents. The tank is maintained at a slight negative pressure relative to the Category A area[2] by venting to the anolyte offgas condenser. A nitrogen blanketing system is provided, with a regulator set at a slight negative pressure.

Treatment of Agent and Energetics by the SILVER II™ Process

Overall Chemistry

The SILVER II™ process is based on the highly oxidizing nature of Ag^{2+} ions in an aqueous HNO_3 solution. Ag^{2+} ions are among the strongest oxidizing agents known. HNO_3 is also a strong oxidizing agent and contributes to the overall destruction process, although the amount of HNO_3 that reacts appears to be less than 5 percent of the reaction required for total destruction (AEA, 2001b).

In each of the SILVER II™ cells, a pair of electrodes (anode and cathode) is housed in a compartment within the cell. A semipermeable membrane is placed between the electrodes. The membrane maintains electrical continuity between the electrodes and prevents mixing of the anolyte and catholyte solutions. The electrochemical cells operate at 190°F and essentially atmospheric pressure. Direct current is applied to the electrochemical cells at a potential of 2 volts, resulting in an electrochemical reaction that generates Ag^{2+} ions.

Since the Ag^{2+} ions simply mediate the reaction process, the overall reaction across the anolyte and catholyte is essentially the reaction of the organic feed with nitric acid, forming NO_x, water, CO_2, and inorganic acids:

> Organic (anolyte) + HNO_3 (catholyte) $\rightarrow$ CO_2 (anolyte) + H_2O (catholyte) + inorganic acids (anolyte) + NO_x (anolyte and catholyte)

Ag^{2+} ions generated at the anode react with the water and HNO_3 of the anolyte solution to form a range of radicals (e.g., $OH^{\bullet}$, $NO_3^{\bullet}$) that in turn oxidize the organic material in the anolyte solution completely and irreversibly to carbon dioxide, some nitrogen oxides (NO_x from the direct reaction with the acid, which proceeds to a moderate extent), inorganic ions, additional hydrogen ions (H^+), and small amounts of carbon monoxide. Ag^{2+} can also react directly with water in the anode compartment to form oxygen gas. Both Ag^+ and cationic impurities in the anolyte can migrate across the membrane to the catholyte compartment. To balance the electrochemical reaction in the anolyte, there is a corresponding cathode reaction that involves reducing nitric acid to nitrous acid, which in turn partially decomposes to NO_x gases and water. The water balance is complex and involves two countercurrent fluxes. Water is transferred across the membrane from the anolyte to the catholyte in the form of hydrated protons generated as a product of the anode reaction. Water also flows in the opposite direction from the cathode compartment to the anode compartment owing to the lower acidity (higher water concentration) in the cathode compartment. A more detailed discussion of the SILVER II™ reaction chemistry is given in the ACW I Committee report and is not discussed further in this report (NRC, 1999).

A summary block flow diagram depicting the SILVER II™ systems applicable to the processing of both agent and energetics is given in Figure 3-2. The flow circuit for AgCl separation (shown by dotted lines) is used only when processing H or HD, which contain chlorine. As shown in Figure 3-2, the SILVER II™ systems incorporate internal recycle streams: (1) from the NO_x reformer to replenish nitric acid lost from the anolyte and catholyte solutions and (2) from the catholyte circuit to return silver ions, unreacted organic

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[2] An area where agent contamination is to be expected. Personnel performing maintenance in Category A areas must be in DPE suits. Category B areas require the highest level of respiratory protection but a lesser degree of skin protection. Category C areas are those where the concentration of airborne substances are known and the criteria for using air-purifying respirators are met.

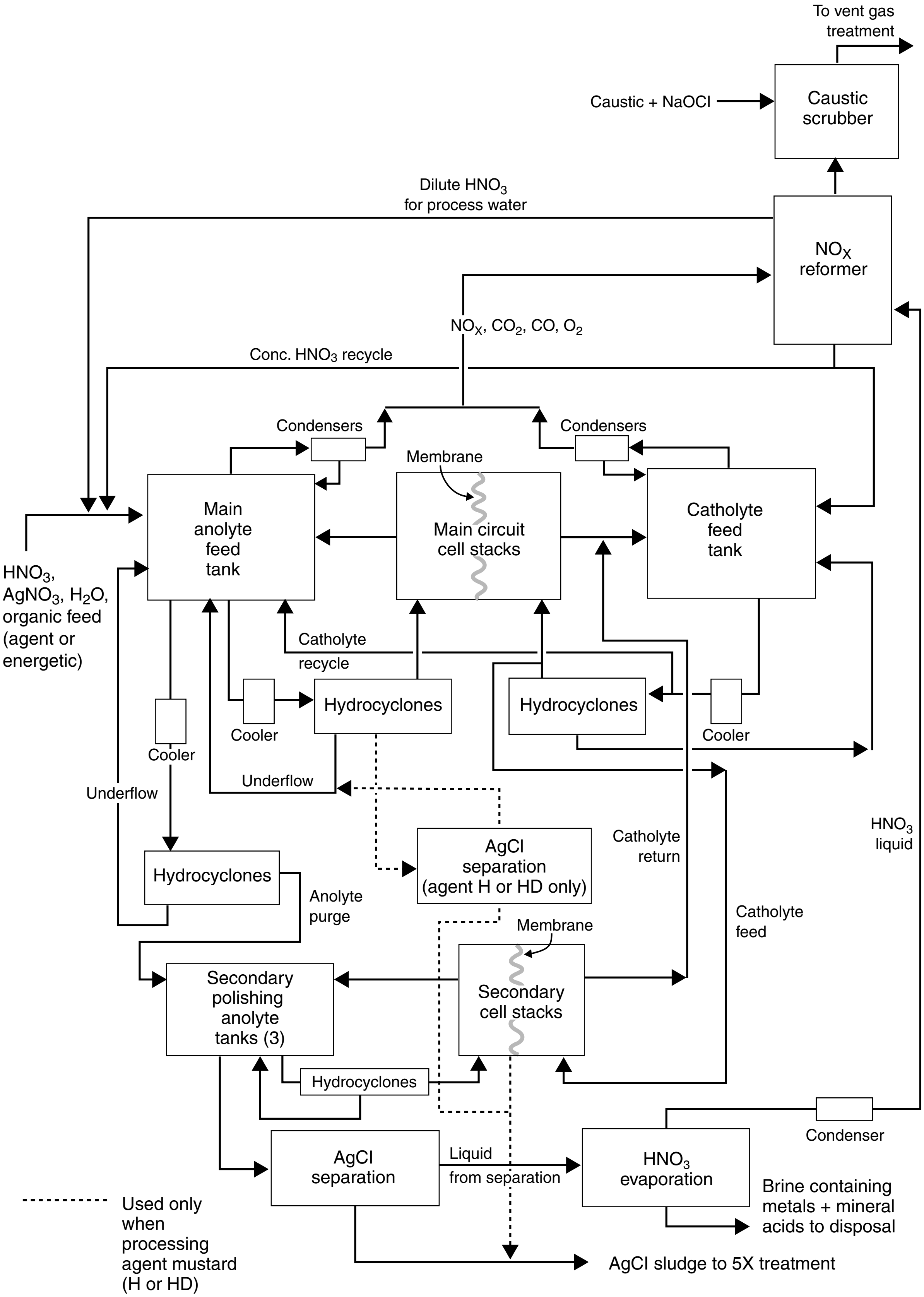

FIGURE 3-2 SILVER II™ process system for agent or energetic destruction.

material, and mineral acid ions that have crossed the membrane from the anolyte to the catholyte circuit. There is also an anolyte purge stream to the impurities removal system (IRS).[3] The anolyte purge stream is processed by the IRS in a batchwise sequence through SILVER II™ electrochemical cells in a polishing circuit, where remaining organics are destroyed. After polishing, the purge stream content undergoes AgCl precipitation and separation using hydrocyclones and a centrifuge. The liquid from AgCl precipitation is then sent to an HNO_3 evaporation operation for recovery of the nitric acid and to concentrate the impurities in the evaporator bottoms or brine stream. Batchwise processing of the anolyte purge stream is accomplished by collecting the stream in one of three tanks and then processing the contents of that tank through the preceding three steps. The contents collected in each of the other two tanks are similarly processed in sequence.

Process Outputs

Solids. Treatment of mustard agents by the SILVER II™ process results in the precipitation of silver chloride, which, if not removed, could cause cell plugging. This precipitate is removed from the main circuit in a slurry underflow from hydrocyclones in the anolyte feed stream circuit. Subsequently, this slurry is dewatered in a centrifuge to approximately 50 weight percent AgCl, with the remainder of the slurry consisting of water, nitric acid, silver nitrate, and trace amounts of other materials in the anolyte loop.

The SILVER II™ polishing circuits for both agent and energetics systems continuously produce a larger stream of AgCl than what is produced in the main circuit when H or HD feeds are being processed. It should be noted that essentially all silver in the main circuits from processing any agent or energetics is eventually removed in the AgCl precipitation step following the polishing of agent and energetic anolytes. In this operation, hydrochloric acid (35 weight percent) is added to the feed tank containing the polished anolyte to form a slurry that contains AgCl precipitate. The slurry is passed through a hydrocyclone and the underflow is sent to a centrifuge for dewatering to approximately 50 weight percent AgCl. The hydrocyclone overflow of the slurry consists of water, HNO_3, trace amounts of other materials in the anolyte loop and about 2 percent unreacted silver nitrate. The AgCl sludge is then combined with AgCl sludge from the main circuit when processing H or HD feeds. This resulting sludge stream is then washed with 18 percent NaOH to neutralize the remaining acid. The neutralized sludge is then thermally treated to a 5X decontamination level in an enclosed auger heated by hot oil. The 5X silver chloride is sent to an off-site silver reclaiming contractor, which then returns the silver to the plant for preparing silver nitrate makeup.

The liquid overflow from the hydrocyclones and the AgCl centrifuge in the polishing circuit is sent to an evaporator for recovery of HNO_3, which is condensed from the overhead vapor stream and pumped to the NO_x reformer. AEA states that the evaporator is expected to recover approximately 70 percent of the HNO_3 in the evaporator feed for subsequent recycling to the reformer (AEA, 2001a). The recovery percentage will vary depending on the amount of metals present as impurities in the feed and the final concentrations of metal and sulfuric, phosphoric, and hydrofluoric acids. For agent feed streams, the concentration of acids other than nitric places a limit on the evaporator operation, because carryover of these other acids in the evaporator vapor may occur. Energetic feed streams have no S, P, or F, and the acidic recovery is limited only by the solubility of iron and aluminum nitrates in the strongly acid evaporator bottoms. The bottoms containing mineral acids and metals are then treated, tested for agent, and prepared for off-site disposal.

Liquids. Liquid effluents are expected to be limited to excess concentrated HNO_3 of undefined purity that will be produced when processing energetics.

Gases. The anolyte offgas from the primary and polishing systems contains O_2, CO_2, and NO_x and is saturated with water vapor and HNO_3 at 176°F. Small, variable amounts of CO are also formed from the oxidation, the amount depending on the organic feedstock. Most volatile organics from decomposition of feed material are expected to be condensed with water and acid vapor in the main and polishing circuit offgas condensers. These anolyte offgas condensers from the main and polishing circuits are cooled with chilled water to operate at 91°F and 40°F, respectively. The condensates are returned to the anolyte in the main system.

The offgas from the catholyte feed tank contains O_2

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[3] The IRS consists of the secondary polishing circuits of the SILVER II™ process and the associated components, including hydrocyclones, AgCl separation equipment, and the HNO_3 evaporator.

(sparged into the catholyte feed tank for use in the NO_x reformer), plus NO and NO_2, and is saturated with water vapor and HNO_3 at 176°F. This offgas first passes through a condenser to recover water and acid, which is returned to the catholyte feed tank. The anolyte and catholyte offgas streams are combined downstream of their respective condensers and flow to the NO_x reformer to produce concentrated nitric acid. Offgas from the reformer system is scrubbed with NaOH/NaOCl solution to remove unreacted NO_x. The scrubbed NO_x reformer offgas, which contains CO_2, volatile organic compounds (VOCs), and small amounts of CO, flows to the gaseous effluent treatment systems (as shown in Figure 3-1)—of the MPT in the case of agent processing and of the dunnage treatment system (DTS) in the case of energetics processing. There, thermal treatment and CATOX units oxidize the CO and organics. The gaseous effluent treatment systems are described in a later section.

Process Equipment

The electrochemical cell used for the SILVER II™ process is based on a standard industrial cell design. Detailed discussion of the design, installation, and operation of the cells is provided in the EDP report, Volume 2 (AEA, 2001a).

For a full-scale facility at Blue Grass, the main processing circuits would consist of six operating cell stacks of 48 cells each for agent and six operating stacks of 42 cells each for the energetics. In addition, the SILVER II™ processes for both agent and energetics each have polishing systems consisting of three cell stacks, each configured identically to the main circuit stacks. The total power requirements for the primary (main) circuit and secondary (polishing) cell stacks for both the agent and energetics systems in the AEA SILVER II™ process are given in Table 3-2. In addition to the operating stacks, both the agent and energetics systems have one installed spare cell stack. Five more cell stacks are stored as spares. All operating stacks are connected in series to a single direct current power supply. The cells within each stack are connected in parallel. The installed spare is electrically shorted and isolated from the anolyte and catholyte circuits by valves. Demo II testing established that the electrodes and membranes can operate at a current of up to 2 kiloamperes per electrode pair (AEA, 2000).

The anodes are platinized titanium (titanium plated with 5 microns of platinum); the cathodes are also titanium plated with 2.5 microns of platinum. All gaskets are Viton GF (peroxide grade), and the cell membranes are DuPont Nafion 324. Flow through the cells is in parallel using manifolds with $^1/_4$-inch fluid-flow inlet port orifices to provide equal flow to all cells.

As noted in Table 3-2, the secondary polishing system cells require electrical power at 50 percent of the level of the main circuit cells because the electrochemical organic destruction efficiency (i.e., the fraction of the electric current used to oxidize organics) decreases sharply as the organic concentration is reduced from an initial concentration of ~3,000 ppm to 1 to 10 ppm. The average destruction efficiency of the polishing cells is expected to be 2 to 10 percent, with the main competing reaction being the oxidation of water. Consequently, the offgases from the polishing system cells

TABLE 3-2 Summary of SILVER II™ Plant Electrochemical Cells

				Cell Stack Quantities			
Description	Power (kW)	Operating Pressure (psig)	Material	Electrode Pairs/ Cell Stack	In Service	Installed Standby	In Storage
Agent							
Primary electrochemical cell	1,120	~0	PVDF/Ti/Pt	48	6	1	3
Secondary electrochemical cell	560	~0	PVDF/Ti/Pt	48	3	0	2
Energetics							
Primary electrochemical cell	991	~0	PVDF/Ti/Pt	42	6	1	3
Secondary electrochemical cell	495.5	~0	PVDF/Ti/Pt	42	3	0	2

SOURCE: AEA (2001a), Volume 2.

contain a high percentage of oxygen, which flows to the NO_x reformer with the other offgases from the main circuit. This oxygen, along with the oxygen that was sparged into the catholyte feed tank, is used to generate nitric acid in the reformer.

AEA is primarily relying on lining all vessels and piping in the agent SILVER II™ system with polytetrafluoroethylene (PTFE, or Teflon) or PFA for corrosion resistance. To prevent HF from returning to the SILVER II™ system via the nitric acid stream from the evaporator, AEA is evaluating methods of keeping the HF in the liquid phase of the nitric acid evaporator bottoms by using a complexing chemical. The method of trapping the fluoride in the evaporator bottoms had not been selected at the time this report was prepared. Therefore, additional unit operations yet to be defined may be required to deal with this issue.

Processing and Treatment of Metal Parts, Dunnage, and Other Solid Waste

Metal Parts Processing

Metal parts, fuzes, and fiberglass segments of shipping and firing tubes from the munitions disassembly lines, and drums containing particulate matter from the candle filters and cyclones of the gaseous effluent treatment systems are conveyed in bins to one of two MPTs for decontamination to the 5X level. Normally only one MPT is required; however, AEA has identified disposal campaigns where a second MPT would also be used (AEA, 2001a). In the MPT, metal and fiberglass parts are heated to over 1000°F for at least 15 minutes.

The MPT is a modified industrial oven with the following four zones:

- zone 1 for purging air with nitrogen and providing an inert gas barrier for the following zones
- zone 2 for warming the materials contained in bins to 300°F to volatilize organic materials from the fiberglass and other materials being treated
- zone 3 for heating the parts above 1,000°F for more than 15 minutes to achieve 5X decontamination
- zone 4 for cooling and verifying that the parts are acceptable for release

Zones 1 and 4 use inert gas atmospheres and are separately exhausted directly to the carbon filters of the MDB HVAC system. Zones 2 and 3 operate with steam atmospheres and are exhausted through a dedicated gaseous-effluent treatment system to remove entrained water and control the level of trace organics released. (The system is described in a later section.) Steam used in zones 2 and 3 is recirculated through electrically heated exchangers or heaters. The cycle time for each batch is approximately 1 hour to ensure that all of the contents of the metal parts bins reach 1,000°F for 15 minutes and that all organic material in the fiberglass shipping tube segments is decomposed. This thermal treatment also is expected to decompose (possibly through initiation) fuzes and igniters contained in the metal parts bins. Temperature markers[4] are placed in each batch to verify that the required time at temperature has been achieved for 5X decontamination. Treated parts are moved to zone 4, where they are cooled and the atmosphere is sampled to verify that the parts are free of agent to the limits established for release off-site. If agent is detected above release limits, the bin is recycled through the MPT. After cooling, the metal parts are sent to a metal parts crusher and then off-site for disposal or recycling.

Dunnage Processing

Wood dunnage, DPE suit material, and spent carbon are treated to the 5X decontamination level using a DTS (AEA, 2001a). The major components of the DTS include the carbon carrier medium silo, the DPE suit shredder, the wood shredder, the feed hopper, and two redundant, full-capacity dunnage treaters. The DTS design is based on a design by Parsons that uses a continuous steam treater (CST) (Parsons, 2001). Wood pallets and DPE suit material are processed in their respective shredders and the shredded material transferred to one of two feed hoppers along with carbon carrier media. The feed hoppers discharge into the dunnage treater through a rotary air lock. Each feed hopper will feed either of the two dunnage treaters. Normally, one dunnage treater is on standby.

Each dunnage treater is a 4-ft-diameter horizontal vessel made of Hastelloy C276 material, within which

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[4] Devices inserted in material being treated that undergo observable physical change after experiencing the required time at temperature.

is mounted a screw auger to transport wood dunnage, spent carbon, or a mixture of carbon carrier media and either wood chips or shredded DPE suits from one end to the other. The treater is operated under a slight negative pressure, and high-temperature, low-pressure steam sweeps offgases generated by thermal decomposition into the offgas treatment system and contributes to thermal decomposition of the dunnage. The residence time of the dunnage material inside the treater is sufficient to ensure that all of the material contained in the enclosed auger is heated to the conditions required for 5X decontamination. In EDS testing of the CST, residence times of 1 hour were found to be adequate (Parsons, 2001). Actual residence times in the dunnage treater would be determined by using temperature markers during start-up operations. At the discharge end of the dunnage treater, the treated material is discharged through double rotary valves to prevent air backflow into the treater. The treated dunnage drops into a discharge hopper, where it is quenched with a water spray to extinguish any residual embers. Steam generated from the quench water is returned to the dunnage treater as part of the sweep steam. The hopper atmosphere is then sampled to ensure agent levels are below the limits set for discharge. The treated dunnage char is conveyed outside the MDB to a storage silo and transported to off-site disposal.

Each dunnage treater is electrically heated using multiple, equally spaced high-performance mineral-insulated band heaters.[5] The entire dunnage treater, including heaters, is insulated by ceramic fiber insulation blankets contained within a steel shroud. Since the dunnage treater is operated under only slightly negative pressure, all openings are equipped with seals to prevent fugitive fumes from escaping. The slight negative pressure ensures proper flow of steam and the gases generated in the treater. The dunnage treater operates at temperatures from 1,000 to 1,100°F. Using start-up data from thermal markers, the rotational speed of the screw conveyor or auger is adjusted to provide the residence time required for 5X decontamination. As the screw rotates, the solid material is conveyed through the length of the treater.

Offgases and volatile materials are primarily VOCs, CO, CO_2, and H_2O generated by thermal decomposition and, to a lesser extent, steam reforming reactions with the dunnage material (Parsons, 2001). When processing DPE suit material, HCl will also be present in the sweep gas stream. The process gases are treated in a gaseous effluent treatment system (described in the following section).

Processing and Treatment of Gaseous Effluent Streams

Two emissions control systems are used prior to discharging the gaseous effluents to the carbon filters in the MDB HVAC system. One emissions control system is provided for the gaseous effluents from the agent SILVER II™ system and the MPTs. The other emissions control system handles gaseous effluent from the energetics SILVER II™ system and the DTS (see the process flow diagram in Figure 3-1). Each system is redundant in that a system is associated with each of the two MPTs and each of the two DTSs in the full-scale design.

The two emissions control systems are very similar. In order of occurrence, the flow paths for both systems contain the following elements. Where an element is unique to one of the systems, it is so noted.

- *Cyclone*. The first element in the system for DTS gaseous effluent is the cyclone provided to separate large particulate matter that could rapidly plug the downstream candle filters. This element is not required in the system for MPT gaseous effluent. Separated particulate matter is discharged into a steel container through a rotary air lock. The particulates are processed in the MPT to ensure 5X decontamination.
- *Candle filters*. These consist of multiple high-temperature ceramic or sintered-metal candles fitted within a plenum and a nitrogen supply for online periodic back flushing. Captured particulate matter is discharged into a steel container through a rotary air lock. The particulates are processed in the MPT to ensure 5X decontamination.

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[5] Both the Departments of the Army and the Navy have used mineral-insulated band heaters for various superheating applications. For example, the Department of the Navy uses similar equipment to test chemical stability of components within artillery shells (AEA, 2001a). In addition to high watt densities, these heaters provide even temperature profiles and fairly precise temperature control.

- *Post heater*. Located immediately downstream of the candle filters, a post heater heats the offgas to a temperature of 1,000 to 1,100°F to destroy any agent that might exist in the gaseous effluent. The post heater is designed to provide a minimum residence time of 3 seconds at temperature.
- *Quench elbow and primary scrubber*. Here, the gases are scrubbed in a packed tower scrubber for particulates and acid gases, using a scrubber solution with a pH of 7.0 to 8.5. The recirculating scrubber liquor is cooled and, in turn, cools the hot gas from 1,000°F to approximately 60°F.
- *Blowers*. Effluent gases in the MPT and DTS trains then flow through positive displacement blowers. Ambient air is added to the streams to provide the oxygen that will be necessary for catalytic oxidation of organic compounds and carbon monoxide. Similar blowers are provided on the offgas streams from the NO_x reformers of the agent and energetics SILVER II™ systems so that these streams can be added to the respective MPT and DTS gaseous effluent streams.
- *HEPA filters*. A set of parallel HEPA filters removes fine mist, including residual organic aerosols that pass through the scrubber mist eliminator and condensable particulate matter that was not removed in the primary scrubber. At this point in the gaseous effluent system, offgas from the NO_x reformers for agent and energetics SILVER II™ systems is added to the offgas from the MPT and DTS systems, respectively. Offgas from the NO_x reformer of the agent SILVER II™ system is preheated prior to being added to the MPT gaseous effluent gas stream. These offgas streams increase the gas volume to be treated for the remaining elements of each emissions control train.
- *Carbon filters*. For the combined effluent from the NO_x reformers of the agent SILVER II™ system and the MPT, three drums of activated carbon, in series, are used to remove any residual agent and PCB vapor. Each of the drums in the filter contains 150 pounds of activated carbon. These filters are not used for the combined gaseous effluent from the energetics SILVER II™ system and the DTS.
- *CATOX preheaters*. Each of the combined streams on both systems is heated to approximately 500°F in an electric heater before treatment in the CATOX unit.
- *CATOX unit*. Organic vapors and carbon monoxide are destroyed in the CATOX unit of each system using a proprietary precious metal catalyst developed by Engelhard Corporation. The operating range of the catalyst is from 500 to 1,000°F. For maximum destruction efficiency (99 percent) for organic compounds, the catalyst should operate at 900°F.
- *Quench elbow and secondary scrubber*. This system is used to remove acid gases formed from the catalytic oxidation of halogenated organic compounds. The operation of the secondary scrubber is identical to that of the primary scrubber with the exception of a recirculation cooler that maintains scrubber exit gas temperature at 120°F. The secondary scrubber has a constant liquid blowdown that is collected in holding tanks and tested for agent prior to release.
- *Blower*. Following the secondary scrubber in each system is a blower that is used to maintain negative pressure throughout upstream portions of the gaseous effluent emissions control systems. The blower discharges directly to the MDB HVAC system carbon filters.

Effluent Management

The various processes and streams involved in effluent management include brine from the polishing systems evaporator, scrubber blowdown liquids, concentrated and dilute HNO_3, 5X-decontaminated metal parts, fiberglass, dunnage char, and treated process vent gas from the two emissions control systems. Hold-test-release processing is used on all solid and liquid streams (AEA, 2001a). Gaseous effluents, including processing area HVAC air streams and treated process gas, pass through MDB HEPA and activated carbon filters prior to release to the atmosphere.

By-product Recovery

By-product silver chloride from the SILVER II™ process is separated as sludge, decontaminated to a 5X level, and shipped off-site, where it is reduced to silver metal, and returned to the plant for making fresh silver nitrate for the process. Based on material balances provided in the EDP, AEA expects no excess concentrated nitric acid to be produced since it will all be returned to the SILVER II™ processes as makeup acid or used in making fresh silver nitrate (AEA, 2001a).

INFORMATION USED IN DEVELOPING THE ASSESSMENT

Engineering Design Package

On December 15, 2001, AEA Technology/CH2M Hill issued a final draft of the EDP for a full-scale pilot plant implementing the SILVER II™ technology package at Blue Grass Army Depot (AEA, 2001a). The EDP includes technical descriptions and data, drawings, a preliminary hazard analysis, and cost and schedule analyses. It has been used as the primary source of information for this assessment. Other EDP-related documents used in the assessment include the initial draft of the EDP and the study plan for the EDS submitted by AEA (AEA, 2001b, 2001c).

Overview of Engineering Design Studies and Tests

The Demo II testing phase of the ACWA program for the AEA technology package was conducted between February and October 2000. The Demo II testing confirmed the ability of the core process of the SILVER II™ technology to destroy both energetic and agent materials (AEA, 2000). Although the overall Demo II testing was deemed successful, areas were identified that required further testing prior to progressing to a full-scale pilot plant design.

In April 2001, AEA issued its study plan for the EDS II program (AEA, 2001c). This plan set forth a detailed test program to provide the additional information required to enable the SILVER II™ technology to progress to a full-scale pilot plant design stage. The test program also assumed that the results of EDS tests conducted by other technology providers in the ACWA program could be used where appropriate. Table 3-3 lists all tests and studies pertinent to AEA's SILVER II™ technology package.

The EDS II test program includes testing and design studies needed for a preliminary design of a full-scale pilot plant, a preliminary hazard analysis, development of a full-scale cost estimate, and a schedule for construction and operation of a facility employing the AEA technology.

The test program consisted of two discrete packages (AEA, 2001c):

- *Engineering tests*. These tests demonstrated the operation of modifications to the existing energetics feed system and SILVER II™ plant to eliminate the operational problems discussed in AEA's final report for Demo II (AEA, 2000). They also demonstrated the long-term operation of a 12-kW SILVER II™ system on feeds of agent simulant and energetics.

TABLE 3-3 EDS Tests and Studies Used for SILVER II™

Title	Organization and Report Number
Engineering tests	
Propellant grinding test	Eco Logic, Inc., no report number, November 5, 2001
M28 and Comp B SILVER II™ processing trials	AEA, 20/3034/13/006
DMMP (agent simulant) 500-hr run SILVER II™ processing trials	AEA, 20/3034/13/006
Laboratory-scale tests	
High-shear mixer trials for the 12-kW SILVER II™ plant	AEA, 20/3034/002
Hydrocyclone trials for the 12-kW SILVER II™ plant	AEA, 20/3034/001
FM01 SILVER II™—organic transfer	AEA, 20/3034/007
Fluoride mobility report	AEA, 20/3034/011
Fluoride removal system	AEA, 20/3034/009
Cell membrane life tests	AEA, 20/3034/13/004
Investigation into the 12-kW SILVER II™ plant membrane failures	AEA, 20/3034/13/005
Silver recovery from silver chloride	AEA, 20/3034/13/003
Evaporator trials for the full-scale SILVER II™ EDP plant	AEA, 20/3034/13/008
CST/CATOX testing	Parsons, CW7/I&T/737204EDS
Desktop studies (literature surveys)	
Water jet cutting and burster washout for chemical agent munitions	El Dorado Engineering, Inc., no report number, December 3, 2001
Literature review on energetic slurry concentrations and handling	El Dorado Engineering, Inc., no report number, August 21, 2001

TABLE 3-4 AEA 12-kW SILVER II™ Tests

Feed	Total Quantity (kg)	No. of Tests	Test Duration	Description
TBP/OK	30	Chemical systemization	4 days	Liquid, easily pumpable, not water-miscible
DNT	83	Energetic systemization	7 days	Granular form, slurried, 20 wt% w/water
Composition B	60	1 workup	5 days	Granular form, slurried, 20 wt% w/water
Composition B	123	1	259 hr	Granular form, slurried, 20 wt% w/water
Composition B/M28	23/113	1	Included in 259 hr above	Granular form, slurried, 20 wt% w/water
DMMP (agent simulant)	156	1	500 hr	Liquid, easily pumpable, not water-miscible

SOURCE: AEA (2001d, 2001e).

- *Laboratory-scale tests and desktop studies*. These activities provided data needed to complete a full-scale design for the EDP for a facility at Blue Grass Army Depot.

Summaries of the tests and studies performed and used in the development of the final EDP for AEA's SILVER II™ technology package are discussed in the following sections.

Engineering Tests

Propellant Grinding Test

Specific objectives and results of propellant grinding tests performed in support of the EDS II program for the Eco Logic technology package are discussed in detail in Chapter 4. The description of the tests and the committee's evaluation of the results presented in Chapter 4 are also applicable to the use of this unit operation in the SILVER II™ technology package.

Long-Term 12-kW Testing on Feeds of Energetics and Agent

The EDS II test program for the 12-kW SILVER II™ plant involved the operation of two campaigns, the first to destroy Composition B and then a mixture of Composition B and M28 propellant and the second to destroy the agent simulant dimethylmethylphosphonate (DMMP). For the EDS II tests, the 12-kW plant used in Demo II tests was upgraded to incorporate lessons learned from Demo II and demonstrate proposed solutions for problems that were encountered during Demo II. A summary of EDS II tests using the 12-kW system is given in Table 3-4, and the test setup for this system is shown in Figure 3-3. Subsequent to authorizing the tests listed in Table 3-4, PMACWA authorized a demonstration of tetrytol destruction using the EDS II equipment and operating conditions. Tetrytol destruction had been problematic in prior Demo II tests. Results were not available at the time this report was prepared.

SILVER II™ Processing Trials with M28 Propellant and Composition B

The EDS II test objectives for the energetics feed system were to design, build, and test a system that allows consistent operation of the SILVER II™ plant, including the following (AEA, 2001c):

- dispensing known volumes of energetics slurry with up to 20 percent (by weight) solids into the SILVER II™ anolyte feed tank
- monitoring the level of the energetics slurry within the feed tank
- operating the energetics system for long periods with limited maintenance
- verifying that the slurry is homogeneous and that stratification does not occur

The EDS II test objectives for the Composition B/M28 propellant destruction campaigns were these (AEA, 2001d):

- Verify long-term, continuous operability, reliability, and maintainability (i.e., operation of the full length of the test without unintended shutdown)

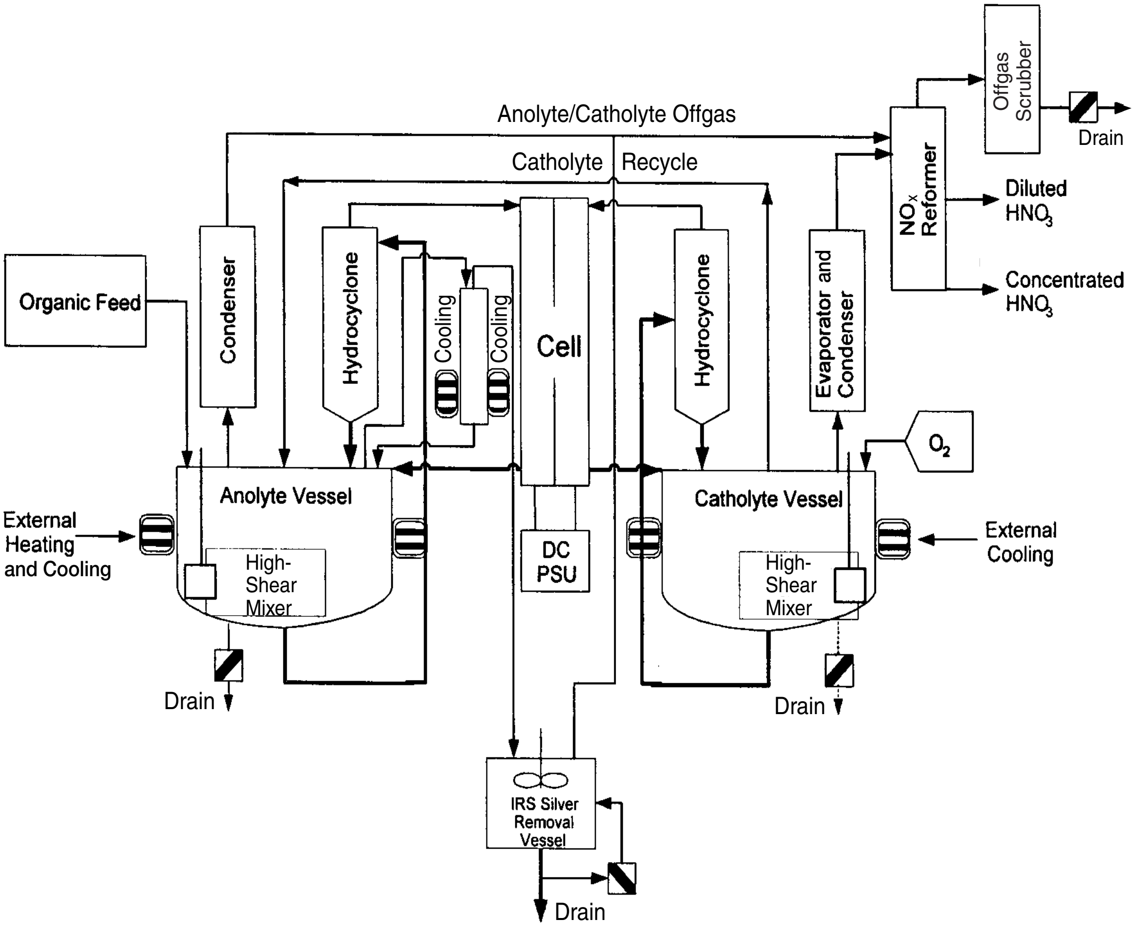

FIGURE 3-3 AEA 12-kW SILVER II™ test setup for EDS II. SOURCE: AEA (2001d).

of the SILVER II™ system as proposed for the full-scale version.

- Demonstrate that organic materials, silver, acid, and water in the catholyte circuit can be effectively managed over prolonged operational periods.
- Demonstrate that process impurities that build up in the anolyte circuit can be effectively managed over prolonged operational periods.
- Determine the cell current efficiency to be used in the full-scale design.
- Verify that system modifications (i.e., high shear mixers and hydrocyclones) effectively treat organic material.
- Demonstrate that the impurities removal system (IRS) design for the 12-kW plant is applicable to the full-scale design and develop the data necessary for the design of the full-scale IRS.
- Confirm and supplement Demo II process effluent characterization.
- Determine impact of operations on materials of construction to be used in a full-scale system—for example, PTFE- and PFA-lined piping.
- Validate the ability of the SILVER II™ unit operation to achieve a destruction and removal efficiency (DRE) of 99.999 percent for Composition B—cyclotrimethylenetrinitramine (RDX) and trinitrotoluene (TNT).
- Demonstrate the operation and performance of key process components for future scale-up, including instrumentation, valves, pumps, electrochemical cells (electrodes and membranes), the full-height NO_x reformer, and the offgas scrubber operating in conjunction with the NO_x reformer.
- Demonstrate the ability to recycle, reuse, or dispose of nitric acid.
- Characterize gas, liquid, and solid process streams of the SILVER II™ process for selected chemical constituents and physical parameters and for hazardous and toxic compounds.

After systemization, a test run of approximately 145 hours was made on Composition B using a slurry feed control system specifically designed for the demonstration test. A radar level probe successfully monitored the level in the feed system after optimizing the amount of surfactant added to wet the Composition B while avoiding foaming of the slurry. No foaming occurred during the treatment of the mixed energetics, so no surfactant was added. Subsequently, a test run of approximately 145 hours was made on a mixture of Composition B and M28 propellant similar to that expected when processing the energetics from rockets.

During both tests, the chemical environment within the anolyte and catholyte circuits was successfully maintained by using the catholyte-to-anolyte recycle system and by periodically purging a stream of anolyte to simulate operation of the IRS.

The hydrocyclones and high-shear mixers operated throughout the testing. During energetics destruction, no deposits of crystalline material or flow blockages were observed with the hydrocyclones and high-shear mixers in place.

Plant maintenance during the tests was limited to correcting a few small leaks. At the end of the Composition B part of the run, a transmembrane breach occurred as a result of membrane shrinking and tearing. This was remedied by using a membrane soaking solution that contained aluminum nitrate to preshrink the membranes prior to placing them in the cell. No further transmembrane breaches were experienced in the remaining runs with energetics.

The offgas system performed within specification. The postreformer NO_x level was around 2,000 ppm, while the plant offgas NO_x level was less than 100 ppm for the Composition B part of the run and around 400 ppm when mixed energetics feed was used.

Based on the test runs, AEA concluded that catholyte-to-anolyte recycle and periodic removal of an anolyte purge stream (to simulate IRS operation) controlled the anolyte acid concentration, as well as the catholyte silver and organics concentrations. The catholyte silver ion concentration was held at the target 1 M level proposed for the full-scale system.

Accidental operation with an anolyte total organic concentration (TOC) of 6,000 mg/L (rather than the intended 3,000 mg/L) during a test run of Composition B oxidation demonstrated an electrochemical efficiency of approximately 70 percent versus the target 40 percent. This is consistent with the behavior of the plant during the oxidation of M28 propellant in Demo II. The committee notes that other research in mediated electrochemical processes has shown that the coulombic or electrochemical efficiency of the process is directly proportional to the concentration of the material being oxidized and rapidly decreases as the destruction approaches 100 percent—that is, as the concentration of oxidizable material becomes very small (Chiba et al., 1995).

The high-shear mixers and hydrocyclones in the anolyte and catholyte circuits, along with trace heating and elimination of dead legs, successfully maintained the particulate energetics in the anolyte and catholyte circuits in a finely divided state. AEA concluded that this result confirmed the ability of SILVER II™ to handle solids at its design loading (AEA, 2001a). However, AEA also noted that the small particle size resulted in inconclusive performance results for the anolyte hydrocyclone. Therefore, some uncertainties still exist with regard to successful demonstration of solids handling.

There was no accumulation of metals in either the anolyte or catholyte circuits when a spike of metals was fed with the M28 propellant to simulate particles from antiresonance rods. AEA attributes this success to the use of the catholyte-to-anolyte recycle and the anolyte purge operation. Lead, present in M28 propellant as lead stearate (approximately 0.5 weight percent), was oxidized to lead oxide (PbO_2) and did not accumulate in solution. Lead oxide was found on the electrode surfaces and as a deposit in the bottom of the cell cavities (AEA, 2001d). A demonstration test successfully removed the lead oxide using an offline formic acid wash of the cell electrode cavities. This is the planned approach for removing accumulating lead oxide. No lead material balance was provided.

The NO_x reformer operated for most of the run with a bottoms composition in the boiler reasonably close to the target 60 weight percent acid used in the full-scale EDP design. The offgas scrubber NO_x was typically less than 100 ppm during the Composition B phase of the run but somewhat higher during the Composition B plus M28 phase. Based on the available analytical data, the most abundant organic breakdown product from Composition B was trinitrobenzene. Alkyl nitrates were found at a low ppm level in the plant offgas and the scrubber liquor, as was observed during previous trials.

The committee notes that trinitrobenzene is an energetic material and that safety considerations warrant a thorough understanding of the expected quantity and location of this material.

DMMP 500-Hour SILVER II™ Processing Trials

The objectives of trials with the agent simulant DMMP were similar to those with energetics (AEA, 2001e). The trials did not simulate the conditions associated with fluorine- or chlorine-bearing agent feeds. Shortly after beginning the DMMP trial, breaches of the membrane were again experienced. Investigation of these breaches is discussed in detail in reports prepared by AEA and Arthur D. Little Corp. (AEA, 2001f; ADL, 2001). The basic causes of failure (discussed in more detail in a later section on cell membrane failure analysis) were these:

- membrane contact on the cathode surface when the formation of a gas, possibly from decomposition of the hydroxylamine in the membrane material, causes it to swell
- holes in the membrane caused by arcing from dendritic silver crystals growing on the catholyte electrode (the silver deposition was believed to be caused by low nitric acid concentration, which had dropped to approximately 1 M, in the catholyte solution during the test)

AEA subsequently implemented the following corrective actions:

- installation of spacers between the membrane and cathode to provide greater separation between the membrane and the cathode
- monitoring of nitric acid concentration as well as total acid concentration in the catholyte, and control of the nitric acid concentration in the 6 M to 8 M range
- pressure control to ensure that electrolyte pressure in the cathode space is higher than in the anode space

With these corrective actions in place, the DMMP run was completed without any further interruption, and test results were within expected parameters. Using normal feed concentration and rundown operations, agent simulant destruction to the required 99.9999 percent was completed.

A PTFE-lined pipe was placed into the anolyte loop to demonstrate the resistance of PTFE (Teflon), a material of construction to be used in the full-scale system, to electrolyte corrosion attack. This pipe was removed and inspected at the end of the DMMP run. Visual inspection showed no evidence of corrosion of the pipe or damage to the liner integrity. This test verifies the suitability of using PTFE lining in SILVER II™ systems processing energetics and DMMP but not the reliability of the array of connections required when using this material in a full-scale plant.

Laboratory-Scale Testing

High-Shear Mixers

During Demo II operation of the 12-kW SILVER II™ plant, problems were experienced with the buildup and handling of solids in the anolyte circuit of the plant (AEA, 2000). The solids were both parent material and intermediate products from the destruction of energetics. Although all of the energetics tested (M28, TNT, and tetrytol) exhibited this problem, it was most pronounced during the processing of tetrytol, when a lowering of the feed rate became necessary to reduce the risk of plugging the process piping. The reduced feed rate and organic inventory led to reduced plant efficiency and destruction rate. As a result, PMACWA required EDS II tests to determine a suitable design for a high-shear mixer to size-reduce solid organic material, primarily from M28 propellant, in both the anolyte and catholyte circuits of the 12-kW SILVER II™ plant. The EDS II test objectives for the high-shear mixers were as follows (AEA, 2001g):

- Demonstrate that the mixers will size-reduce the crystalline solids that caused most of the operational difficulties in the plant during the Demo II tests.
- Demonstrate that the mixers will operate without operational difficulties with a slurry that contains M28 propellant.

The high-shear mixers must operate in the presence of energetics and energetic breakdown products and in the highly acidic conditions that are present in the anolyte and catholyte feed tanks of the energetics SILVER II™ plant. Most of the typical industrial solid-liquid mixers were found to be unsuitable for this application. However, tests were conducted using equipment supplied by a high-shear mixer manufacturer, Maelstrom, and included two generic types of high-shear mixers and a high-shear agitator blade. The two types of high-shear mixers were an integral pump mixer and a fluid distributive mixer, also called a high-shear vortex mixer. Both mixers subject the process fluid-solid mixture to high shear to produce a slurry, but the method by which they do so is different. Based on results of these tests, only the high-shear vortex mixer would meet operational goals.

High-shear vortex mixers were installed in the anolyte and catholyte feed tanks for the EDS II tests of the 12-kW SILVER II™ plant and performed well during the tests with Composition B and M28 propellant. AEA reported that the initial test showed that high-shear vortex mixers would effectively size-reduce the particles of Composition B and M28 propellant (and any other crystalline energetic particles that may have formed) and also provide bulk agitation (AEA, 2001a). AEA has also included conventional stirrers in all full-scale pilot plant feed tanks to further assure bulk agitation since the mixer head is small (18 inches in diameter) compared with the head in the full-scale vessel (10 ft in diameter) (AEA, 2001h). The full-scale mixing configuration has not been demonstrated. AEA also noted that the physical properties of energetics slurry could cause operational difficulties, with safety implications for equipment if unprotected, close-tolerance rotating parts were to come in contact with the slurry. Mixer design specifications address this requirement.

Hydrocyclone Trials for the 12-kW SILVER II™ Plant

As part of the EDS II laboratory-scale tests, trials were performed to determine the optimum hydrocyclone geometry for removal of solids from both the anolyte and catholyte circuits of the 12-kW SILVER II™ plant (AEA, 2001i). The trials established and demonstrated a hydrocyclone geometry capable of removing the simulated energetic material to a satisfactory level, less than 100 microns. In addition, the trials showed that the titanium hydrocyclones that had previously been installed on the 12-kW plant during Demo II for silver chloride removal could remove simulated energetic material after minor modification and reconfiguring. The simulant for the trials was chosen to closely mimic the behavior of the energetics to be processed.

Specific test conclusions by AEA are the following:

- The existing titanium hydrocyclones, modified to incorporate a 14-mm vortex finder[6] and a 6.4-mm underflow spigot, are suitable for the particle removal duty specified for a SILVER II™ facility at Blue Grass.
- The existing titanium hydrocyclones are close in design to the units to be provided by the Mozley Company for a full-scale facility in multiple parallel flow units (sometimes called "multiclones"). Therefore, the results for the titanium system are anticipated to be similar to those for the Mozley units.

The flow rate to the hydrocyclones for the full-scale SILVER II™ unit, based on a 14-mm vortex finder and a 6.4-mm underflow spigot, would need to be approximately 3.2 m^3/hr at a pressure of around 3.5 bar. This flow rate should be sufficient to achieve the overflow flow rate of 1.8 m^3/hr required to feed the SILVER II™ cells. As noted previously, the hydrocyclones used in the 12-kW energetics and agent simulant trials for EDS II handled solids at the planned design loading (AEA, 2001a).

The committee notes that the laboratory tests established operating parameters for hydrocyclone operation that involve careful control of pressures and flows to achieve desired separation efficiencies. No tests were performed to demonstrate the robustness of hydrocyclone operation during the pressure and flow swings that might be expected during normal operations of a full-scale facility.

Organic Transfer Across Cell Membrane

The objectives of this laboratory-scale test were to quantitatively assess the rate and determine the mechanism of transfer (diffusional versus electrochemical) of organics and their breakdown products across the membrane (AEA, 2001j). For the test, a small, laboratory-scale SILVER II™ plant was constructed at Aberdeen Test Center at Aberdeen Proving Ground, Maryland, to accomplish the following:

- Determine which species move across the membrane for a given substrate and their rate of transport.
- Optimize the chemistry for the destruction of specific organic feeds to achieve maximum current efficiency.

The equipment included an Imperial Chemical Industries FM01 test cell that had been modified to en-

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[6] A scoop that is positioned in the hydrocyclone vortex to obtain the desired particle size distribution in the overflow stream.

able it to treat mixtures containing energetic solids. The test cell contained platinized titanium electrodes and a Nafion N324 membrane and operated at a current density of 500 mA/cm^2. These parameters are used in the 12-kW pilot plant system as well as in the full-scale design.

The coulombic efficiencies for the destruction were calculated to be as follows (figures in parentheses include the total rate of destruction when direct chemical oxidation by HNO_3 is included):

- Composition B, 62 percent (63 percent)
- Composition B/M28, 78 percent (78 percent)
- DMMP, 26 percent (29 percent)

As can be seen, the Ag^{2+} is responsible for most of the destruction; direct reaction with the HNO_3 is a minor contributor for these organic materials.

The tests demonstrated that diffusion is the most likely mechanism for transfer of energetic organic species across the membrane from the anolyte to the catholyte chamber. Both the parent species in Composition B (TNT and RDX) as well as a TNT breakdown product, trinitrobenzene (TNB), move to the catholyte. Under operational conditions that provided a processing efficiency of approximately 80 percent for the energetics mixture, the rates of transfer were TNT = 2.6 g/hr/m^2, TNB = 0.6 g/hr/m^2, and RDX = 1.4 g/hr/m^2.

The tests also demonstrated that the movement of metals and other ionic species could be estimated from knowledge of the system chemistry and the appropriate ionic mobilities. Table 3-5 gives typical transfer rates for concentrations of 0.5 moles/L of feed.

TABLE 3-5 Typical Transfer Rates for Concentrations of 0.5 Moles/L of Feed

Parameter	Value
Current	5 kA
Area	1 m^2
Fe^{3+}	2.8 moles/hr
Al^{3+}	2.4 moles/hr
SO_4^{2-}	0.033 moles/hr
PO_4^{3-}	0.012 moles/hr

SOURCE: AEA (2001m).

Fluoride Transport Test

Agent GB contains fluoride. During SILVER II™ processing, the fluoride in GB is converted into HF. The ability of the HF to migrate through the plant is a factor in the selection of materials for different sections of the plant. A series of 8- and 72-hour tests were carried out in a laboratory SILVER II™ setup using the FM01 cell to confirm how fluorine moves around the plant, especially across the cell membrane and in the gas phase under different operating conditions. The tests, performed in a matrix of current "on," current "off", "silver present," and "silver absent," could be replicated. In each set of conditions, the change in concentration of fluoride with time in the anolyte and catholyte feed tanks and in the offgas was tracked after the introduction of sodium fluoride—equivalent to 0.1 M in the anolyte solution—at the beginning of each run (AEA, 2001k).

Test results showed that in both the 8-hour and 72-hour tests the concentration gradient caused a small diffusion of F (possibly as HF) through the membrane from anolyte to catholyte. Comparison of these results with runs using applied electric current showed the transfer flux is enhanced significantly by the electric field (possibly H_2F^+). However, even at the end of the 72-hour runs, the concentration of fluoride in the catholyte was no more than 0.01 M. In all of the runs, barely any fluoride was detected in any of the offgas scrubbers. This indicates that any HF in either the anolyte or catholyte offgas was returned in the condensate from the offgas condensers fitted to each of the anolyte and catholyte feed tanks.

The most significant test result was severe corrosion of the titanium anode. Based on inspection of the anodes after 8 hours of testing, it was concluded that attack in the presence of fluoride was occurring through imperfections in the platinum coating, undercutting it and causing it to spall off. As a consequence, the titanium was left exposed for corrosion during the 72-hour runs. The presence of silver is believed to enhance the titanium corrosion—possibly through local deposition and redissolution in nitric acid.

To address the imperfections of the anode coating, new Pt coating methods are being investigated by the electrode suppliers to improve deposition and eliminate pinholes. Also, no evidence of corrosion was observed during GB treatment in Demo II tests with the 2-kW SILVER II™ pilot plant, which is constructed of glass (AEA, 2000). AEA has indicated that the ability of F complexation with Si, A1, Ti, and Zr to reduce corrosion of the electrodes should also be investigated.

AEA also concluded that the agent offgas/scrubbing systems could be fabricated from stainless steel, while the agent anolyte and catholyte circuits should be lined with materials resistant to corrosion by free fluoride, e.g., PTFE or PFA.

Fluoride Removal System Test

In addition to the preceding fluoride transport tests, laboratory-scale tests were conducted to investigate the possibility of containing or removing fluoride from the system to allow more economical materials of construction to be used in the design of the full-scale plant (AEA, 2001l). A series of nine tests was to be conducted to obtain kinetic data on the use of calcium as an agent for fluoride removal from the GB simulant, fluorophosphoric acid. Data were to be obtained for the hydrolysis reaction under acidic, neutral, and alkaline conditions.

No test data were available at the time this report was prepared. However, the fluoride transport test results discussed previously confirm the need to consider some form of removal or complexation of fluoride.

Cell Membrane Life Tests

Tests on the lifetime of cell membranes were performed to provide design, testing, and operations data for a full-scale SILVER II™ plant (AEA, 2001m). The test objectives were these:

- Confirm the selection of membrane and gasket material for a full-scale facility.
- Estimate membrane and gasket life.

The test of membrane lifetime consisted of a single test of 95 days cumulative duration on a laboratory-scale SILVER II™ plant. Two grades of Nafion membrane and four grades of Viton gaskets were tested for the full test period. Worst-case conditions, defined as exposure to the highly oxidizing conditions of SILVER II™ operating in the absence of an organic feed, were used. These conditions correspond to operation in continuous rundown or polishing mode and expose the membrane to the highest concentration of Ag^{2+}. Analysis of the test data shows that the grades of membrane and gaskets currently used in the 12-kW pilot plant have a lifetime of at least 95 days under the conditions of this test.

The committee notes that the analysis of the cell membrane failure that occurred in the 500-hour DMMP (discussed in the next section) suggests that the cell membrane tests did not represent worst-case conditions for membrane life under actual operating conditions and that limited credence can be given to the conclusions from this test (AEA, 2001k). It also notes that in the absence of organics, parasitic oxidation of water may offset the Ag^{2+} attacks that were anticipated in this test.

Cell Membrane Failure Analysis

Repeated membrane failures during the early part of the 12-kW plant 500-hour DMMP run prevented effective control of water balance and levels of silver and organic material in the catholyte system. After laboratory-scale testing from October 11 to October 21, 2001, AEA concluded that the failures resulted from foaming or pockmarks in the lattice structure of the PTFE support in the Nafion membrane and that the pockmarks formed only if the membrane came into contact with the cathode.

The cell in the 12-kW plant was then modified to incorporate a system of spacers between the cathode and the membrane to ensure that the membrane did not contact the cathode. When the 12-kW plant was restarted, the catholyte level had risen more than would be expected during normal process operating conditions after 36 hours of operation. The cell was dismantled and the membrane was found to have small pinholes along the bottom edge. In addition, metallic silver deposits in the form of dendritic silver crystals were found in the cell.

DMMP testing was then stopped until PMACWA was convinced that the failure mechanisms were understood in sufficient detail to ensure that the plant could be operated without future membrane failures. A test program was conducted that looked at the following phenomena (AEA, 2001f):

- *Dendrite growth.* Solid silver crystal needles grow on the cathode that can lead to arcing, short circuits, and membrane breach.
- *Foaming of Nafion.* The interstices in the Teflon support lattice become discolored and develop pockmarks. This leads to zones of low mechanical stability from which pinholes may form, lead-

ing to localized heating, membrane failure, and arcing.

- *Membrane swelling.* Such swelling, which is caused by the chemicals present in the anolyte solution, specifically organophosphates such as DMMP, can lead to deformation and mechanical damage as well as contact with the cathode.

Laboratory-scale tests were performed using both an FM01 cell and an 'H' cell. The tests show that silver dendrite growth in the catholyte solution is prevalent when the concentration of HNO_3 is below 4 M. At concentrations between 4 M and 6 M, dendrite growth occurs at room temperature but is much reduced. It is not visible at elevated temperatures. With HNO_3 at concentrations above 6 M, there is no dendrite growth at room temperature. AEA concluded that the concentration of HNO_3 in the catholyte must be kept above 6 M to ensure that silver dendrite does not grow. Also, the formation of pockmarks appeared to be eliminated if the membrane was kept away from the cathode.

To validate the findings of the laboratory-scale testing, the 12-kW plant was reconfigured as a 4-kW system, with one operational anode and two operational cathodes. The cell was assembled with spacers between the cathode and the membrane to ensure that under operating conditions, the membrane could not come in contact with the cathode. The cell current was 2,000 A to provide the same current density across the membrane as that for the normally configured 12-kW plant. The catholyte offgas system was modified to provide a back pressure on the catholyte return system; this also had the effect of forcing the membrane toward the anode rather than the cathode. The plant was run in this configuration for 24 hours. Inspection of the membrane after this test showed that the membrane had been in contact with the anode and that there were no signs of silver deposits or damage to the membrane surfaces.

The plant was reassembled in its normal 12-kW configuration with cathode spacers fitted. To provide a worst-case operating condition, the back pressure on the catholyte system was removed so that the spacer was the only mechanism that kept the membrane from contacting the cathode. The plant was run for 24 hours under these conditions, and the cell was dismantled to inspect the membranes. There were no signs of damage to the membrane surfaces and no signs of silver deposits.

Electron microscope examination of a membrane has shown that the pockmarks have largely the same chemical composition as undamaged membrane components but contain a small amount of silicon. Burnt areas around pinholes have appreciable levels of platinum and titanium, which is probably a result of arc damage to the metal surface of the electrode coating. AEA noted that small amounts of this type of damage are of little consequence to the electrode since the electrochemical processes seal any damaged areas by passivation. AEA stated that it is not clear why pockmarks occur with DMMP (an organophosphate) and not with the energetics (AEA, 2001f). AEA has noted that in other electrochemical systems, pinholes can lead to local areas of high current density. This produces a significant amount of ohmic heating and melting, leading to membrane failure. It is unclear if this is a failure mechanism in this application.

The committee notes that the electrode damage may become more severe when feeds containing fluorine and chlorine are processed. In the fluoride transfer test discussed earlier, the titanium electrodes were severely corroded and the Pt coating was pitted or peeling off. This more severe damage would be unacceptable during full-scale plant operation.

AEA concluded from the EDS II tests that all of the mechanisms that can give rise to premature membrane failure had been identified (AEA, 2001f). Subsequent completion of the DMMP 500-hour run without membrane failure appears to validate this conclusion for DMMP operation. Although the DMMP run was successfully completed, it is not certain that other membrane failures will not occur when agent feeds containing fluorine or chlorine are used. Whereas organophosphates can lead to swelling of the membrane by the mechanism previously described, both concentrated acids and multivalent metal ions can cause shrinkage of the membrane. Further, it is not clear that additional membrane failure mechanisms will not be experienced as a consequence of flow and pressure variations in the six cell stacks of up to 48 cells each, recommended for the full-scale facility at Blue Grass.

Silver Recovery from Silver Chloride

These tests were conducted to provide sufficient data to select a silver recovery method (AEA, 2001n). They had three objectives:

- Demonstrate silver recovery from (contaminated) silver chloride.
- Determine the levels of impurities in the silver.

- Characterize the slag for purposes of reuse and disposal.

The tests involved mixing known amounts of silver chloride with sodium hydroxide. The silver chloride was precipitated from a simulated anolyte solution with composition similar to that expected after the anolyte solution has been passed through the polishing circuit of the IRS. Metals in the simulated anolyte solution included Fe, Al, and Cu. The silver chloride/sodium hydroxide mixture was placed in a nickel crucible and then into a furnace heated to 600°C. After termination of the reaction, the crucible contents were quenched in water, washed out with water, and filtered. A silver mass balance was then performed.

The results demonstrated that low-temperature caustic fusion at 600°C is effective in recovering more than 93 percent of the silver with a purity of greater than 99 percent. Furthermore, the silver is recovered in a form suitable for quick reaction with HNO_3 to form silver nitrate makeup without a significant amount of metal impurities. The committee notes that the 93 percent yield of silver suggests that 7 percent of the silver is not recovered, and the cost of this unrecovered silver and its disposal via a process waste stream must be considered when calculating the cost of operation. As reported in the EDP, AEA anticipates using off-site reclamation for silver recovery. An evaluation by Ames Goldsmith Corporation, a potential off-site reclaimer, states that 98 percent of the silver could be recovered at a purity level of 99 percent (AEA, 2001a). Thus, the EDP is based on off-site reclamation with a silver recovery of 98 percent.

Evaporator Trials for the Full-Scale SILVER II™ Plant

According to the EDP, a full-scale SILVER II™ plant at Blue Grass would include an evaporator circuit to reduce the volume of waste that arises from metals and mineral acids accumulated in the IRS. (See Figure 3-2 for the location of the evaporator in the IRS.) This test program investigated evaporator operation and selected suitable materials of construction (AEA, 2001o). Specific objectives were as follows:

- Conduct evaporator tests on a solution that simulates the solution to be evaporated in the full-scale plant for the IRS system.
- Demonstrate ease of evaporation and recovery of water and nitric acid from simulated feed solutions.
- Provide stream composition data for the overheads and bottoms streams during the evaporation process.
- Establish the maximum evaporation ratio (i.e., evaporated quantity/feed quantity) without compromising the pumpability of the bottoms discharge (or impurity) stream and without excessive decomposition of the bottoms stream while minimizing the volatility of any bottoms components (such as HF or HCl) or decomposition of the overheads components.
- Identify appropriate materials of construction for the evaporator.

The evaporator tests were conducted in a laboratory-scale test setup. The bottoms product after the worst-case volume reduction (70 percent) was found to be similar in density and viscosity to that of concentrated HNO_3. No solids and no decomposition were observed after cooling, and AEA expects that the resulting bottoms product after evaporation will not compromise pump operation.

Based on corrosion coupons in place during this test, Inconel 690 was found to be an unsuitable material of construction; however, PTFE-lined metal was suitably resistant to the most aggressive feed solution.

The committee notes that the test solutions fed to the evaporator contained nitric, sulfuric, and phosphoric acids and fluoride for establishing fluoride volatility. No silver or organic carbon was included, although design conditions assume up to 100 ppm total organic carbon and approximately 2 weight percent of the silver since complete silver separation is not expected. Also, some chlorides may be present after separation of the AgCl precipitate in the preceding process step. These low-level concentrations are problematic for good evaporator operation. In particular, foaming may result from the presence of organic materials, and fluorides and chlorides may migrate to the NO_x reformer system and pose added materials challenges.

Desktop Studies

AEA conducted a series of desktop studies (literature surveys) to establish certain design parameters for the full-scale plant. These studies were conducted in place of further tests. The conclusions of these studies are summarized below.

Water Jet Cutting for Conventional and Chemical Agent Munitions

This study, performed by El Dorado Engineering, Inc., comprised reviews of earlier studies on the use of water jets to cut munitions to access agent and energetics and the use of water jets to wash out energetics from the burster tubes of both rockets and projectiles (EDE, 2001b). After the study had been completed, water jet cutting was abandoned by AEA because of concerns that the garnet grit could cause problems downstream.

Earlier tests of the washout of energetics from bursters reported in the study by El Dorado Engineering used pressures up to 15,000 psig pressure with a water jet flow rate of 6.3 gal/min (EDE, 2001b). This study did not report whether the burster (or rocket body) was rotated during the washout process or whether any energetic material remained inside the burster tube afterwards. Also, the study noted that the earlier tests did not discuss how completion of washout was determined. From available test literature, the time to move and align the washout nozzle with the burster opening, start the water jet, rotate the rocket, advance the boring end effector down into the burster well, complete the washout, and return and shut off the nozzle and rocket rotator was estimated to be approximately 30 minutes using a 15,000-psig water jet and garnet grit. Based on other studies and the unacceptability of garnet grit in the SILVER II™ process, El Dorado Engineering, Inc., concluded that a 50,000-psig water jet could be safely used for washout of rocket and projectile burster energetics and that washout times of approximately 1 minute were achievable. The committee notes that neither test data nor literature were cited to support this projected washout cycle time.

Energetic Slurry Concentrations and Handling Study

Another study done by El Dorado Engineering reviewed the literature on handling energetic materials in water slurries. A primary objective was to establish safe concentration limits for energetics in slurry mixtures (EDE, 2001a). In the study, three papers were identified wherein energetic materials at various concentrations had been evaluated with standard testing methods for their shock initiation sensitivity. No data for the shock initiation sensitivity of M28 rocket propellant were available, but another propellant, M-9, with similar major ingredients was tested. The tests evaluated slurries that simulated homogeneous pumped (gelled) water slurries and settled water slurries.

The homogeneous pumped water slurries did not propagate detonation reactions at test concentrations of 20 to 40 percent of M-9 by weight in water. The settled slurries of M-9 did not propagate detonations at test concentrations of 35 percent to 40 percent M-9 by weight in water. At a test concentration of 45 percent, in both cases the reactions were in the nondetonation-to-detonation transition phase and exhibited some explosive (decaying detonation) reaction. At a test concentration of 50 percent by weight M-9 in water, the tests on both types of slurries propagated a high-order detonation.

El Dorado Engineering recommended that the M28 propellant (which is similar to M-9 propellant) not be pumped or handled or allowed to settle in concentrations above 40 percent by weight in water (EDE, 2001b). In this way, inadvertent initiation of detonations would be prevented, and no nondetonation-to-detonation, transition-phase-type reactions would occur in the process if the slurries were shocked.

The study noted that the three main reports reviewed studied only the shock initiation sensitivity of the aforementioned energetic materials (and the flame initiation sensitivity of RDX and TNT). M28 propellant might be subjected to other ignition sources in the slurry handling operations (e.g., metal sparking from antiresonance rods in the propellant grinding operation). The study recommends that these other initiation sources be studied in a comprehensive safety evaluation program that would assess the potential hazards that might accompany demilitarization of M55 rockets with M28 propellant.[7]

CST/CATOX Unit Testing

Although not part of AEA's SILVER II™ EDS II testing program, the CST/CATOX unit tests for EDS I conducted by Parsons/Honeywell are reviewed here since they serve as the basis for design decisions on the DTS and DTS offgas emissions control systems for the SILVER II™ technology package. Parsons/Honeywell performed a series of three tests of the CST in conjunction with its EDS I program (Parsons, 2001). No separate DTS tests were performed for the AEA EDP.

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[7] See discussion of the propellant grinding tests conducted by Eco Logic in Chapter 4.

Tests performed to determine the effectiveness of the CST system for processing shredded wood particles (simulated by sawdust) combined with a carbon-carrier medium encountered several problems, including the following:

- Quench tower foaming was caused by inappropriate recipes for antifoam and emulsifier additions.
- The vacuum in the CST main chamber was insufficient. This was partially alleviated by tightening down the gland seals on either end of the main chamber auger. However, insufficient vacuum levels in the main chamber were a chronic problem during all CST testing and would have to be addressed in any final design.
- Clogging by ash was the result of temporary shutdown of the auger without stopping the steam flow to the main chamber. The result was that a wet ash/charcoal mixture eventually caused the auger to jam and break the drive belt. The lesson learned from this problem was the importance of keeping the ash auger running whenever there is still some discharge from the system. Steam flow is required to sweep offgas from the CST.
- Feed materials in the feed hopper became segregated. This was a problem throughout the testing and caused a relatively large amount of wood to be fed into the system over short time periods. This, in turn, caused swings in the CATOX unit outlet temperature and poor treatment of gaseous emissions. The final design will have to include a means of maintaining feed uniformity to provide a basis for permitting.
- During restart, the feed auger variable-frequency drive failed as a result of overloads, necessitating replacement of the drive.

An evaluation of the CST (including its effluent gas treatment system) for treatment of wood dunnage and DPE suit material indicates successful treatment of both materials. Also, the test report noted that additional design and development for the solid materials handling and gaseous effluent systems were under way as this report was being prepared.

Solids materials handling issues include these:

- Preventing the segregation of the carbon-carrier medium and shredded DPE suit material and wood dunnage in the solids feed to the CST to minimize swings in performance and the challenges that these swings pose for the gaseous effluent treatment system.
- Selection of materials of construction with satisfactory corrosion resistance for the auger and air preheater—the test report indicates that the auger corrosion appears related to the chlorine contained in shredded DPE suit material (Parsons, 2001).
- Further design and development to prevent the accumulation of solids outside the auger trough at the feed end and in the bottom of the main chamber to prevent overheating of the chamber wall.
- Improved design for control of dust, especially carbon dust generated by the carbon carrier.

Gaseous effluent treatment issues still to be addressed include these:

- improving the temperature controls of the CATOX unit to prevent over-temperature damage to the catalyst, as occurred in earlier testing
- improved mitigation of solids separation upstream of the CATOX unit to prevent fouling/plugging of the catalyst by fine particulates
- changes in the design and operating conditions of the CST and CATOX units to reduce the formation of dioxins and furans when processing DPE material

In view of the Parsons experience with CST testing, it seems advisable for additional testing to be performed on the enclosed auger design for the SILVER II™ dunnage treater to ensure that auger plugging will not result in excessive downtime and maintenance. Testing is also recommended to verify the effectiveness of the sweep gas (steam) flow in removing volatile materials generated in an enclosed auger configuration.

The acceptability of the dunnage treater for permitting also remains an issue. For the CST, the committee notes that Parsons/Honeywell has stated as follows (Parsons, 2001):

> Several further modifications have been proposed to help lower/eliminate dioxin formation within the CST system. These modifications are major in scale and budget/schedule constraints preempted their implementation in the test system, however the risk associated with the problem of dioxin/furan formation may warrant their imple-

> mentation/testing before completing the detailed design of the full scale pilot facility (FSPF) which will require characterization of the treated gaseous effluent.

ASSESSMENT OF PROCESS COMPONENT DESIGN

Based on previously cited EDP documentation and EDS II testing results of the AEA SILVER II™ process, as well as the committee's consideration of test results obtained during Demo II, the following assessment of process component design and operation has been prepared (NRC, 2001b).

Disassembly of Munitions

Projectile Disassembly

AEA's technology package includes the use of the baseline system projectile mortar demilitarization (PMD) machine with modification for a water jet BWM, which uses a high-pressure water wash to remove the energetic material from the burster tube. The committee notes that a similar system has been proposed as part of the Parsons/Honeywell technology package put forth for disposal of the mustard agent munitions at Pueblo Chemical Depot (NRC, 2001a). The committee stated the following concerning the Parsons/Honeywell BWM:

> The BWM has not yet been built, much less tested, and the potential for separating larger energetics particles from the washout solution has not been evaluated. Shredding of the particles without sufficient washout solution could result in ignition. If larger energetic particles cannot be separated from the bulk washout solution, Parsons should consider design modifications to prevent or mitigate ignition.

In view of AEA's reliance on earlier burster washout tests, these observations also apply to the SILVER II™ burster washout design.

Rocket Disassembly

As described earlier, the RDM planned by AEA is derived from the baseline system design but has significant modifications. Tests performed to demonstrate the operability of these modifications would be done at the vendor's plant prior to shipment or during systemization of a facility at Blue Grass. No Demo II or EDS tests were performed. Committee concerns include the reliability of tube cutting technology for this application and underlying assumptions about the impact of this technology on downstream processes. The AEA proposal relies heavily on the success of an unusual application of tube cutting technology—that is, a configuration similar to a rocket in its shipping/firing tube, for which no demonstrable is available.

Further, the committee observes that the downstream energetics SILVER II™ process is being designed on the assumption that it will operate in a Category C area since agent-contaminated energetics are not normally expected to exist.

Preparing Energetics for Destruction

To process the M28 double-base rocket propellant in the SILVER II™ process, the propellant grains must be broken down into small particles. AEA uses commercially available shredding/grinding equipment to process the extracted propellant grain before introducing it to the SILVER II™ process. The propellant grinding tests conducted under EDS II testing for Eco Logic are discussed in Chapter 4 and are applicable to the AEA process. However, the committee notes that the AEA process requires a greater degree of size reduction than was demonstrated in EDS II (to $^1/_8$-inch versus $^1/_4$-inch). There also is some concern that the size of energetic particles from the burster washout process may be too large for processing by the high-shear mixers used in the energetics slurry feed tanks. Therefore, AEA has included an inline static macerator for burster energetics from both projectiles and rockets. The committee concludes that the proposed energetics preparation steps should be readily implementable in the full-scale plant.

Agent and Energetics Destruction

The technology provider has stated (AEA, 2001a) that information generated from the Demos I and II and EDS II tests have been used to fully define the features of the full-scale SILVER II™ process systems for a Blue Grass facility. The committee notes that many significant changes have been made in arriving at the design concept in the current EDP. The changes include these:

- Solids handling features have been enhanced by elimination of dead legs; use of hydrocyclones to manage solids in the feed preparation steps and in the streams passing to anolyte and catholyte sections of each cell for both agent and energetics processing; use of in-vessel high-shear vortex mixers as well as conventional stirrers to improve uniformity and provide final size reduction of particulates in the energetics and agent slurries; improved trace heating to prevent solids deposition on cooler surfaces in the electrolyte circuits; and improved materials of construction for process piping and components based on data gathered from the Demo II and EDS evaluations.

- A continuous recycle stream from the catholyte to the anolyte circuit in the main process system has been added. This recycle stream is needed to maintain concentrations of silver, organic material, dissolved metals, and other acids at acceptably low levels in the catholyte.
- A continuous purge stream on both agent and energetic anolyte circuits has been included to complete the destruction of organics, remove and recycle silver, and remove and concentrate impurities that include sulfuric acid, phosphoric acid, hydrofluoric acid, and dissolved metals. This purge stream is first treated in electrochemical cells requiring approximately 50 percent of the power of the main primary circuit cells to reduce organic concentrations to required levels. These cells have low electrochemical efficiency because the final organic concentration in the anolyte circuit decreases to less than 100 ppm. The committee notes that the polishing step had not been anticipated in the SILVER II™ design until the conclusion of Demo II (NRC, 2001b).
- Process steps have been added to remove and recover silver from the polishing system anolyte by use of 35 percent HCl following the completion of organics destruction. These steps include precipitation of Ag as AgCl, dewatering of the precipitate in a centrifuge, and evaporation of the liquid from the centrifuge in an evaporator to recover as much HNO_3 as possible for recycle to the reformer. The evaporator bottoms containing the S, P, F, and metal impurities in the plant feeds become a concentrated waste stream for further treatment and disposal.
- Caustic washing and thermal treatment of silver chloride precipitate are performed to achieve a 5X decontamination level for shipment to an off-site silver recovery operation.
- PTFE- or PFA-lined piping and vessels are used for the electrolyte circuits.
- The operation has been completely automated to eliminate hazards to personnel. The implementation of automatic operation is described in the final EDP (AEA, 2001a).

The SILVER II™ processes have become increasingly complex, apparently owing to the impact of phenomena not previously anticipated or considered important—for example, the need for extensive use of hydrocyclones to manage suspended solids throughout the processing system. The committee believes this increased complexity heightens uncertainty about the successful implementation of the technology package as a whole. Operation of a three-cell system is much simpler than that of full-scale 432-cell catholyte flow system that has all cells in a parallel flow path, with fluids containing significant levels of suspended solids.[8] In short, the current SILVER II™ EDP design is still too immature to consider full-scale plant implementation.

Other testing experience and process changes indicating the immaturity of the SILVER II™ process include these:

- Cell membrane failures not predicted by tests of cell membrane life and lack of long-term testing on feeds containing Cl and F.
- Planned removal of lead oxide using a formic acid wash without prior demonstration in a multicell configuration.
- Extrapolation of 3-ft-diameter vessel experience to 10-ft-diameter full-scale operation without having developed or demonstrated scaling parameters for high-shear vortex mixers and stirrers working together.
- Continuing discoveries of electrolyte chemistries that need to be carefully controlled—for example, the need to track HNO_3 as well as total acid molarity in the catholyte to avoid silver deposition.
- A full-scale design that has effectively increased cell power requirements by 50 percent to achieve the required destruction efficiencies in spite of efforts to demonstrate good electrochemical or coulombic efficiency; earlier studies of mediated electrochemical oxidation (MEO) processes anticipated this outcome, noting that low coulombic

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[8] Forty-eight cell stacks times nine (six main plus three polishing cells per stack), or 432.

efficiency occurs when high overall destruction efficiency is required (Chiba et al., 1995).

- Excessive corrosion of platinum-plated titanium electrodes in the presence of fluorine-containing anolyte feed streams; improvements in plating techniques are expected to solve this problem but have not yet been demonstrated.

Separation of Agent from Munition Bodies

The design basis for the MPT assumes 99 percent removal of agent from the munitions. This requires extra agent washout provisions for both projectiles and rockets, which can contain gelled agent heels. While the design seems reasonable, the ability of these provisions to achieve the required 99 percent removal of agent in the allotted time has not been demonstrated. It would be prudent to show that this cleaning performance can be achieved or that the MPT can handle higher agent loadings and still achieve required production rates. The committee notes that the proposed MPT would be demonstrated by the vendor prior to delivery for installation in the full-scale process, raising the potential need for design changes late in the construction process.

Agent Preparation for Destruction

The mixing of recovered agent with dilute HNO_3 before feeding it to the anolyte tank appears to be readily achievable using the proposed feed preparation system design.

Metal Parts Treatment

All contaminated metal parts will be treated to a 5X decontamination level in an MPT that consists of a four-zone metal enclosure containing a conveyor system on which bins filled with metal parts and fiberglass are placed. The full-scale design for Blue Grass calls for two MPTs, each capable of treating up to 8,000 pounds of metal (4,000 pounds of munitions-derived metal and 4,000 pounds of metal parts containers) in 4-hour cycles. The second and third zones of the MPT are heated by recirculating steam from electrically heated heat exchangers. The input and output zones are purged with nitrogen and then with steam. Vent gas from the MPT will be sent to a gaseous effluent treatment system. The MPT will operate in batch mode. All treated metal will be subject to a hold-test-release test to verify the absence of agent.

While the MPT design is considered generally robust, the committee has less confidence in the gaseous effluent system supporting the MPT operation. The design basis for the MPT assumes 99 percent removal of agent from the munitions. This removal efficiency is expected to be achieved by adding extra agent washout provisions for both the projectiles and rockets. However, these washout provisions have not been demonstrated, and lower agent removal efficiencies (higher MPT agent loading) may occur, requiring longer processing times.

The gaseous effluent treatment system for the MPT is similar to that used in the Parsons/Honeywell WHEAT technology. However, test results for the WHEAT system are not directly applicable to the gaseous effluent from the SILVER II™ MPT, because feed metal to the WHEAT MPT has been washed in caustic, which hydrolyzes the agent. In contrast, the SILVER II™ munitions washing process prior to MPT treatment may not significantly hydrolyze the agent. Thus, any agent on metal parts entering the MPT may be vaporized in a lower temperature zone of the treater and could exit in the separately exhausted gas streams of these zones before significant decomposition takes place. After filtration by the candle filters, this gas is subjected to 3 seconds residence time in the post-heater at 1,000°F to decompose residual agent expected to be present. Some data on the rate of thermal decomposition of agents suggest that even 5 minutes at 1,000°F is inadequate to fully destroy GB (NRC, 1993). However, the gas subsequently flows through a CATOX unit, a scrubber, HEPA and carbon filters, and then into the MDB HVAC system carbon filters before release to the atmosphere. The committee believes that this added filtering should ensure environmentally acceptable treatment of MPT gaseous effluent.

Dunnage Preparation and Treatment

The SILVER II™ DTS is similar to the Parsons CST (NRC, 2001a; Parsons, 2001). AEA has proposed improvements intended to eliminate the issue of shell burnout that occurred in the CST during EDS testing. This burnout resulted from ash overflowing the top of the auger trough and insulating the outer shell of the CST. This additional insulation caused the shell to be overheated and burnt by the external heaters. The improvements include an enclosed screw conveyor or auger rather than one running in a trough. Operation of an enclosed auger has not been tested under the conditions and feeds associated with dunnage treatment. The committee is concerned that the enclosed auger design may be more prone to blockage or plugging.

Catalytic Oxidation Units

Both the MPT and DTS use CATOX units to destroy VOCs in the gaseous effluent streams. The CATOX units use a Pt/Pd oxidation catalyst. AEA uses a scrubber and filter upstream of the CATOX units to remove phosphorus, fluorine, and chlorine compounds that could poison the catalyst.

With regard to dioxin/furan formation, AEA states that the use of carbon and HEPA filters downstream of the CATOX units should adequately control emissions of these materials. As noted earlier, testing of the CATOX units of the Parsons/Honeywell WHEAT process has revealed the need for changes in the design and operating conditions of both the CST and the CATOX units in order to reduce the formation of dioxins and furans when processing DPE suit material (Parsons, 2001). Thus, while the CATOX units appear to be capable of operating reliably, data from existing EDS testing of the Parsons CST and CATOX units indicate that there is still a significant risk of added delays and cost to achieve acceptable emissions performance (Parsons, 2001).

According to the AEA's EDP, carbon dioxide emissions from a full-scale facility at Blue Grass would be significant, 90 ton/yr, even with the anticipated 98-99 percent efficiency of the CATOX units (AEA, 2001a). The carbon dioxide arises primarily from the treatment of carbon and wood and DPE suits in the dunnage treatment CST and of fiberglass in the MPT.

ASSESSMENT OF INTEGRATION ISSUES

Component Integration

The committee notes that the SILVER II™ technology package is a combination of many continuous and batch-processing steps with buffering capabilities between most of the processing steps. The throughput and availability of each process step, in combination with equipment redundancy and sufficient buffer storage capacity between process steps, must result in the specified destruction rate. Integrating the individual processing steps will require effective process monitoring and control to ensure that appropriate materials are fed at appropriate rates at each step and that all material discharged from the plant meets safety and environmental specifications. In addition, attainment of the required process availability depends on the durability of all materials of construction and on the effectiveness of the plant operating and maintenance force.

Process Operability

Destruction of the Blue Grass stockpile within the time specified by the CWC treaty requires that the overall process achieve the required throughput levels and process availability (i.e., the fraction of time that the plant can operate). The SILVER II™ process has been designed to enable disposal of the entire stockpile at Blue Grass in 475 days. This includes 112 days of zero production for mandated periods (holidays, maintenance, and external causes like power outages). Shakedown/ramp-up and other activities that must be performed when switching between rockets and projectiles, and when switching between agent types, would require additional time.

AEA believes that the SILVER II™ technology package is a conservative design; that is, it has sufficient equipment redundancy and overcapacity to readily meet the required destruction rate at the assumed equipment reliabilities.

To the committee's knowledge, although large numbers of electrochemical cells are operated in parallel in the chemical industry, such operations have not faced the materials handling challenges that AEA's SILVER II™ agent and energetics destruction systems face. Hence, assumed equipment reliabilities may be optimistic.

Materials of Construction

Even in the relatively short-duration Demo II tests, it was apparent that corrosion from hydrogen fluoride (HF) in the anolyte and catholyte circuits would be a serious problem (NRC, 2001b). Results of Demo II coupon tests showed significant weight losses during exposure to simulated operating environments. Therefore, AEA conducted additional material coupon tests in the EDS II testing in addition to testing conducted with PTFE-lined piping segments. Specifically, these tests addressed the performance of two of the proposed lining materials, PTFE and PFA, and an alloy, Inconel 690. PTFE-lined piping segments were also tested during the 12-kW engineering tests. Piping with PTFE lining only $^{1}/_{16}$-inch thick performed acceptably in the severe environment of the evaporator tests. AEA also

TABLE 3-6 Materials Selection for Key Sections of a SILVER II™ Full-Scale Plant

Section or Unit Operation	Provisional Materials Selections
Anolyte circuits	Agent: generally all process-fluid-wetted parts to be lined with PFA, PTFE, or PVDF. Piping to be PFA-lined carbon steel throughout. Pumps, valves, etc. to be PFA-lined, ductile cast iron. Agitators etc. to be PTFE-coated carbon steel. Heat exchangers to use PFA for all wetted surfaces. Seals and gaskets to be Kalrez, PTFE, or PTFE-clad; hydrocyclones to be PFA-lined carbon steel. Energetics: generally all process-fluid-wetted parts to be PFA, PTFE, PVDF, or titanium; piping to be stainless steel with PFA lining; pumps to be titanium; valves to be PFA-lined ductile cast iron; agitators to be PTFE-coated or PVDF-clad carbon steel; heat exchangers to be titanium with PFA or PVDF-wetted surfaces; seals and gaskets to be Kalrez, PTFE, or PTFE-clad; hydrocyclones to be titanium.
Electrochemical cells	End plates and ports to be PVDF-lined ductile cast iron. Gaskets to be Viton for agent and Viton GF for energetics. Electrodes to be platinized titanium, pinhole-free, to a thickness of 5 micrometers for the anodes and 2.5 micrometers for the cathodes.
Catholyte circuits	Agent: same as anolyte circuit. Energetics: piping to be PFA-lined carbon steel; pumps to be titanium; valves to be PFA-lined ductile cast iron; agitators to be PTFE-coated stainless steel; seals and gaskets to be Kalrez, PTFE, or PTFE-clad. Heat exchangers to be 304L stainless steel. Catholyte evaporator to be Inconel 690/625; hydrocyclones to be titanium.
Polishing circuits	Agent: same as main anolyte and catholyte circuits. Energetics: same as main anolyte and catholyte circuits.
NO_x reformer	Agent: piping to be PFA-lined stainless steel; pumps and valves to be PFA-lined ductile cast iron; heat exchangers to be PFA or PVDF on wetted surfaces; seals and gaskets to be Kalrez, PTFE, or PTFE-clad; column sections to be PFA-lined carbon steel; packing to be PVDF. Energetics: piping to be PFA-lined stainless steel; pumps to be PFA-lined carbon steel; valves to be PFA-lined ductile cast iron; heat exchangers to be PFA or PVDF on wetted surfaces; seals and gaskets to be Kalrez, PTFE, or PTFE-clad; column sections including trays to be Inconel 690/625; packing to be Inconel 690/625.
Offgas scrubber	Agent and energetics: piping, column, and packing to be 304L stainless steel; pumps to be polypropylene-lined carbon steel.
Reagent feed systems	Agent and energetics: process water, silver nitrate, sodium hydroxide, and sodium hypochlorite feed system tanks, pumps, pipes, and valves all to be 304L stainless steel; gaskets to be EPDM or similar. Agent: nitric acid and hydrochloric acid feed system tanks, pumps, pipes, and valves all to be PFA-lined carbon steel; gaskets to be PTFE, Viton GF, or similar (e.g., Kalrez); hydrochloric acid pumps and valves to be PFA-lined ductile iron. Energetics: nitric acid and hydrochloric acid feed system tanks, pumps, pipes, and valves all to be 304L stainless steel; gaskets to be PTFE, Viton GF, or similar (e.g., Kalrez).
Fasteners, coatings, etc.	All fasteners (nuts, bolts, etc.) and other unpainted structural elements to be 316L or 304L stainless steel depending on the application. Protective coating for structural steelwork and carbon steel or ductile cast iron plant items—to be developed. All spill containment to be 304L stainless steel.
Wiring and instrumentation	Wetted instrument parts and pockets generally in accordance with wetted plant items in applicable area. Transmitter housings generally coated as structural steelwork above or constructed from 304L stainless steel according to availability and life-cycle cost. Cable insulation to be PVC, and carbon steel conduit (coated in accordance with guidance above) to be employed throughout.

SOURCE: Adapted from AEA (2001b).

reports that PTFE is the preferred material in the semiconductor industry, where aqueous HF is used extensively and leaching from HF containers would be unacceptable for maintaining product purity (AEA, 2001o).

Based on this information and additional research, AEA has made the provisional selection of the materials of construction for the SILVER II™ process systems shown in Table 3-6. This selection of materials should be adequate, assuming that AEA addresses the issue of assuring reliability in achieving leaktight joints after repeated disconnection and reconnection of piping and other system components. The committee also notes that AEA had not resolved corrosion of the platinized titanium electrodes observed during EDS II fluoride transport tests but expected to achieve a satisfactory solution.

Maintenance Issues

The full-scale plant provides for maintenance to be performed in DPE suits in areas where agent may be present. To minimize downtime, AEA has developed a detailed spare parts policy covering installed and stored spares. All pumps and hydrocyclones within the plant will have an installed spare (i.e., a standby) to allow remote changeover. The pumps have an automatic changeover system initiated by flow instruments and motor drives and a manual override. Hydrocyclones have manually initiated changeover only, since failure would be detected by a combination of installed instrumentation and operator judgment. An additional spare for each pump and hydrocyclone is held in storage. Isolating valves and flushing lines are provided to allow repair or replacement without requiring a plant shutdown.

One spare electrochemical cell stack is installed in the primary anolyte circuit. Manual intervention is required to connect the spare cell stack and disconnect a faulty cell stack. Five spare cell stacks are kept in storage, allowing replacement of all primary or secondary electrochemical cell stacks (but not both at once) in the case of common-mode failure, e.g., severe blockage. The inventory of spare cell stacks was not deemed necessary to cover common-mode failure of both primary and polishing (secondary) electrochemical cells, because their anolyte circuits are separate and the catholyte circuit is much less likely to be the source of failure (AEA, 2001a).

The use of installed spares and rotating equipment out of service for periodic maintenance should reduce the impacts on production rate from failures in equipment. This conclusion assumes that the resulting complexity of piping layout and increased manipulation of piping connections does not negate the goal of assuring process reliability.

Process Safety

Preliminary hazard analyses (PHAs) have been conducted for the SILVER II™ process at various stages of design and have served as building blocks for the EDP PHA effort (AEA, 2001a). These PHAs use the Failure Modes and Effects Analysis (FMEA) technique in accordance with the following regulations and standards:

- MIL-STD-882D, Department of Defense Standard Practice for System Safety (DoD, 2000)
- American Institute of Chemical Engineers (AIChE) Guidelines for Hazard Evaluation Procedures, Second Edition with Worked Examples (AIChE, 1992)
- Occupational Safety and Health Administration (OSHA) Regulation 29 CFR Part 1910.119, Process Safety Management of Highly Hazardous Chemicals
- Environmental Protection Agency Regulation 40 CFR Part 68, Risk Management Program

The FMEA is used to identify single equipment and system failure modes and each failure mode's potential effect(s) on the system or plant. The analysis generates recommendations for increasing process safety and equipment reliability. All equipment failure modes are assigned a risk assessment code (RAC) based on frequency and consequence severity categories assigned to the failure mode in accordance with MIL-STD-882D (DoD, 2000). Those failure modes with RACs of 1 or 2 require recommendations for design or procedural reductions in risk to RAC 3 or 4. After proposed fixes are identified, the failure mode is reranked as if the recommendation or fix had been implemented. This process is repeated until all items have an acceptable RAC (3 or 4).

The approach described is appropriate for assuring process safety for the preliminary design and is commonly used in industry where hazardous materials are handled. The committee further notes that a full-scale quantitative risk assessment (QRA) will be required in conjunction with the completion of the final design to assure that all process safety issues have been fully addressed.

An operational safety aspect of particular concern is the control of the oxidation reactions in the SILVER II™ agent and energetics systems. Since the process depends primarily on the generation of Ag^{2+} ions, the electrochemical oxidation reaction stops immediately when the power is switched off. Some chemical oxidation by nitric acid reaction also occurs, but AEA has reported that this reaction is a minor (less than 5 percent) contributor to the agent or energetic oxidation (AEA, 2001b). Since the technology provider has designed the process to operate on process streams with low concentrations of organics, the SILVER II™ process can be safely and quickly shut off at any time (e.g., from safety interlocks at other stages of the overall process) without any consequences associated with thermal or chemical inertia.

Precipitation of trinitrobenzene or other sensitive energetic materials in the energetics slurry handling and SILVER II™ system was identified as a concern during Demo II testing. Design changes identified in the final EDP and demonstrated in EDS II 12-kW tests appear to adequately prevent precipitation.

Worker Health and Safety

As in the baseline system facilities, much of the operation for a SILVER II™ plant is controlled from remote locations that protect workers from explosions and exposure to agent. ECVs and explosion containment rooms (ECRs) are used in the same manner as in the baseline technology. An area of possible concern is the technology provider's decision that all SILVER II™ cells processing energetics will be operated in Category C areas in the MDB layout. AEA indicated that the energetic slurry would normally be agent-free. The committee is concerned that cross-contamination of energetic materials with agent could occur during planned agent/energetics accessing and removal operations using modified disassembly machines. This cross-contamination may require reclassification of Category C areas to Category A areas for SILVER II™ energetics processing since normal maintenance might expose internal cell parts that have become agent-contaminated.

Since DPE suits were used in conjunction with the 12-kW tests, the committee concludes that they will be suitably safe for use in the possible presence of concentrated (60 percent) nitric acid. Other hazardous chemicals that would be found in an AEA SILVER II™ plant are typical of those in any large chemical or electrochemical plant and appropriate worker safety equipment and procedures would be used.

The SILVER II™ NO_x reformers require oxygen (introduced through the catholyte vessels). Preliminary material balances indicate the quantity of oxygen consumed in a disposal campaign for GB rockets at Blue Grass would be approximately 1.6 million pounds. The oxygen would be supplied as liquefied oxygen and vaporized to a gas on-site. Although liquefied oxygen is a hazardous material, it is routinely used in chemical plants and in the NASA space shuttle program without significant safety problems.

Public Safety

Accidental releases of agent or other hazardous materials are expected to be no more likely for a SILVER II™ plant than for plants using other technologies for destruction of agent. The most significant source of accidental exposure to the public remains the rockets in storage and transport to the chemical demilitarization facility.

Human Health and the Environment

Characterization of Effluents

In its Demo II report, the committee noted that the following gaseous effluents from the SILVER II™ process were analyzed (NRC, 2000a):

- Anolyte gas was measured for CO, SO_2, VOCs, semivolatile organic compounds (SVOCs), agent, and Schedule 2 decomposition compounds.
- Prereformer and postreformer gas was measured for O_2 and NO_x.
- Discharged offgas was measured for CO_2, O_2, CO, N_2, N_2O, H_2, SO_x, NO_x, VOCs, SVOCs, agent, and Schedule 2 decomposition compounds.

The Demo II tests revealed the presence of VOCs in the offgas stream and, as described earlier, the technology provider has included thermal treatment and a CATOX unit in the final design for the combined offgas streams. Full characterization of the gases from the combined SILVER II™ agent process and MPT process streams has not been possible since such a stream was not produced in testing. The same is true for the combined SILVER II™ energetics process and DTS process streams. However, testing of the Parsons/Honeywell WHEAT CST technology has allowed characterizing the results for treatment of dunnage and DPE suit material. This testing, while not directly representative of SILVER II™ DTS operation, indicates the need for changes in design and operating conditions of the DTS and CATOX units to reduce the formation of dioxins and furans when processing DPE suit material.

As noted earlier, CO emissions at a SILVER II™ facility at Blue Grass would be significant, 90 ton/yr, even with the anticipated 98-99 percent efficiency of the CATOX units.

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 this report was prepared, not all of the data were available to the committee. As noted in the Demo II test report, the Army has concluded that the characterization of the products from agent and propellant destruction showed that most hazardous intermediates were encountered at relatively low levels that could be effectively destroyed with additional treatment steps (NRC, 2001b).

Completeness of Effluent Characterization

The effluents from the integrated SILVER II™ processes had not been completely characterized when this report was prepared. Prior committee assessments of effluent characterization (NRC, 1999, 2001b) are still considered valid.

Effluent Management Strategy

The AEA effluent management system proposal is to send dilute nitric acid that is not used as process water makeup as a waste stream to a publicly owned treatment works (POTW) under a pretreatment exemption. An analysis of the characteristics of effluent from the Demo II tests shows that it could be disposed of in a POTW; however, it is not known whether such a facility exists.

At the time this report was prepared, the solid products from the MPT, DTS, and the evaporator had not been characterized. Tests of silver recovery from silver chloride demonstrated that economic silver recovery was possible (AEA, 2001m). Although the silver chloride used in the tests was not generated from purged anolyte, impurities in the silver chloride effluent produced from purged anolyte are not expected to alter this conclusion, which is based on EDS II tests using a simulated anolyte purge solution (AEA, 2001n).

Off-site Disposal Operations

The anolyte in agent and energetics SILVER II™ systems will be constantly purged so that metals and mineral acids that accumulate in the catholyte and anolyte circuits can be removed. These contaminants will be collected in the evaporator bottoms and disposed of using standard industrial waste treatment techniques. The contaminants will include iron, lead, and aluminum from the munitions as well as sulfur, phosphorus, and fluorine from the agent treatment; small amounts of silver not recovered during the AgCl precipitation; and trace amounts of organic carbon remaining after the polishing step. The occurrences of all species but silver and organic carbon are generic to all processes that destroy agents and/or energetics.

Some concentrated nitric acid may be produced as a by-product in the SILVER II™ process when processing energetics compounds, which contain nitrogen. The material will be collected and sold to off-site users. During Demo II testing, analysis of nitric acid samples by the Ensign-Bickford Company determined that the acid could be used in the manufacture of energetics at its Kentucky facility (NRC, 2001b).

While it is currently planned to process uncontaminated dunnage in the DTS, the committee notes that this material could also be disposed of off-site.

Environmental Compliance and Permitting

The permitting process for the SILVER II™ technology package is expected to be substantially more complex than for current baseline facilities. The permitting protocol is well established for the baseline facilities. SILVER II™ will require a Subpart X permit, for which there is no precedent. The committee also notes that the SILVER II™ total solution technology package for Blue Grass entails a very large scale-up from 3 cells during Demo II and EDS II testing to 432 cells for agent destruction and 378 cells for energetics destruction, which will be a concern for permit writers.[9] In seeking an RD&D permit,[10] for example, permit writers would need to be assured that the applicant knows where the materials, particularly the metals, are going, which in turn demands that permitters have confidence in the mass balances for the technology. Mass balances currently in the EDP do not fully characterize the process streams, particularly the effluent streams. Although the committee sees no reason to conclude that permitting cannot be accomplished, notwithstanding the current absence of a full characterization of all process effluents, it believes the permitting will be very complex. There is also a need to develop a very comprehensive set of performance data to allow operating conditions to be established even after a facility has been designed and built.

Further insight into the permitting challenge is given by considering the following list of candidate permitting requirements and issues being developed by the Department of Energy for the SILVER II™ process that it is considering for destruction of spent organic solvent from its plutonium-uranium solvent extraction processes (DOE, 2001). These requirements and issues include:

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[9] The 12-kW plant used for Demo II and EDS tests uses three cells operating at 2,000 amps maximum per cell at 2 V.

[10] RD&D stands for research, development, and demonstration. This is a special type of RCRA permit that allows testing of treatment units for development and demonstration purposes. These types of operations require a permit even though it is much less stringent and demanding than a full RCRA operating permit.

1. Establishing regulatory-based performance standards for the technology covering regulatory parameters of interest such as these:
   - A DRE for organics in offgas of 99.99 percent and a risk-based, site-specific analysis.
   - Site-specific risk analysis of VOC emissions in vent gases.
   - Levels of NO_x emissions in relation to Clean Air Act ambient standards and the risk-based alternative acute emissions guidance levels from Volume I of the *Hazardous Waste Combustion Risk Analysis Guidance Document* (EPA, 1998). This will address NO_x as potentially convertible to nitric acid.
   - Characteristics of solid and liquid residuals in relation to land disposal restrictions requirements using the toxic constituent leaching procedure and analysis of the underlying hazardous constituents such that implementation of proper disposal options can be ensured.
   - Site-specific risk analysis of oxidation by-product emissions, including dioxins and nitrated polyaromatic hydrocarbons.
   - Metals emissions, e.g., mercury emissions, in relation to EPA's hazardous waste combustion maximum achievable control technology (MACT) standards and a site-specific, risk-based analysis that is particularly focused on silver and other metals impacted by the formation of chlorinated and nitrated volatile metals.
   - Particulate matter emissions in relation to hazardous waste combustion standards.
2. Establishing operating parameters of interest to permit writers (compliance assurance):
   - SILVER II™ oxidation conditions
     —Current flow to cell
     —Silver content of anolyte circuit ($AgNO_3$) makeup rate
     —Waste feed cutoff tied to current flow
     —Temperature of anolyte feed tank (main concern is precipitation of by-products)
     —Anolyte mixer operation
     —Anolyte circulating pump flow rate
     —Total organic level in anolyte circuit
   - Waste feed characteristics
     —Waste feed rate to anolyte cell
     —Maximum particle size
     —Maximum chlorine feed rate (AgCl precipitation)
     —Tramp metal and other inorganic impurity concentration
   - Hydrocyclone
     —Minimum and maximum pressure drop
   - NO_x reformer operating conditions
     —Condenser maximum temperature
     —Oxygen feed rate
     —Distillation operating temperature
   - Offgas condenser
     —Maximum operating temperature
     —Cooling liquid flow
   - Scrubber operating conditions
     —Caustic flow rate
     —Scrubber pH
   - Catalytic oxidizer operating conditions
     —Minimum and maximum operating temperature
     —CO destruction efficiency continuously or at set intervals
     —Confirmatory dioxin tests
   - HEPA filter operating conditions (separate analysis under way)
     —Minimum and maximum pressure drop
     —Installation testing
     —Maximum shelf life
     —Maximum temperature
     —Relative humidity limits
   - Carbon filter operating conditions
     —Maximum operating temperature
     —Carbon replacement rate
     —Offgas flow rate
   - Emissions monitoring
     —Continuous emissions measurements of $CO/CO_2/O_2$ in vent gases
     —Continuous measurement of NO_x emissions in vent gases
     —Continuous emissions measurements of total hydrocarbons
     —Chlorine emissions monitoring
     —Hold-test-release protocols

It is clear from the preceding list that permitting will be challenging for the AEA SILVER II™ technology package for Blue Grass.

ASSESSMENT OF OVERARCHING TECHNICAL ISSUES

Overall Engineering Design Package

The overall EDP offered for AEA's SILVER II™ technology has grown in complexity from what was presented during the Demo I and Demo II phases of the ACWA program. Some of this growth in complexity was to be expected as the level of design developed. The EDP design does appear to reflect most of the configuration and equipment necessary to implement a full-scale pilot plant final design for the current state of understanding of the SILVER II™ technology.

The complexity of steps identified for this technology has continued to increase as greater understanding of the detailed requirements of the process is developed. These new requirements arise not just from more detail in design, but also from the identification of additional unit operations that must be included for satisfactory operation, particularly in the agent and energetics destruction systems. Some of these requirements were identified in Demo II testing, others have continued to be identified as EDS II testing proceeds.

These changes appear to arise from the fact that the basic process, while understood in principle, is not well understood in detail. This reflects a significant immaturity in the SILVER II™ technology for this very demanding application and raises concerns that more complexity would arise as the design is further developed. For example, the current assumption that all energetics destruction by SILVER II™ will take place in a Category C area may not be valid if the current methods of agent and energetics separation cannot provide satisfactory assurance that the energetics going to SILVER II™ processing are agent-free. Should the current assumption prove invalid, major changes in MDB design and costs might be required. Likewise, the MPT design is based on removal of 99 percent of agent from metal parts before MPT processing. If this efficiency is not achieved, further changes in the metals washing and/or MPT processes may be required.

The committee believes that further testing during systemization of a full-scale facility would reveal a need for more than the usual number of design changes. Some aspects of the current design that point to the likelihood of further change are the following:

- The RDM agent cavity washout scheme and the use of a tube cutter to cut shipping tube and rocket is a significant departure from the baseline process and AEA's previously specified process for removing firing tubes and cutting the rockets with a high-pressure water jet using garnet grit (AEA, 2001b). The committee notes that the method of cutting is unproven for this application, and there are significant uncertainties associated with differential movement between the tube and the rocket body as both are rotated and cut. In addition, the proposed method of washing or steaming agent out of the rocket agent cavity has not been demonstrated and, if this method is not as effective in removing agent as planned, it is unclear that the MPT and its associated gaseous effluent system would be capable of handling the additional agent loading.
- The destruction of fuzes and supplemental charges has changed. Instead of using a standard detonation chamber, they are simply placed in the MPT and allowed to decompose or detonate. At the same time, the MPT design has changed—it now uses a steam-heated industrial oven with internal steam recirculation, which has not been demonstrated for destruction of fuzes and supplemental charges.
- The technology provider now plans to use electrical steam heaters and internal steam recirculation for the MPT. Although this configuration seems feasible and is a variant of the MPT technology tested for the Parsons/Honeywell WHEAT technology package, the committee notes that it has not been demonstrated in its proposed configuration and that the steam-reactant environment may pose significant materials technology challenges and result in different chemical loadings in the gaseous effluent treatment system. Also, the operating duty of the recirculation blowers, which are required to cycle between near ambient and over 1,000°F every 4 to 6 hours, will challenge their reliability. (When the MPT is recharged every 4 to 6 hours, the high-temperature zones are exposed to cooler temperatures as a result of charging and discharging.)
- The DTS is based on the CST of the Parsons/Honeywell WHEAT technology package (Parsons, 2001). However, the DTS design uses an enclosed auger to move the dunnage as it is being decontaminated to a 5X condition. This enclosed auger was selected to prevent dust and debris from accumulating in the bottom of the dunnage treater. The ability of the enclosed screw to operate without significant plugging and with adequate flow of the steam sweep gas has not been demonstrated.
- Many major design changes have taken place in the agent and energetics SILVER II™ process systems. For example,

  1. High-shear vortex mixers are included in all feed tanks to provide final size reduction and mixing of particulates/precipitates that may exist in electrolyte slurries. These mixers replace inline mixers that had been proposed at the end of Demo II testing. Also, conventional stirrers will be installed in large-diameter tanks to ensure that the high-shear vortex mixers contact the entire contents of the tank (AEA, 2001a). The basis for this combination of mixing devices is experience in a 3-foot-diameter vessel and will require revalidation in the geometry of the 10-foot-diameter full-scale vessel.
  2. A catholyte recycle circuit was added following Demo II testing because of greater than anticipated flow of metal ions, mineral acids, and organic material from the anolyte to the catholyte in the electrochemical cells.
  3. EDS II testing revealed the need to monitor nitric acid concentration as well as total acid concentration in the catholyte circuit to prevent formation of silver deposits that led to arcing to the cell membrane and the development of holes in the membrane. More changes may be necessary to maintain the higher nitric acid concentrations in the catholyte.
  4. Demo II testing identified the necessity of an

anolyte polishing step in the IRS. The design used is based on limited rundown tests at the end of the EDS II 12-kW testing.

5. The IRS has been modified to incorporate a batch step for removal and recovery of silver ions accumulating in the IRS circuit. This removal operation involves the addition of 35 percent HCl and subsequent collection and treatment of the AgCl precipitate for off-site removal. The process has not been demonstrated on purged anolyte solution with metal, mineral acid, and trace organic impurities.
6. The purge of anolyte to the IRS also requires use of an evaporator to recover nitric acid and produce an anolyte-derived brine waste containing mineral acids and metals from anolyte (i.e., the impurities removal operation).
7. Concern for cavity plugging of the cell electrodes from slurry particulates has resulted in the use of hydrocyclones on all cell feed streams in both the main circuit and the polishing circuit. AEA states (AEA, 2001o) that

> the anolyte and catholyte circuits use a 'multi-clone' arrangement of hydrocyclones with their overflows combining to a single process line, which is then fed to the electrochemical cells. Their underflows are combined to a common discharge line and returned to the respective anolyte or catholyte tanks. . . . This arrangement is common in industry where it is necessary to achieve a high flow capacity and a particular particle cut size.
>
> The proposed multi-clone arrangement has been designed with slurry handling and trace heating considerations to prevent any premature settling and blockage formation. In addition, the full-scale plant employs an additional multi-clone as a stand-by spare so that any changes in performance due to blocking or plugging (observed by pressure or flow changes) can be rectified by switching to the installed spare.

Integrated operation of these hydrocyclones has been demonstrated in a three-cell configuration. However, scale-up to 432 cells may present new challenges in flow and pressure management to sustain satisfactory hydrocyclone operation.

Reevaluation of Steps Required for Implementation

In 1999 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 were first reevaluated by the ACW II Committee following the Demo II tests (NRC, 2001b). These steps are again reevaluated here on the basis of the results of the EDS II testing and EDP.

> 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 1/4 inch in diameter.

This still needs to be evaluated. The technology provider plans to test the new RDM at the vendor's facility prior to shipment. No proof of principle prior to this time is anticipated, especially demonstration of the efficacy of the tube-cutting concept for concurrent cutting of firing tubes and contained rockets. Also, the capability of the punch-and-drain and rocket-cutting operations to prevent agent contamination of energetics has not been demonstrated.

Reduction of propellant size by grinding under water appears to be feasible with the current concept. However, it is noted that this feasibility depends very much on successful propellant grain extraction using the untested RDM concept.

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

Since no mines will be processed at Blue Grass, there is no need to test mine shearing.

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

This is no longer relevant, because fuzes and any supplemental charges will be fed to the MPT for deactivation.

> 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 byproducts. At the time the committee was preparing its Demo II report, not all of the data were available and the impact of trace species, particularly in brines and atmospheric releases, on facility permitting remains to be determined. This information was still unavailable to the committee as of the time the present report was being prepared.

> 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 committee again stresses the importance of this step in light of the increased complexity of the process after the changes proposed to address problems revealed during the Demo II tests. As noted in the earlier discussion on design changes, the technology provider has included hydrocyclones on all cell feed streams. While the hydrocyclones will surely improve particulate management, the committee remains concerned about the robustness of cell flow control in the presence of particulates that pass the hydrocyclones, especially considering the large array of parallel flow paths (up to 432 on the catholyte circuit that is common to both the main anolyte and polishing circuit for agent destruction).

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

This recommendation was not addressed in the Demo II tests or EDS II tests. It remains valid even with the proposed RDM design that uses tube cutting and a hollow upper punch with steam/hot water washout. The technology provider plans to conduct testing at the vendor's facility, but this testing can only be conducted under simulated conditions, and it may be insufficient to reveal design deficiencies with respect to removal of agent heels to the levels required by the MPT design.

> 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 Demo II successfully 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, which AEA was still evaluating at the time the Demo II report was prepared (NRC, 2001b).

Subsequent EDS II testing in the 12-kW system established that a better design that eliminates dead legs, along with trace heating that removes cold spots in the piping and vessels, can eliminate the precipitation of recalcitrant intermediate products. Examination of the 12-kW system following test runs with energetics mixtures containing M28 propellant (containing ~0.5 weight percent lead as lead stearate) also showed that the lead in the lead stearate oxidizes to lead oxide and that much of this material is deposited in the electrode cavity (AEA, 2001d). The committee notes that a material balance was not performed on lead, so it is unclear whether lead oxide will also be carried to the IRS with the anolyte purge stream. If so, it would be expected to be removed in the evaporator brine as a salt.

AEA says that it has demonstrated in laboratory-scale tests that lead oxide can be removed from the cells as lead formate using off-line flushing with formic acid solution (AEA, 2001a). The EDP includes provision for removing one cell stack at a time and performing a formic acid wash or flush to remove the accumulated lead oxide. An installed spare cell stack would be put online during the formic acid flush, and the flushed cell would become the installed spare. While this operation was not demonstrated during EDS II testing, it appears to be implementable. Therefore, the committee concludes that energetics destruction by the SILVER II™ process has been satisfactorily demonstrated under planned design and operating conditions.

> 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 now uses an MPT to decontaminate metal parts to a 5X level using high-temperature (>1,000°F) steam at near atmospheric pressure.

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

This is no longer relevant because the shredded wood and DPE suit material will be treated in a dunnage treater similar in design to the CST of the Parsons/Honeywell technology package demonstrated in EDS I testing, and which achieved 5X decontamination. The enclosed auger design of the dunnage treater used in AEA's DTS remains to be demonstrated.

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

The ACW I Committee's concern at the time this step was listed was plugging in the cell channels from the formation of AgCl precipitate. AEA has since provided hydrocyclones to remove this precipitate. These hydrocyclones were used in EDS II 12-kW test operations and were found to perform well. However, the committee still remains uncertain about successful operation in a full-scale plant (where there are up to 432 parallel paths vs. the 3 parallel paths that have been tested), since particulates, albeit less than 100 microns, still remain in the feed to the cells and can cause plugging if not managed properly.

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

While the committee recognizes the viability of the liner materials chosen following Demo II and EDS II corrosion tests to deal with the severe corrosion conditions, it remains concerned about the ability to readily achieve leaktight connections routinely after both scheduled and unplanned maintenance activities. It is also concerned about AEA's conclusion that stainless steel can be used in the agent NO_x reformers based on the EDS II fluoride transport tests. From these tests, it did not appear that fluorides existed in the gas stream to the reformer; however, the volatility of HF suggests that such an outcome would not be unexpected. More long-term testing of fluorine-containing electrolyte derived from organic materials containing fluorine would be prudent.

> 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.

During EDS II testing, it was demonstrated that AgCl decontaminated to a 5X level could be sent to an off-site silver recycler and that the silver could be economically recovered and returned to the plant for silver nitrate makeup. The committee notes that this testing used a simulated anolyte purge that was doped with metals and minerals acids but did not include lead or the organic carbon that remains after polishing. Thus, while recycling of silver seems achievable, there may be further challenges to process efficacy when material from actual anolyte purge streams is used.

FINDINGS AND RECOMMENDATIONS

Findings

Finding (Blue Grass) AEA-1. The SILVER II™ systems for processing agent and energetics have become increasingly complex since Demo II. The increasing complexity appears to be driven primarily by the impacts of phenomena not previously considered important. This indicates that AEA's EDP design is still too immature for implementation.

Test experience pertinent to the stage of maturity of the process includes the following:

- During Demo II and EDS II testing, only a 3-cell system was operated, which is much simpler than a full-scale 432-cell flow system with all cells in a parallel flow path and fluids containing significant levels of suspended solids.
- Cell membrane failures are not predicted by cell membrane life tests, and there has been no long-term testing on feeds containing Cl and F.
- There are plans for the removal of lead oxide using formic acid wash without prior demonstration in a multicell configuration.
- Tests with a 3-ft-diameter vessel will be assumed to apply to a 10-ft-diameter, full-scale vessel without development of demonstrated scaling parameters for high-shear vortex mixers and stirrers working together.
- Electrolyte chemistries continue to be discovered and need to be carefully controlled, e.g., there is a need to track nitric acid molarity as well as total acid molarity in the catholyte to avoid silver deposition, and the deposition of lead dioxide on cell electrodes and in electrode cavities, which has required the development of a formic acid wash.
- The full-scale design has effectively increased cell power requirements by 50 percent to achieve the required destruction efficiencies. These increases were not identified until EDS II testing was performed.
- There was excessive corrosion of platinum-plated titanium electrodes in the presence of fluorine-containing anolyte feed streams. Improvements in plating techniques are expected to solve this problem, but they have not been demonstrated.

Finding (Blue Grass) AEA-2. Although the RDM proposed by AEA follows the basic steps used in the baseline system, it implements the steps with a suite of new equipment that has not been built or demonstrated.

No evidence has been provided of such equipment (e.g., tube cutting, burster washout, and grain extraction) being used in the manner required for an integrated RDM for the SILVER II™ technology package. Past experience suggests the design will have to be modified as testing occurs, and it is not clear that modifications will ensure that energetics feeds are agent-free, thereby allowing operation and maintenance in a Category C area.

Finding (Blue Grass) AEA-3. Long-term operation of the SILVER II™ cell membrane on all the required feed streams has not been demonstrated. Because the SILVER II™ process is the primary treatment process for agent and energetics, the committee is particularly concerned that the life of the cell membranes, on which the efficacy of the SILVER II™ process depends, has not been demonstrated.

Reasons for this finding include the following:

- Long-term operation on H and GB feeds containing chlorine and fluorine has not been demonstrated. The agent simulant, DMMP, does not contain these elements.
- The laboratory-scale fluoride transport tests revealed severe attack on the Pt plating of the electrodes. AEA is now seeking resolution of this problem with electrode manufacturers. Pt and Ti released in these attacks may plug electrode cavities or impact membrane performance.
- The slurry flow management scheme to the cells has large numbers of parallel flow paths through the hydrocyclones and through individual electrode cavities. Upsets in these paths can lead to upsets in the quality and quantity of slurry flowing to the electrode cavities, with possible impact on membrane operation.

Finding (Blue Grass) AEA-4. The efficacy of using candle filters such as those that AEA has proposed has not been demonstrated.

Experience with fouling of the CATOX unit during the Parsons/Honeywell CST tests suggests that the particulates are sticky and may not be readily removed from the ceramic candle filters by back flushes with nitrogen gas. The committee notes that the tests conducted on the CST system (which used cyclones rather than candle filters) are the only tests pertinent to the design proposed for AEA's SILVER II™ DTS, and there are differences in the latter design that present untested/undemonstrated challenges to successful operation.

Finding (Blue Grass) AEA-5. Existing CATOX unit EDS I tests to date indicate that there is still a significant risk of added delays and cost to achieve reliable operation, particularly with respect to meeting acceptable dioxin and furan levels (Parsons, 2001).

This finding is based on EDS I CATOX tests by Parsons/Honeywell and also on the differences between the design tested and the offgas effluent treatment system proposed for the SILVER II™ process. Since the AEA CATOX unit is modeled after the Parsons/Honeywell design, it can be expected to produce similar amounts of dioxin and furan. In addition, the gas treated by the AEA CATOX unit has a different source and therefore a different composition. No CATOX units have been tested on gases from this source.

Finding (Blue Grass) AEA-6. The committee believes that a very complex permitting process will be required for the SILVER II™ technology package for Blue Grass, and that there is a need to develop a very comprehensive set of performance data to allow the operating conditions to be established even after a facility has been designed and built.

Recommendation

Recommendation (Blue Grass) AEA-1. Based on the above findings, the committee recommends that the AEA SILVER II™ process not be implemented at Blue Grass.

4

Eco Logic Technology Package

INTRODUCTION AND OVERVIEW

The Eco Logic technology package is being prepared by a team composed of ELI Eco Logic International, Inc. (Eco Logic), Foster Wheeler, El Dorado Engineering, and Kvaerner Process Systems, Inc. Eco Logic is the prime contractor and the technology provider for the gas-phase chemical reduction (GPCR™) system. Foster Wheeler is the technology provider for the transpiring-wall supercritical water oxidation (SCWO) system. El Dorado is the technology provider for the reverse assembly equipment design and the materials handling equipment design. Kvaerner is the provider of the hydrolysis design and performs plant-wide process integration services. The Eco Logic technology package is shown schematically in Figure 4-1 and described in Table 4-1. It includes the following steps:

- Chemical agent, energetic materials, and metal parts are separated using a modified version of the Army's baseline system disassembly process.
- Chemical agent and energetic materials are decomposed in separate hydrolysis systems.
- The hydrolysates are treated in a transpiring-wall SCWO system in which organic compounds are destroyed.
- Metal parts and dunnage are decontaminated to a 5X level, and gaseous effluents from the hydrolysis processes are treated by reaction with high-temperature hydrogen in the GPCR™ system.

DESCRIPTION OF THE PROCESS

The following process description is based on both the final and draft engineering design package reports submitted by Eco Logic and on two design review briefings presented to the committee by Eco Logic representatives (Eco Logic, 2001a, 2001b, 2001c, 2001d).

Area 100—Reverse Assembly/Munitions Access

Area 100 is the designation given to the set of operations performed during reverse assembly (disassembly) and munitions accessing for agent and energetics removal. This area establishes the throughput configuration for the entire facility design. In this area, munitions are received, agent drained, munitions bodies opened, energetics removed, and the separate agent and energetics streams sent to Area 200 for hydrolysis or deactivation. Area 100 is divided into three major areas: the rocket line, the projectile line, and the continuous indexing neutralization system (COINS) area. The major components of Area 100 are shown in Figure 4-1. It consists of multiple parallel processing trains within compartmented, closed areas to confine agent contami-

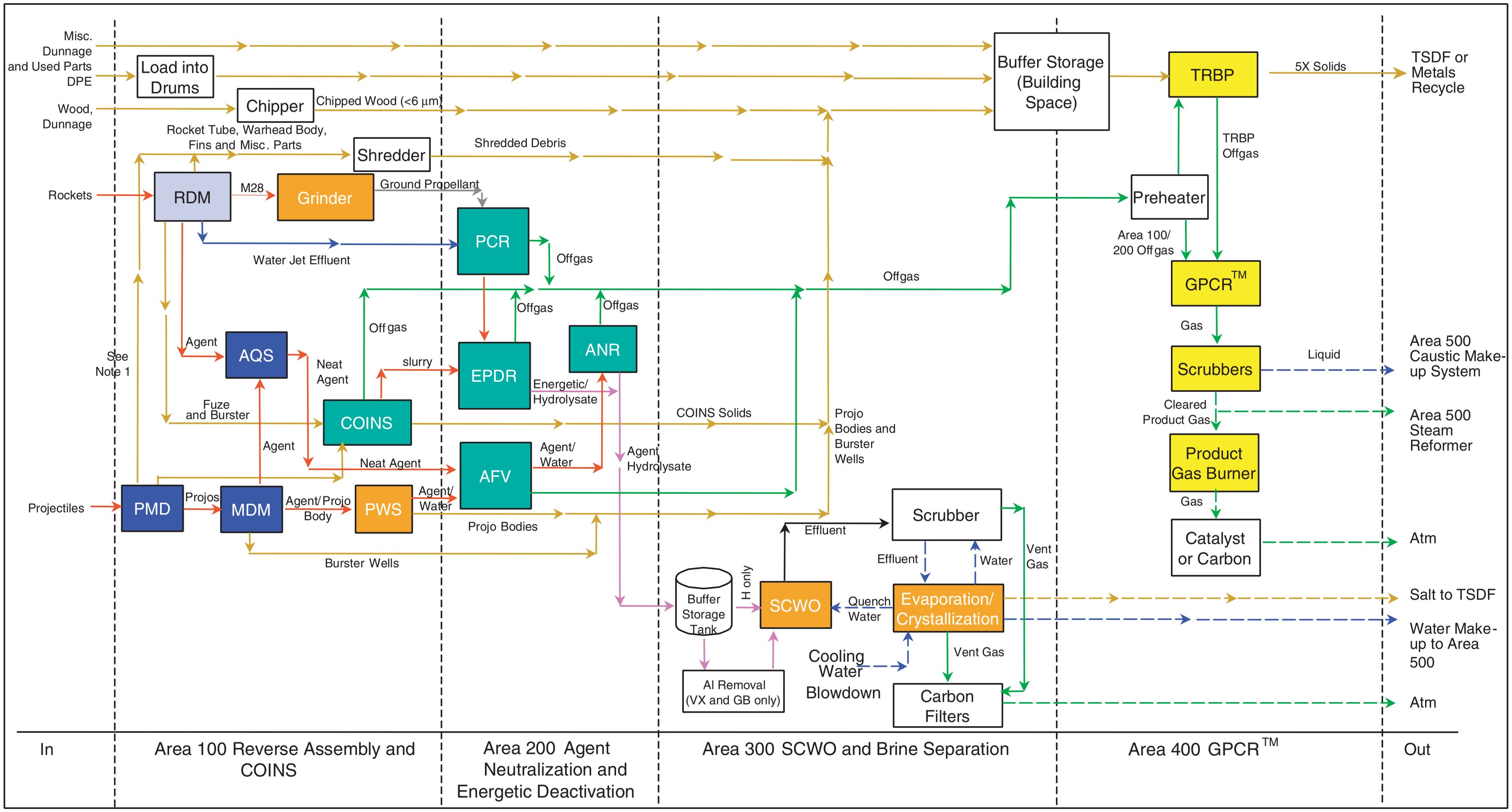

FIGURE 4-1 Schematic flow diagram of the Eco Logic technology package. SOURCE: Eco Logic (2001a).

TABLE 4-1 Summary of the Eco Logic Technology

Total Solution Treatment Process	Description
Area 100—reverse assembly/munitions access	In this area all munitions are received, agent drained, munitions bodies opened, energetics removed and the separate agent and energetics streams sent to Area 200 for hydrolysis or deactivation. The resulting solids are loaded under automatic or remotely operated control into process bins for transport to Area 400 for final 5X solids processing. Uncontaminated gases generated in this area are swept into a standard cascade air system; contaminated gas is fed to the GPCR™ in Area 400.
Area 200—hydrolysis of agent and energetics	This technology area uses hydrolysis at 90°C to destroy agent and to convert energetics to a deactivated, nonexplosive state. The liquid effluent is conveyed via pipe to the SCWO system in Area 300 for organic destruction. Gaseous effluent is fed to Area 400.
Area 300—supercritical water oxidation (SCWO)	Multiple parallel SCWO reactors are used to process the accumulated hydrolysate held in the SCWO feed tank. Liquid effluent from the SCWO system containing inorganic salts is processed in an evaporator/crystallizer, where salts are concentrated into salt cakes for disposal and clean water is recycled. The gaseous effluent from the SCWO, containing primarily carbon dioxide and oxygen, is scrubbed, monitored, and filtered through activated carbon before being released to the atmosphere.
Area 400—gas-phase chemical reduction (GPCR™)	The GPCR™ system is used to treat solid and gaseous streams. Organic compounds are thermally desorbed from the solids, which are 5X treated in the associated thermal reduction batch processors (TRBPs). The GPCR™ reactor treats the desorbed gas-phase organic compounds via chemical reduction with hydrogen.
Area 500—utilities/off-sites	Area 500 accepts or generates, then stores, all of the utilities within the facility. These include caustic, oxygen, demineralized and deionized water, hot water, primary cooling water, recycle water, potable water, secondary cooling water, steam, propylene glycol, hydrogen, instrument air, natural gas, nitrogen, and carbon dioxide.
Area 600—materials handling	Area 600 provides solid materials transfer of (1) munitions components from Area 100, (2) waste salts from Area 300, and (3) plant wastes such as DPE suits, dunnage, and filters to the TRBP/GPCR systems in Area 400.

nation and potential blast effects and to facilitate the repair of individual processing trains. Redundant trains are incorporated to tolerate the temporary failure of any device or devices while still maintaining nearly normal flow.

Area 100 uses munitions accessing devices from the Army's baseline incineration system disassembly operations wherever possible. The rocket disassembly operation is similar to the baseline system for dismantling the M55 rockets and separating the components. The projectile disassembly line is also very similar to that of the baseline system. However, a projectile washout system (PWS) has been added.

Both rockets and projectiles are reverse-assembled (or accessed) to allow the agent to drain away and to be weighed and processed separately from the energetics. Bursters and fuzes, but not the rocket propellant, are separated from the munitions and sent to COINS. Bursters are sheared prior to introduction into the COINS bath. Upon removal from the rocket, propellant is fed into a grinder, where it is mechanically size-reduced to facilitate dissolution, deactivation, and neutralization of any entrained agent and to assist in the processing of the energetic material in the hydrolysis reactors in Area 200.

In the Eco Logic process, rockets and projectiles containing the same agent are processed through the explosion containment rooms (ECRs) at the same time (e.g., GB-filled M55 rockets and 8-inch M426 projectiles or VX-filled M55 rockets and 155-mm M121A1 projectiles). This is unlike processing in the baseline system; it is possible because the projectiles do not contain energetics.

All solids resulting from processing are loaded (under automatic or remotely operated control) into process bins for transport to Area 400 for decontamination to a 5X condition. Gases released from the operations in this area, with the exception of those from the COINS (which are swept to Area 400 for destruction), are swept into the standard cascade air system utilized to control and condition the plant air in baseline system facilities.

Projectile/Mortar Disassembly

In the ECR, the projectiles are transferred one at a time onto the projectile/mortar disassembly (PMD) machine. This is a standard baseline system PMD machine, located inside an ECR and equipped with various tooling kits depending on the munition being processed and whether or not it is reprocessing rejects or processing leaking projectiles. The PMD machine is equipped with an eight-station indexing table and five special-purpose workstations oriented about the periphery of the indexing table. The indexing table receives projectiles and sequentially positions them in front of each station for the disassembly process. Four projectiles are processed in each PMD machine at a time, except when processing 8-inch projectiles. The greater length of the 8-inch projectiles means they must be processed one at a time in the PMD machine.

The PMD machine removes the lifting plug/fuze adapter assembly at the nose closure removal station, the fuze well cup (155-mm, M110 H projectile only) at the miscellaneous parts removal station (MPRS), and the burster (155-mm M110 H projectile only) at the burster removal station and discharges the projectile body containing agent out of the ECR on a projectile discharge conveyor to the burster removal verification station. If the PMD fails to remove the lifting plugs/fuze adapter assembly from the projectile due to galling, or if the fuze well cups or bursters cannot be removed, the projectile is sent back to the projectile reject system in the explosion containment vestibule. This system can resolve problems encountered with the nose closures and burster wells of the three types of projectiles at Blue Grass and problems with the fuze well cups and burster of the 155-mm M110H projectiles. Upon removal by the PMD, projectile lifting plug/fuze adapter assemblies and fuze well cups are transferred by the MPRS conveyor of the PMD machine to the projectile component discharge hopper.

Bursters from 155-mm, M110 mustard agent projectiles are transferred by the PMD burster transfer conveyor to the burster size-reduction machine. This is a baseline-system rocket shear machine (RSM) modified to perform burster size reduction. The M6 bursters from these projectiles are cut into three equal-length sections to reduce the time required to separate/deactivate the tetrytol explosive in the COINS. From the burster size-reduction machine, the burster sections drop into the projectile component discharge hopper, through double blast gates, to the projectile component discharge chute and into the COINS for processing. When processing nonenergetically loaded 155-mm M121A1 and 8-inch M426 projectiles, the lifting plug/fuze adapter assemblies follow the same path, but instead of going to the COINS, are conveyed by the projectile component transfer conveyor to bins that feed projectile and rocket metal parts to the thermal reduction batch processor (TRBP).

Munitions Demilitarization Machine

The function of the munitions demilitarization machine (MDM) is to remove, by reverse assembly, the burster well and agent from 105-mm, 155-mm, and 8-inch projectiles. The MDM is equipped with a six-station indexing table and four special-purpose workstations oriented about the periphery of the indexing table. The load/unload station receives projectiles from the pick-and-place machine. The projectile is then processed from station to station as it is indexed around the table until it returns back to station 1. The pick-and-place machine then removes the disassembled projectile and delivers it either to the processing tray on the MDM indexing hydraulic feed conveyor or, if the burster pull or agent drain operations were unsuccessful, to the MDM reject stand. (A boring machine is then used either to cut burster wells that cannot be removed at the pull-and-drain station or to cut burster wells loose from projectiles that have the burster wells welded to the projectile casing.)

The MDM operation in the baseline system has been modified in the Eco Logic process with the addition of a burster well transfer station. This device removes the pulled burster well from the MDM and drops the burster well into a collection hopper, which is periodically emptied into a TRBP bin for subsequent processing. To address the issue of foaming rounds,[1] Eco Logic proposes to develop a froth collection system for mustard agent projectiles similar to that used during disposal operations at the Johnston Atoll baseline system facility (Eco Logic, 2001a). The froth collection system would include a sealing/venting cap that seals the projectile nose to capture and direct the foamed/frothed agent and gases to an appropriate collection chamber. A vacuum drain system evacuates this chamber and transfers agent to the agent collection tank. The projec-

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[1] Foaming rounds refer to the phenomenon experienced at baseline facilities when agent overflows and/or sprays from a mustard agent munition during the burster pull operation.

tile body is drained of agent, indexed to the crimp station (not used at Blue Grass), and then indexed to station 1 for removal.

It is noteworthy that the agent drain portion of the MDM pull-and-drain station is a major cause of downtime in the baseline process. When processing projectiles with crystallized or gelled GB, or with mustard agent, it is the limiting production rate factor. A major contributor to the downtime of the projectile agent drain system is the agent quantification system (AQS). Improvements to the projectile agent drain system that would be considered during further development include the following (Eco Logic, 2001a):

- enlarging the drain tube through which the agents are drawn out of the projectile to reduce plugging
- increasing the purge air pressure, rerouting piping, and increasing preventive maintenance
- providing a PWS for 155-mm, M110 mustard agent projectiles

The Projectile Washout System

The PWS removes residual agent and agent heels from 155-mm and 8-inch projectiles after they have been deburstered and drained of liquid agent. A PWS is to be incorporated into the technology provider's design to ensure the removal of mustard agent heels or gelled GB nerve agent from projectiles. This has been a recent addition to the Eco Logic process design; consequently, the design of the equipment had only begun when this report was prepared, and only a few details were available. The PWS will be incorporated as a station on or adjacent to the MDM machine and will be used for the 8-inch M426 GB projectile and for the 155-mm, M110 mustard agent projectile. These modifications may be added at a sixth station of the MDM since this is a spare station on the baseline machine configuration and it follows the pull-and-drain station. A liquid handling system is expected to be part of the design to handle incoming and outgoing washout liquids. The results of PWS testing performed at the Army's CAMDS facility are being studied for possible incorporation into the PWS design for the Eco Logic process.[2] Test data developed at CAMDS by Parsons (another technology provider in the ACWA program) for washing out 4.2-inch mortars will also undoubtedly be helpful in developing a projectile washout system for the Eco Logic process.

The Rocket Dismantling Machine

The RDM processes two rockets at a time, the same as the rocket shear machine (RSM) in the baseline system. While one rocket is being processed at the punch-and-drain station, another is being processed through the dismantling sequence. This is done in the same way as on the RSM in the baseline system. In the Eco Logic design for RDM operations, the baseline rocket metering feed assembly, located in the unpack area (UPA), will be modified to accommodate rockets oriented with the fin assembly heading first into the RDM.

Once gripped in place in the RDM, the rocket (still in its shipping/firing tube) is punched and agent is gravity-drained to the AQS and then collected in an agent collection system. One punch hole on top serves as a vent for incoming air, hot water, or steam, while agent drains by gravity from two other punch holes on the bottom. The three punch holes are oriented exactly as they are in the baseline operation. Drain time is approximately 1 minute, which is also the same as in the baseline operation.

A rotating tube cutter severs the fiberglass shipping tube at the punch-and-drain station to expose the fuze and adapter of the rocket. This cut is made prior to all of the other cuts that are made on the rocket itself. This operation occurs simultaneously with the draining of agent. When the rocket is transferred to the cutting station, the short fiberglass section and the aluminum fuze end cap that covered the fuze and adapter of the rocket remain in place until the next rocket enters the punch-and-drain station. The next rocket entering the ECR pushes the short fiberglass section and end cap downstream to be discharged into the metal parts hopper.

Four design improvements have been made to the punch-and-drain station (Eco Logic, 2001a):

- O-ring seals are installed around each of the punches to provide a liquid tight seal between the fiberglass shipping tube and the punch clamps.
- Troughs are installed to bridge the gaps between all of the clamp arms and the two feed conveyors.
- The vent punch cylinder has been changed to a double rod cylinder, with the center of the cylinder rod drilled out to pass steam and liquid into the warhead. The steam or water exits the cylin-

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[2] Note that washout of the liquid agent and residues/heels together might be possible if the treaty requirements for agent quantification can be satisfied.

der on the punch end through two ports from the sidewall of the cylinder rod to maximize the cleaning action of the steam or water.

- An overhead roller is used on the first feed conveyor to ensure that constant pressure is applied to the rocket while it is being driven through the rotating tube cutter and the punch-and-drain station. This roller is located between the rotating tube cutter and the first clamp assembly of the punch-and-drain station.

When processing GB agent rockets, low-pressure (~5 psig) saturated steam at less than 228°F (109°C) (Eco Logic, 2001a) is fed through the vent punch to rinse out any residual agent. The process steam discharged from the warhead is then piped through a condensation system to ensure that all steam is fully condensed. After the warhead has been steamed out, it is rinsed with approximately one warhead-volume (~1 gallon) of water. This condenses the steam vapor inside the warhead and assists in washing residue agent out of the warhead cavity.

The rinse water, steam/condensate, and residual agent bypass the AQS and go to the agent collection system. After the agent is removed from the warhead and the warhead has been steamed out, the rocket is rotated 90 degrees to minimize agent leakage from the punch holes.

The rocket in its shipping/firing tube is moved to a cutting station, where it is cut into five sections. The first three cuts use a tube cutter and remove (1) the fuze, (2) the burster, and (3) the warhead, leaving the motor casing and fin assembly. The fuze is fed via a dumping conveyor section into the COINS hopper. The fourth cut is done by a water jet cutter with garnet abrasive and is made just above the nozzle plate to separate the motor casing from the fin assembly. These last two cuts, which remove the warhead and separate the motor casing from the fin assembly, also cut through the fiberglass shipping/firing tube.

The Propellant Grinder

M28 double base propellant removed from a rocket by the RDM is sent to a propellant grinder to decrease the time required for hydrolysis. The grinder is designed to receive a single 31-inch propellant grain from the RDM. Although antiresonance rods associated with the propellant grain should be removed at an earlier station within the RDM, the grinder will be designed to allow safe grinding of propellant and antiresonance rods, as well as coaxial igniter cables. The propellant is reduced to granules that will pass through a $\frac{1}{4}$-inch screen before exiting the grinder.

A constant water level is maintained inside the grinder hopper at all times, while the propellant is ground under water to prevent spark ignition and eliminate the potential for fires. Only one propellant grain will be processed at a time. To ensure that propellant does not enter into the shaft bearings, the seals are designed to be flooded with clean water to force any propellant away from the seals and bearings. Another safety feature is equipping the grinding rotor with an automatic reverse rotation feature to clear any jams that occur. To minimize impact initiation of the propellant, a low-speed grinder is used.

To conserve the amount of water used in the grinding system and to ensure that the correct ratio of water to propellant grain granules is delivered to the storage tank, a series of two pinch valves will be used on the pipeline discharging from the grinder hopper. Since the specific gravity of the propellant is higher than that of water, the size-reduced propellant will settle on the bottom of the grinder discharge hopper. The first pinch valve will open, allowing the size-reduced propellant to settle in between the two pinch valves. (The propellant will displace any water in this space.) The volume of the piping between the two pinch valves will be sized so that a ratio of 35 percent by weight of propellant to water can be maintained. When the upper pinch valve is closed and the lower pinch valve is opened, only a propellant-water mixture containing 35 percent by weight propellant will be discharged to the propellant reactor tank.

COINS

The COINS process uses an overhead conveyor system that collects metal parts containing explosive energetics components (fuzes and bursters) into baskets that are moved through a tank containing a caustic bath that hydrolyzes the energetic materials in the metal parts. No propellant is sent to the COINS.

The Eco Logic EDP for Blue Grass uses two COINS troughs that operate in parallel (Eco Logic, 2001a). The COINS troughs are totally independent, and each has its own airtight enclosure. The two troughs, each measuring approximately 11 feet wide by 20 feet long, are filled with 20 weight percent sodium hydroxide solution in which the baskets full of munition parts are suspended. The caustic dissolves the energetics from the steel parts; it also dissolves the aluminum and zinc as well as the energetics in the fuzes. The aluminum-caustic reaction produces hydrogen; therefore, the enclosure is purged with nitrogen to preclude the formation of an explosive mixture.

An overhead chain conveyor above each trough carries the attached baskets through the solution using an indexing system. This system includes three dwell stations, each of 1 hour duration. After passing through the bath, the conveyor path raises the baskets just before the wash station to an elevation above the COINS trough for the wash operation. Water sprays remove most of the caustic solution from the baskets and the parts, and the baskets are moved to a TRBP bin loading station. Upon reaching the loading station, the baskets are rotated approximately 90 degrees, discharging the basket contents into a gravity chute that leads to a TRBP bin located below. The TRBP bins are positioned on a roller conveyor, and entry and exit to the COINS enclosure is via an airlock to maintain the nitrogen atmosphere in the enclosure. The spent caustic from the COINS operation is sent to Area 200.

Area 200—Hydrolysis of Agent and Energetics

The key features of Area 200 are shown on Figure 4-1. One of two parallel hydrolysis trains in Area 200 processes agent; the other processes rocket propellant and other energetics from rockets and projectiles. Each train consists of three reactors that are operated in a batch mode. While one reactor is being loaded, the contents of another are undergoing reaction, and the product in the third is being tested and unloaded. Agent and energetics are hydrolyzed with caustic. However, mustard agent must be treated in a two-step process involving reaction with warm water. When the reaction is complete, the pH of the strongly acidic hydrolysate is adjusted with caustic solution. This prevents the formation of undesirable vinyl compounds, which are toxic and resist further treatment.

Energetics Hydrolysis

The energetics from the RDM, the propellant grinder, and COINS operations are hydrolyzed to completion in Area 200 within the propellant collection reactor (PCR) and the energetics/propellant deactivation reactor (EPDR). The PCR is used as a collection vessel for the material processed by the propellant grinder. Both the grinder and the PCR are operated in a continuous manner. As the propellant is discharged through the pinch valve arrangement associated with the grinder, the water and propellant mix is accumulated in the PCR. In addition, the water jet effluent from the RDM is filtered to remove the garnet abrasive and is also collected in the PCR. The filter is designed to remove 100 micron or larger garnet from the cutter effluent.

Hydrolysis can occur in the PCR, which operates at 194°F (90°C) with a caustic concentration of nominally 20 weight percent. Caustic (50 weight percent) is added to the PCR to control the concentration of NaOH. A hot water heat exchanger (240°F; 116°C) is used to heat and maintain the reactor temperature at 194°F, and cooling is accomplished with 93°F (34°C) water if necessary. This allows the rate of heat release of the hydrolysis reaction to be controlled. Mixing in the PCR is accomplished with an agitator, and a recirculation pump with a 3/16-inch mesh filter is used to remove any solids that may have passed through the COINS process or the propellant grinder, as a potential exists for plugging either by the metal parts or by residual propellant. The filters are designed to remove metal parts, such as pieces of antiresonance rods and residual propellant. The hydrolyzed (or partially hydrolyzed) product from the PCR is fed to the EPDRs.

As previously noted, the three parallel EPDRs are run in batch mode. They receive feed from the COINS and the PCRs. The following describes the planned 24-hour reaction sequence that would be used for each batch. The first 8 hours are used to fill the reactor with the product streams from the COINS and PCRs. After these react for 2 hours, a sample is taken to check for the presence of energetics by HPLC and determine whether or not the concentration of agent is less than an established limit. The next 6 hours are used as hold time and additional reaction time during testing. After a minimum of 13 to 16 hours in the reactor, the hydrolysate is pumped to the SCWO buffer storage tanks. The final 2 hours of the 24-hour cycle are used for recharging the reactor with caustic. The planned operation of the three reactors will stagger the batch cycles such that at any time one reactor is filling, one is being used for reaction or undergoing analysis of reaction products and one is discharging to Area 300.

At the completion of the batch operation, the hydrolysate is discharged into a buffer storage tank prior to processing through the SCWO reactor. The buffer storage tank has a volume equivalent to 24 hours of feed to the SCWO reactor. Any offgas that is produced in the PCR or EPDR is collected in the offgas system and sent to the GPCR™ in Area 400 for further processing.

Gases from the hydrolysis reactors pass through a vent condenser. Liquids from the condenser are returned to the EPDR, and gases are sent to the GPCR™ in Area 400.

Agent Hydrolysis

Immediately after being drained from the munitions, neat agent is pumped into the agent feed vessel, which holds approximately 1,800 gallons at its normal operating level (65 percent full), and provides the interface between Area 100 and the agent neutralization reactor (ANR). A second vessel, the agent emergency collection vessel, is also provided for collecting neat agent or spent decontamination solution. Liquid from these vessels is fed to the ANR for hydrolysis.

For both the VX and GB disposal campaigns, the first step is to fill the ANR with sodium hydroxide solution followed by the agent. A 5 weight percent NaOH solution is used for the GB campaign and a 20 weight percent solution for VX. The VX reaction requires a pH greater than 12 and the GB reaction requires a pH greater than 7 to prevent agent reformation reactions from occurring. The reactor contents are continuously agitated to keep the VX or GB agent from separating from the aqueous NaOH to form two liquid phases. For mustard agent, the process entails first reacting the agent with warm water, during which the pH drops to less than 1. The pH is then brought up to between 11 and 12 using 20 weight percent NaOH solution. The reactor contents are continuously circulated by the agent hydrolysate pumps through the agent reactor circulation heat exchanger.

Exiting gases pass through the agent reactor vent condenser, where the vapors are cooled with secondary cooling water. Liquids are returned to the ANR, and gases are sent to the GPCR™ reactor. As with the processing of energetics, reactor temperature is monitored and controlled very carefully to prevent excessive heat buildup and a runaway reaction. The heat exchanger and a water jacket on the reactor control the temperature of the reactor contents, accepting either hot or cold water as necessary to maintain 194°F. Additional caustic solution can be added to meet the specific pH requirement for each disposal campaign.

When the reaction step is complete, the reactor contents undergo a hold-test-release program. The contents are tested to confirm the total hydrolysis of agent, and extra reaction time is allowed if the sample results indicate that agent is still present. Once complete hydrolysis of the agent has been confirmed, the reactor contents are pumped to the agent hydrolysate surge vessel (AHSV), which provides the surge capacity necessary to interface the semibatch process of the reactors in Area 200 and the continuous operation of the SCWO reactors in Area 300. The AHSV also has the capacity to hold one extra day of Area 200 production should Area 300 be operating at reduced throughput or be totally shut down. Likewise, the AHSV can be used to continue a limited supply of hydrolysate feed to Area 300 should Area 200 be temporarily out of service. Exiting gases pass through the agent surge tank vent condenser, where the vapors are cooled with cooling water. Liquids are returned to the AHSV and gases are sent to the GPCR™ in Area 400.

The contents of the AHSV are continuously recirculated using the agent hydrolysate transfer pump and are returned to the AHSV through a jet mixer. The vessel contents are agitated and maintained at 194°F. The pH is monitored and controlled at all times to eliminate the possibility of agent reformation. Hydrolysate from the AHSV is supplied to the SCWO reactor units in Area 300 through the agent hydrolysate transfer pump.

Area 300—Supercritical Water Oxidation

The SCWO process is designed to destroy CWC Schedule 2 compounds and other hazardous aqueous organics in the hydrolysates by converting them to CO_2, water, and inorganic salts. Key components of the Area 300 design are shown in Figure 4-1. A diagram of the SCWO reactor is shown in Figure 4-2. The process takes advantage of the properties of supercritical water (above 374°C and 22.20 MPa), in which organics, oxygen, and water are completely miscible in a single, dense gas phase with properties conducive to fast reaction and high mass-transfer rates. High destruction efficiencies are possible with reactor residence times on the order of seconds.

The agent hydrolysate and energetics hydrolysate produced in Area 200 are combined and treated by SCWO in Area 300. The Eco Logic design for a Blue Grass facility uses five transpiring-wall SCWO reactors (Eco Logic, 2001a). Only three SCWO reactors would be in use during normal operations. Each of the five identical SCWO reactors has an 11-inch internal

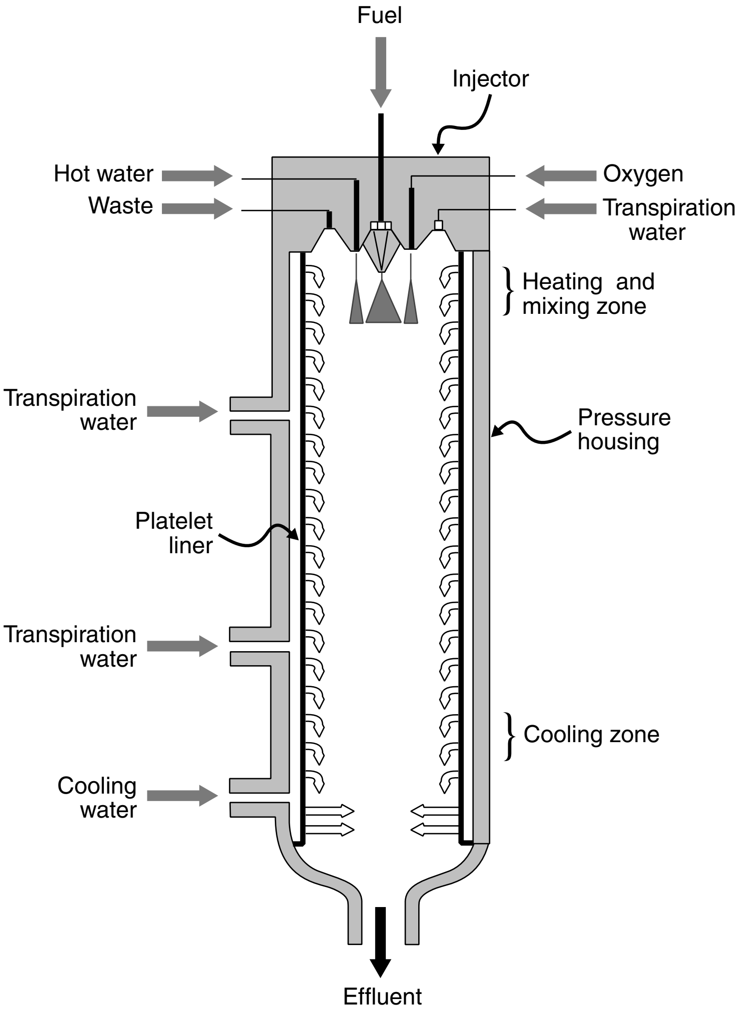

FIGURE 4-2 Schematic diagram of Foster Wheeler transpiring-wall SCWO reactor. SOURCE: Eco Logic (2001a).

diameter and is 7.5 ft in length, compared with the 6-inch diameter and 6-ft length of the reactor used in Demo II. The SCWO reactors are fed from a hydrolysate feed tank. Since suspended solids may be present in the feed, the hydrolysate recirculation pump maintains a continuous circulation of feed to and from the supply drum. When processing mustard agent hydrolysate, the total aluminum content is expected to be less than 400 ppm, which the technology provider considers to be an acceptable level to prevent scale buildup in the SCWO reactors. When processing VX and GB, an aluminum removal system will be needed to reduce aluminum in the energetics hydrolysate feed to less than 500 ppm so that the total in the combined hydrolysate feed is less than 400 ppm (Eco Logic, 2001a). At present, Eco Logic does not possess and has not developed this technology.

The EDP design uses oxygen as the oxidant and propylene glycol as the supplemental fuel, whereas in the Demo II tests, the reactor used air and kerosene, respectively. Kerosene is used during start-up as fuel, but in a recent change—instituted as this report was being prepared—is now also continued at a low flow rate during operation (Foster Wheeler, 2001). The change from air to oxygen is being made to increase SCWO reactor capacity, and the partial change of fuel to propylene glycol is being made for safety reasons (propylene glycol has a much higher flash point than kerosene).

The input to each SCWO reactor will be an aqueous solution containing agent and energetics hydrolysates. Mixing these streams provides a single feed stream for the SCWO step and simplifies the overall process. In the reaction zone at the upper end of the reactor, operating conditions are approximately 815°C and 23.5 MPa gauge, which is well above the critical temperature and pressure of pure water. The hydrolysis oxidation reaction starts at approximately 500°C. The oxidation reaction is autogenic (that is, the heat released from the oxidation reaction is sufficient to maintain the reactor temperature). Kerosene is added as an auxiliary fuel at start-up, and propylene glycol and kerosene supplement the fuel value of the hydrolysate feed as needed to maintain an autogenic feed for the SCWO reactor. As shown in Figure 4-2, the injector at the top of the reactor has separate nozzles for feeding oxygen, hot water, the hydrolysate/fuel mixture, and transpiration water. Oxygen is fed through the reactor injector as the oxidant. The flow of oxygen is controlled to maintain a slight excess of oxygen during reaction. During start-up and shutdown, a nitrogen-to-oxygen blend in the ratio of 4:1 or greater is used to ensure that high oxygen concentrations do not occur in downstream equipment. If necessary for pH control of the reactor effluent, sodium hydroxide solution is added to the feed/water mixture.

The hydrolysis of VX and GB in hot sodium hydroxide solution and of mustard in hot water results in the formation of complex organic compounds. These molecules contain heteroatoms such as sulfur, chlorine, fluorine, and phosphorus, which, when oxidized, form acids. The excess sodium hydroxide present in the hydrolysate neutralizes these acids to form salts. For example, the following reactions are likely:

$$S + O_2 + H_2O \rightarrow H_2SO_4 \text{ (not balanced)}$$
$$H_2SO_4 + 2\ NaOH \rightarrow Na_2SO_4 + 2\ H_2O$$
$$P + H_2O + O_2 \rightarrow H_3PO_4 \text{ (not balanced)}$$
$$H_3PO_4 + NaOH \rightarrow NaH_2PO_4 + H_2O$$
$$NaH_2PO_4 + 2\ NaOH \rightarrow Na_3PO_4 + 2\ H_2O$$

These salts could precipitate on the wall of the SCWO reactor as solids at supercritical conditions.

A unique feature of the Foster Wheeler SCWO reactor is its full-length transpiring-wall liner, shown in Figure 4-2. Foster Wheeler claims that this liner design protects the reactor walls from corrosion and salt deposition. The reactor liner is fabricated from multiple layers of Inconel 600 assembled in sheets of what the technology provider refers to as “platelets” to produce transpiration pores. Deionized water is added to the SCWO reactor through transpiration pores in the liner wall. Deionized water is also added to the top of the reactor to protect the injector surface from corrosion and salt deposition. The transpiration water is added along the entire reactor length such that the temperature along the center line decreases from approximately 815°C at the top to 595ºC at the bottom.

The products of hydrolysate oxidation are CO_2, H_2O, and salts; N_2 and excess O_2 are also present. At the exit of the reactor, recycled water recovered from the downstream evaporator/crystallizer unit is injected to quench the reactor products to the subcritical temperature of 315ºC, which results in essentially all of the product salts redissolving.

Other recycled water from the downstream evaporator/crystallizer unit is used as transpiration water for liner and injector protection after being deionized and preheated to 288ºC by heat exchange with the reactor effluent. A heater is used to provide the 595ºC water used as part of the start-up feed to the reactor. At start-up, a measured quantity of the initiating kerosene fuel, 595ºC hot water, and a nitrogen/oxygen blend are added through the reactor injector assembly at the centerline of the reactor.

During operation of the SCWO reactor, the reactor effluent is reduced from 24.2 MPa to approximately 310 to 350 kPa (45-50 psig) through a pressure control valve. The effluents from all reactor trains are combined before entering a venturi scrubber and knockout drum, where the liquid slurry is separated from the vapor and offgas. The overhead from the effluent knockout drum goes into a separator drum, where the vapor and offgas are separated and cooled to 49ºC in a vent condenser with cooling water. The condensate is combined with the slurry. The uncondensed effluent offgas is continually monitored for CO, CO_2, NO_x, N_2O, and O_2, filtered, and vented to atmosphere through the vent gas stack.

The slurry effluent is analyzed for residual organics, and if it meets total organic carbon (TOC) specifications, it is pumped to an evaporator/crystallizer system, where water is evaporated and the salts crystallized for off-site disposal. These inorganic salts are readily stabilized and are suitable for disposal in existing permitted landfills. The recovered water is either reused in the process, sent to a unit that produces deionized water, or used for making caustic solution. If the effluent does not meet TOC specifications, it is routed to an off-specification effluent tank and then returned as part of the SCWO reactor feed.

Area 400—Gas-Phase Chemical Reduction

Key features of Area 400 are shown in Figure 4-1. The GPCR™ system consists of a central GPCR™ using a hydrogen-rich sweep gas reactor for the destruction of organic waste, followed by a multistage scrubbing system to remove inorganic compounds and light hydrocarbons from the reacted gas stream. The scrubbing system is followed by a hold-test-release system. Various mechanisms are used to prepare and feed the various waste types to the GPCR™ reactor. A TRBP processes bulk solid materials and delivers vaporized waste to the GPCR reactor, and a preheater system is used to inject other contaminated gas streams.

Thermal Reduction Batch Processor System

Five TRBPs are employed to desorb organic contaminants from solids such as dunnage and munitions housings and to pyrolyze organic materials such as cellulose and polymers. The organic waste vapors are removed from the TRBP by a flow of sweep gas and are conveyed into the GPCR™ reactor for destruction. The sweep gas may be hydrogen and/or recycled product gas from the GPCR™ reactor.

Each TRBP has an inner vessel and an outer box. The outer box provides the heat source, the physical support structure, and the platform for connecting to utilities and other plant interfaces. Materials to be treated are placed in the inner vessel.

The processing cycle operational sequence of the TRBP involves the following:

1. loading/purging
2. heating
3. soaking
4. cooling
5. unloading

The bulk wastes to be processed are contained in TRBP bins or on racks: projectiles on specially designed racks, other wastes in TRBP bins. DPE suit material, spent activated carbon, garnet, and other maintenance wastes, including hydraulic oil, are loaded into drums that are then placed in TRBP bins. Rocket parts are shredded before loading into TRBP bins, and a hammer mill preprocesses wooden pallets that are loaded directly into TRBP bins. The hammer mill operation is done in an enclosure to control dust and fugitive emissions. All TRBP rack and bin loading operations are part of Area 600. The bins or racks are placed in the TRBP inner vessel using a TRBP loader car. The TRBP inlet door is closed, and the integrity of the seal is verified. The inner vessel is then purged of oxygen (air) using nitrogen.

Because the gas purged from the TRBP may contain agent, it is sent to an activated carbon filter system before being sent to the facility HVAC system. When the measured oxygen is less than 1 percent by volume, the vent line is closed, the TRBP is opened to the GPCR™ reactor, and the flow of sweep gas is begun. The inner vessel is gradually heated, with the heating rate limited by the maximum organic loading to the GPCR™ reactor. During this phase of operations, sludge from the Area 400 scrubbers is fed to the TRBPs.

When the organic vaporization rate drops significantly, the temperature of the TRBP inner vessel is raised to a minimum of 1,100°F for 4 hours to meet 5X decontamination requirements for all contained solids, including the sludge residues. The TRBP burners are then shut off, and the combustion chamber is cooled with air. Cooling of the waste load is aided by the addition of a water spray. The steam that is generated carries through to the GPCR™ reactor and purges the hydrogen from the TRBP inner vessel. When all of the hydrogen has been removed, the gas flow is opened to the TRBP scrubber, and the inner vessel is isolated from the GPCR™ reactor. The scrubber cools the gases (steam) from the TRBP, condenses the water, and circulates the water back to the TRBP spray nozzles. Nitrogen is added to the TRBP inner vessel to maintain a positive pressure. The gases leaving the TRBP scrubber are directed to the facility HVAC system. When the TRBP inner vessel reaches a workable temperature (e.g., below 200°F), the TRBP outlet door is opened and the bulk waste is removed.

Eight isolated rooms, called “smoking rooms,” are used for holding the TRBP bins that contain high-carbon-content materials from processing wood and DPE suits. Residual carbon in the wood/DPE loads can smoke for a few hours or, possibly, 2 or 3 days (Eco Logic, 2001c). The smoking rooms allow the contents of the bins to cool down under controlled conditions after removal from the TRBPs. Each room has a roll-up door on either side and a small water spray, exhaust blower, and carbon filter above the bin space, which vents to the atmosphere through an exterior wall. The water spray is low in volume and has been described as a mist. The oxygen supply to these rooms is limited to prevent the contents of the bins from igniting. Carbon monoxide formation is expected by the technology provider but has not been studied.

GPCR™ Reactor Feed Preheater System

The preheater system includes all of the equipment required to heat any combination of the gaseous inputs to the GPCR™ reactor (including hydrogen, steam, the offgas from Areas 100 and 200, and recycled GPCR™ product gas from storage), as well as any liquid being sent. The outputs from the preheater system are fed to the GPCR™ reactor and/or the TRBPs (hydrogen and steam only for TRBPs).

Three direct-fired heat exchangers are employed in the facility design for Blue Grass. The first heat exchanger preheats the recycled product gas and the offgases from Areas 100 and 200 from 150°F to 1,200°F. These gases are injected directly into one of the two GPCR™ reactors. A second heat exchanger preheats the pure hydrogen feed from 86°F to 1,382°F for injection into the five TRBPs. The third heat exchanger preheats saturated steam from 300°F to 1,382°F for injection into the five TRBPs and two GPCR™ reactors. The three preheaters use fuel gas or GPCR™ product gas from storage tanks. The exhaust gases from these preheaters are passed through an activated carbon filter. Stacks from the three preheater fireboxes combine into a common stack header for all of the other fired heaters in the facility, including the TRBPs and the product gas burner. The combined stack gas stream is polished by passing it through a catalytic oxidizer to ensure the complete destruction of any remaining organic compounds.

GPCR™ Reactor

The GPCR™ reactor chemically reduces the organic contaminants found in the waste streams. As noted previously, the feed streams include preheated hydrogen, superheated steam, Area 100 and 200 offgas, liquids from the preheater, and vaporized waste from the TRBPs. These streams are mixed in static mixers and enter the reactor at 650°C to 750°C. The feed gases enter the vertically oriented reactors tangentially at the top and swirl downward before exiting upward through a vertical center tube. Each reactor is heated internally by a set of 18 vertical, electrical-radiant-tube heating elements that heat the gases from 650°C to 900°C.

The GPCR™ reactor and vertical center tube are designed to provide a residence time of between 2.5 and 10 seconds. Hydrogen and steam react with the organic waste inputs to produce hydrogen chloride, hydrogen fluoride, phosphorus oxides or phosphine, hydrogen sulfide (depending on the input waste composition), and methane. Secondary steam reforming reactions between the hydrocarbons and steam produce carbon monoxide, carbon dioxide, and hydrogen. Undesirable components formed in the reaction include black tarry material, which can cause problems downstream.

Steam aids in the reforming reactions, promotes good heat transfer between the heating elements and the gases, and minimizes the formation of tar resulting from pyrolysis and/or coking of the waste particles. A shroud, which is purged and blanketed with CO_2 gas, is located above the reactor roof where the heating element head connectors and an electrical bus are installed. Blowers recirculate the CO_2 gas through the reactor shroud. The circulating CO_2 gas serves to cool the shroud to below 200°C to prevent failure of the bus bar support insulators and heating element head connectors. The hot CO_2 from the GPCR™ reactor shrouds pass through air-cooled fin heat exchangers and back to the blower suction. Fresh makeup CO_2 is added to the blower suction, and a purge stream of CO_2 is vented to the atmosphere. Online monitoring of CO_2 pressure and makeup rates are used in the process control system to detect leaks in the radiant-tube heating elements while ensuring no oxygen enters the system through any such leaks.

Gas Scrubbing and Water Treatment

The hot gases from the GPCR™ reactor are cleaned by passing them through

1. a quench tower in which the gases are contacted with a slightly acidic aqueous stream
2. a variable-throat venturi scrubber in which the gases are again contacted with a slightly acidic aqueous stream, and
3. a packed-tower scrubbing system in which the gas stream is contacted first with water and then with caustic

Based on experience with similar operating GPCR™ systems, Eco Logic expects that any naphthalene that may be present in the reactor exit gas will be mostly condensed. However, some of the naphthalene may coat the tubes of a heat exchanger in the scrubbing solution circulation system. For this reason, a spare heat exchanger is provided. Naphthalene, with a melting point of 80°C, can be melted off the walls of the heat exchanger tubes using steam. A high pH (above 12) is maintained in the final scrubber to aid in removal of hydrogen sulfide (during the mustard campaign) or phosphine (during the GB campaign) should any be present. The cleaned, cooled product gas exiting the top of the final scrubber goes to the compressors and on to the product gas storage tanks.

Solids that settle from the scrubbing solution will periodically be drained into drums and sent to the TRBPs for processing as site-generated waste.

Because the organic chemicals are destroyed in the GPCR™ reactor by reduction reactions, the main products are gases such as methane, carbon monoxide, and carbon dioxide. These gases, plus the excess feed hydrogen, must be removed at a controlled rate to maintain the set system pressure; fluctuations in the system pressure are undesirable and may lead to process upsets. To accommodate the fluctuating reactor loading and gas production, the compressor must be controlled to remove gas from the system at a variable rate. This is accomplished with a variable-speed drive on the compressor.

Rotary sliding-vane compressors are employed to increase the product gas pressure from 0 kPag to 500 kPag for storage in the product gas storage tanks and to control the pressure profile over the entire system from the TRBPs to the scrubbers. Each of the three compressors is rated for 50 percent of the total flow—two compressors are operated in parallel and the third compressor is on standby. Each compressor has associated water-cooled precoolers, intercoolers, and aftercoolers. Each cooler has a knockout pot for liquid separation. Drained liquids from all the knockout pots are combined, transferred to a knockout collection vessel, and then to an organic liquid settling tank, where any free organic liquids are recovered and eventually processed in Area 300.

Five product gas storage tanks having three functions are employed. The first tank entered is for testing the product gas composition. Compounds indicative of the level of destruction are measured with a gas chromatograph; hydrogen, methane, carbon dioxide, and carbon monoxide are also measured. If these tests are unsatisfactory, the gas is sent to a backup storage tank for recycle to the TRBPs or preheater. If the tests are satisfactory, the gas is released to one of three hold-test-release tanks for further testing (see section on methods development later in the chapter). If the presence of agent is confirmed, the gas will be recycled to the TRBPs or preheater for processing. Gas from the GPCR may also be sent to a product gas burner and then to a catalytic oxidizer.

Area 500—Utilities

Area 500 consists of off-the-shelf package units that accept, generate, and/or store all of the utilities required by the facility. The 16 utilities provided by Area 500 are summarized as follows:

- aqueous NaOH caustic solution at both 50 percent and 20 percent concentration
- primary cooling water used in the heat exchangers in Areas 300, 400, and 500, i.e., those with essentially no possibility of agent on the hot side
- saturated steam produced from demineralized water
- natural gas provided by the depot infrastructure
- liquid oxygen compressed and heated to 4,500 psig and 75°F for use by the SCWO reactors
- recycle water received from the Area 300 evaporation system and the Area 400 stack gas condensate
- propylene glycol for supply to the SCWO reactors
- liquid nitrogen compressed and heated to 4,500 psig and 75°F for use in the SCWO reactors or used as a medium-pressure stream delivered to Areas 100, 200, 400, and 500 for purging
- demineralized and deionized water produced from recycle
- process water provided by the depot infrastructure
- hydrogen produced on-site by an off-the-shelf catalytic steam reformer
- carbon dioxide supplied to Area 400 electrical equipment
- hot water circulated through a closed system to Area 100 and 200 heat exchangers
- secondary cooling water circulated through a closed system to Area 100 and 200 heat exchangers
- instrument and plant air provided by the depot infrastructure
- electricity provided by the depot infrastructure

Area 600—Materials Handling

Area 600 includes handling and shredding of materials. Area 600 handles four waste streams upstream from the TRBP as follows:

- delivers the dunnage from the unpack of the munitions to a shredder, size-reduces these materials, and delivers them to the TRBPs for processing
- picks up metal parts from COINS and delivers them to the TRBPs
- picks up metal parts from munitions disassembly that bypass COINS, size-reduces these materials, and delivers them to the TRBPs for processing
- picks up trays of drained projectile bodies, loads the trays into racks, and delivers them to the TRBPs

Area 600 handles two 5X-decontaminated waste streams downstream from the TRBPs as follows:

- takes the decontaminated materials from the TRBPs to the residue handling area, where the materials are staged for off-site release
- picks up projectile racks, unloads and deforms the treated projectiles, and loads deformed projectiles into skips for off-site transport
- returns trays and racks to the MDB
- picks up treated TRBP bins and dumps the contents across a separator screen into waste containers (drums for small pieces and dumpsters for large pieces) for off-site transport
- returns TRBP bins to the MDB

The materials handling scheme uses a series of charge cars and conveyors to move materials throughout the facility, similar to the method used in the Army's baseline incineration system. The major difference is that the bin used to charge the TRBP is the primary carrier of material. Since these bins weigh up to 20,000 pounds when loaded, the charge car and conveyor carrying capacity has been increased from the baseline design. The main links between the charge car and other system processes are the conveyors. On the upper floor of the facility, some conveyors are modified from the baseline system in length, width, or both, whereas conveyors on the lower floor are also modified to accommodate the heavier loads of the TRBP bins and projectile racks. The drained projectile bodies are placed in trays similar to the baseline system operations, and these trays are stacked two-high in racks that have a footprint similar to that of the TRBP bin. Thus, the charge car and conveyors can carry either bins or racks.

Dunnage is size-reduced to provide a denser load to the TRBPs and thereby reduce the number of TRBPs required. No pretreatment of the dunnage beyond the normal baseline-type operations is required. Dunnage is size-reduced by impact in a hammermill (rather than by cutting) to between 8 and 10 inches. Procedures are to be developed for dust control. Some metal and fiberglass rocket parts bypass COINS and go directly into TRBP bins. A TRBP bin of unshredded parts is brought to the shredder by conveyors. The bin is dumped at a slow rate into a slow-speed, high-torque, rotary-shear shredder that reduces the parts to 4 inches by 4 inches or less. The shredded parts drop into another TRBP bin.

INFORMATION USED IN DEVELOPING THE ASSESSMENT

Engineering-Design-Related Documents

On December 14, 2001, Eco Logic issued a final draft of the EDP for a full-scale pilot plant implementing its technology package at Blue Grass Army Depot (Eco Logic, 2001a).The EDP includes technical descriptions and data, drawings, a preliminary hazard analysis, and cost and schedule analyses. It has been used as the primary source of information for this assessment. Other EDP-related documents used in the assessment include the initial draft of the EDP and the study plan and test reports for the EDS tests submitted by Eco Logic (Eco Logic, 2001b, 2001e). Committee members also attended design review briefings by technology provider team members (Eco Logic, 2001c, 2001d). Assessments of the Eco Logic technology package in earlier NRC reports were also considered (NRC, 1999, 2001b).

Overview of Engineering Design Studies and Tests

Like other technology providers in the ACWA EDS program, PMACWA and Eco Logic determined what testing was required to ensure that its EDP for Blue Grass was completed to a level of detail necessary for proceeding to full-scale design and implementation.

The Demo II tests generally confirmed the performance and efficacy of the GPCR™ process for the waste streams tested. However, problems with the agent detection method hampered a full evaluation of the process, and some concerns were raised about the selection of materials of construction when processing agent material. In addition, the impact of processing explosive energetics material within the TRBP was not fully understood. El Dorado Engineering considered the reverse assembly equipment to be a proven design with the exception of modifications to the RDM by Kvaerner and an effective method for grinding M28 propellant. The EDS test program was designed to specifically address these questions. The study plans from Eco Logic and Foster Wheeler were organized to investigate the following areas:

- methods development
- explosivity testing
- metallurgy testing
- elastomers testing
- grinder tests
- transpiring-wall SCWO reactor tests

The overall testing objective included generating all required design/operating data to facilitate the development of a full-scale design, preliminary hazards analysis, and a detailed, accurate, full-scale cost estimate and schedule. The next sections present the objectives specific to each of the test programs and the key results.

Engineering Tests

Methods Development Testing

The objectives of the methods development tests were as follows (Brewer et al., 2001):

Objective 1. Develop and validate methods for sampling and analysis of agent (GB, VX, and HD) in GPCR™ product gas.

Results: The following methods were developed and validated for the measurement of agent in the GPCR™ product gas under matrix conditions established by Eco Logic:

- HD bubbler method with recovery of 97±12 percent
- GB bubbler method with recovery of 112±26 percent
- VX bubbler method with recovery of 91±38 percent

The VX bubbler method suffers from a problem that decreases recovery and contributes to a low bias. Performance is within criteria to allow use as a qualitative monitoring method by setting an action level at 0.56 time-weighted average (TWA). A 3:1 signal to noise ratio is achieved at approximately 0.5 TWA.

Objective 2. Validate the safe and effective use of MINICAMS for continuous monitoring of agent (GB, VX, and HD) in the GPCR™ product gas.

Results: Issues surrounding the safety of using miniature chemical agent monitors (MINICAMS) were addressed, and a modified method was developed for sampling a hydrogen-based atmosphere. This modification does not require the manufacture of any component and is readily implemented in the field.

Objective 3. Develop and validate methods for sampling and analysis of selected constituents from the list of Schedule 2 and breakdown products (resulting from GB, VX, and HD) in GPCR™ product gas.

Results: Most of the process-related, nonagent compounds present in the GPCR™ product gas are measured using a bubbler and standard EPA collection techniques. The process-related, nonagent compounds for which a method was not developed included vinyl sulfide, mercaptans, dimethylamine, and ethanethiol.

Objective 4. Develop the information necessary to support acceptance/approval of the validated methods.

Results: Standard operating procedures (SOPs) have been prepared for each method developed.

In addition to the results discussed above, the interferences present in the GPCR™ product gas that caused the monitoring methods to fail during the Demo II testing were identified. Interferences for both methodologies—the depot area air monitoring system (DAAMS) and MINICAMS—are as follows:

- Phosphine and hydrogen sulfide create highly acidic conditions that degrade GB on the collection media. Recovery of GB showed a steady decline as analytical holding time increased.

- The presence of naphthalene masks the HD response in the flame photometric detector used for both MINICAMS and DAAMS methods.
- Phosphine significantly reduces the lifetime and efficiency of the analytical columns that serve to identify gas components.

Explosivity Testing

The objective of the explosivity testing was to develop the data necessary to design the TRBP to process residual energetics from the munitions disassembly process (Eco Logic, 2001f). The explosivity testing was conducted at HAAP in Kingsport, Tennessee. Seven energetic materials representative of those found in the Blue Grass stockpile were selected: five explosives (RDX, tetryl, TNT, Composition B, and tetrytol) and two propellants (black powder and M28). Five combinations of energetics were also tested: tetryl-Composition B; tetrytol-black powder; black powder-M28; tetryl-RDX, and TNT-black powder. In total, 48 individual tests were conducted and the accompanying temperature and pressure profiles were recorded.

An apparatus was constructed to mimic both the geometry and heat profile of the full-scale TRBP. Each energetic material (in a series of tests with increasing quantity) was heated at either a slow or a fast heating rate that imitated the heating rates to be employed in a full-scale facility. The temperature and pressure data were plotted for the various energetic materials tested and were compared with the known properties of each energetic material. No explosive or detonation events occurred. The heating of these energetic materials in a simulated TRBP environment caused the materials to decompose in a controlled manner, and the processing of the energetic material resulted in temperature and pressure profiles within the normal design and operating range of full-scale TRBP operations.

Metallurgy Testing

The objectives of the metallurgy testing were as follows (Eco Logic, 2001g):

- Determine the appropriate materials of construction for the full-scale TRBP and reactor.
- Determine (estimate) the expected type and frequency of maintenance required for the TRBP and GPCR™ reactor.

The following are the key results from the test report:

- Recommended materials of construction are
  –for the TRBP, Hastelloy-X
  –for the GPCR™ reactor, HR-188
  –for the GPCR™ reactor radiant tubes, Kanthal APM.
- Preoxidation treatment of the metal surfaces of the TRBPs and the GPCR™ reactors is strongly recommended to significantly enhance the high-temperature corrosion resistance.
- Reoxidation of metal surfaces should be carried out in a controlled oxidizing atmosphere for a 24-hour period following every 500 hours of operation.
- The recommended materials of construction can be welded using standard methods and matched filler metal weld wire.
- Nondestructive evaluation of weld regions is recommended as periodic maintenance.

Elastomers Testing

The objectives and key results of the elastomers testing program were as follows (Eco Logic, 2001h):

Objective 1. Determine the appropriate elastomers for the full-scale system.

Results: Exposure testing and end-point measurements were interpreted to determine mass and volume changes for the product compounds Viton ETP, EPDM, and neoprene. EPDM proved to be the most suitable material for process water service, while Viton ETP was found to be the most suitable material for product gas service in the GPCR™ process environment.

Objective 2. Determine (estimate) the expected elastomer life.

Results: The two most suitable materials, EPDM and Viton ETP, showed little degradation over 500-hour exposure periods. It was concluded by the technology provider that the expected life of these materials would be very long in a GPCR™ process environment.

Propellant Grinder Tests

M28 propellant grains removed from M55 rockets must be ground to a size suitable for subsequent hydrolysis. A grinding test program was performed in part at HAAP (Eco Logic, 2001i).

The grinder used was manufactured by Franklin Miller and was modified to operate with water flooding. Although the production grinder would use stainless steel, the test grinder and cutters were fabricated from carbon steel to keep costs within budget and delivery within project schedule. The test grinder was a high-torque, low-speed, twin-shaft design, with feed opening dimensions of 24 inches by 23 inches. A 50-horsepower electric motor drove two counterrotating cutter assemblies.

The program was divided into seven test series and included the grinding of a propellant simulant (low-density polyethylene) and actual M28 propellant in both normal and upset conditions. Low-density polyethylene has mechanical properties similar to M28 propellant and was inexpensive and readily available. For two of the test series, the Army supplied M28 grain pieces of various sizes (1.7 to 5.1 inches in diameter). Approximately 177 simulant grains and 50 pieces of M28 propellant were ground up during 19 tests. Particle size distribution, throughput rate, and operational performance of the grinding machine were observed and evaluated. No mishaps or accidents occurred. Specific objectives and corresponding results were these:

Objective 1. Validate the ability of the grinding equipment to safely reduce M28 propellant grains to $^3/_4$-, $^1/_2$-, and $^1/_4$-inch pieces.

Results: The grinding equipment could grind M28 propellant grains to pass the $^1/_4$-inch screen.

Objective 2. Determine the output particle size distribution for grinder screen sizes of $^3/_4$, $^1/_2$, and $^1/_4$ inch.

Results: By mass, 99.4 percent of the M28 propellant passed through the $^1/_4$-inch screen. Owing to a limited supply of M28 propellant, particle size distribution data were collected only for the $^1/_4$-inch screen.

Objective 3. Determine the throughput of propellant for grinder screen sizes of $^3/_4$, $^1/_2$, and $^1/_4$ inch.

Results: Based on available data for grinding with the $^1/_4$-inch screen, the grinding equipment was capable of processing M28 propellant grains at a rate of 2 linear in./min.

Objective 4. Demonstrate the ability of grinding equipment to safely process foreign objects such as detached antiresonance rods and igniter cables.

Results: During tests of the grinder, Eco Logic fed simulated antiresonance rods and igniter cables that would be intrinsic to the M28 propellant grains. The grinding equipment was able to safely process these objects. Propellant ignition, as well as other incidents, blockages, or equipment problems, did not occur during these tests (Eco Logic, 2001i). All of the igniter cables were significantly size-reduced and passed through the $^1/_4$-inch mesh exit screen of the grinder with the propellant. Three of the four antiresonance rods passed through the screen without being size-reduced. The fourth rod was size-reduced and passed through the screen. The three antiresonance rods that passed through the grinder without being size-reduced were fed vertically, whereas the fourth rod, which was size-reduced, was fed horizontally.

Objective 5. Determine optimum operating parameters (i.e., water flow) and equipment sizes (i.e., pump and motor sizes).

Results: As a result of the tests, a larger, more powerful grinder was deemed necessary for full-scale production. The test grinder was unable to grind the propellant simulant when it was fed in a horizontal position. Also, the orientation of the grinder teeth pushed the material being ground to one end of the feed hopper. Thus, the testing program was only a partial success. Additional testing with a larger, modified grinder is necessary to determine the effect of design modifications and to select grinder and motor sizes for the full-scale plant.

Transpiring-Wall SCWO Reactor Tests

The purpose of the SCWO reactor tests was to demonstrate that a transpiring-wall SCWO reactor can be operated effectively and reliably under conditions that mimic those planned for the full-scale operation at Blue

Grass (Eco Logic, 2001c). A specific goal was to demonstrate 80 percent availability for the reactor.

At the time this report was prepared, a 6-inch-diameter reactor was in operation at Dugway Proving Ground in Utah. Several short systemization/workup runs of 10- to 72-hour duration were carried out using a feed stream containing energetics and GB hydrolysates. A 500-hour run with GB hydrolysate was completed, and a draft report was issued (Foster Wheeler, 2001). To test the applicability of a process developed by General Atomics for removal of aluminum from hydrolysate, the concentration of aluminum used in the test was adjusted to simulate that expected from the process. The adjustment was done partway through the testing and had no apparent adverse impact. However, several operating problems were reported, which may be enumerated as follows:

1. The oxygen/nitrogen system caused major problems during early phases of the testing. The key problems were related to insufficient and unsteady supply of nitrogen, insufficient capacity of oxygen, and the want of a spare oxygen pump. Once the oxygen/nitrogen supplier corrected these problems, the oxygen/nitrogen system supported test operations without a single incident. Based on experience during Demo II and EDS II tests with GB hydrolysate, Foster Wheeler believes that oxygen (pure or in some ratio with nitrogen) is the optimum oxidant.
2. During early tests, the technology provider found it difficult to initiate the SCWO oxidation reaction from cold conditions with propylene glycol as the initiating fuel. This was attributed to the lower reactivity of propylene glycol compared with kerosene. Thereafter, kerosene replaced propylene glycol as the initiating fuel.
3. A few incidents of loss of reaction were experienced during workup runs at the 500 lb/hr feed rate. Foster Wheeler believes the loss-of-reaction events were caused by the less-than-optimum reactivity of propylene glycol to support such high feed rates. This problem was resolved for the actual EDS II testing (validation runs) by continuing the flows of the kerosene, which replaced propylene glycol as the initiating fuel, and of the start-up hot water during operation.
4. While diagnosing the oxidation instability resulting from the unsteady flow of nitrogen during the early systemization of the system, the pressure relief valve was actuated several times. These events resulted in periods of downtime because spare pressure relief valves were not readily available.
5. There was one instance of a pipe leaking near the pressure control valve. This was readily fixed but resulted in one day of downtime. The leak was attributed to having retrofitted an alternative pipe routing without a thermal expansion analysis.
6. A loss of the deionized water supply occurred during a workup test. Increased water usage for feed mixing, coupled with reduced membrane capacity while operating with colder water during the winter months, caused the deionized water supply to drop.
7. Breaker overload of the instrument air compressor and freezing of the instrument air-line occurred. On separate occasions, the instrument air compressor malfunctioned, causing an electrical circuit overload and a loss of air due to moisture.
8. The most significant issue during the tests was the occurrence of periodic spikes in the concentrations of hydrocarbons and CO in the gaseous effluent from low steady-state values. These spikes lasted about 1 minute each. Foster Wheeler has offered some possible explanations, but further testing will be needed to identify the cause and provide a remedy for the problem.
9. Upon shutdown, several bumplike deformations and pinhole-size leaks were observed in the upper region of the upper liner. Foster Wheeler believes these deformations were caused by the oxidation instability previously noted. The varying oxidation conditions below the injector result in changing heat flux to the neighboring liner, which in turn leads to thermal stresses and deformations.
10. General corrosion in the top 5-inch region of the upper liner after the 500-hour test was also observed. This relatively uniform corrosion is estimated to penetrate the 0.15-inch-thick liner to about 0.008 inch. Had it not been for the pinhole leaks, the liner would have had significant remaining service life. However, Foster Wheeler concluded that the test did indicate the upper

liner has at least a 500-hour life when processing GB hydrolysate.

Two additional 500-hour runs are planned, one with VX hydrolysate and one with HD hydrolysate. Both tests will also contain energetics hydrolysate in the feed stream.

ASSESSMENT OF PROCESS COMPONENT DESIGN

Disassembly of Munitions and Removal of Agent and Energetics (Area 100)

The Area 100 components determine the throughput rate for the remainder of the facility. The reverse assembly process is primarily based on baseline system equipment with some modifications. The PMD, the MDM, and the AQS are essentially baseline system equipment. Because the GB and VX projectiles are not explosively configured, not all of the designated stations of the PMD will be used in the disassembly of these munitions. Only the mustard-containing munitions have explosive bursters. The unit operations that represent significant changes to the baseline system within the reverse assembly area are the RDM, with its modifications from the baseline configuration, and three new operations: the PWS, the COINS, and the propellant grinder.

Projectile Washout System

The current PWS design is a new addition to the final EDP, and further design options are still being considered (Eco Logic, 2001a). The Army requested that this be included to remove agent heels that may be present in the projectiles. A PWS was demonstrated in the EDS program by Parsons. The planned PWS for the Eco Logic process would rely on these testing results and may be identical to the system that has been tested by Parsons. That system performed well in preliminary testing to date (NRC, 2001c).

Eco Logic is considering other approaches to address the removal of agent heels from projectiles. Heels may not be present in every projectile. One potential alternative is the use of systems such as portable x-ray analyzers to nondestructively characterize projectiles that have heels for segregation in the storage facility. This approach could minimize unnecessary processing in the PWS of projectiles not having heels.

Rocket Dismantling Machine

Although similar to existing baseline system technology, the RDM has been modified considerably. The material flow through the RDM has been identified conceptually, but successful engineering and operation of the potentially complex RDM have not been demonstrated. In particular, a potential for problems exists in achieving effective cutting and positioning of the rocket for repeatable operations. Separation of the propellant from the rocket motor case may also be problematic. That is, once the case is cut, the propellant grain is pushed out of the case to separate the energetic materials from the inert materials. If the cut is not clean (e.g., if there are burrs, rolled edges, or other deformations), this mechanical separation may not work.

The Propellant Grinder

The propellant grinder is designed to keep the propellant submerged and flooded with water during the grinding operation to avoid accidental ignition and propagation. A grinder was successfully tested with polyethylene simulant and actual propellant pieces during EDS testing. The testing demonstrated that a low-speed, high-torque grinding operation could be done safely under water with little chance of igniting the propellant by interaction with the grinder teeth or any residual metal impurities (antiresonance rods and cables). Although no tests were done with complete 31-inch propellant grains, the testing does indicate that the process is understood well enough for inclusion in the next phase of the engineering design of the facility.

The COINS Operation

The COINS process is designed to separate the energetics in burster assemblies from the metal hardware. The current conceptual design is an improvement over the earlier design, in which the transfer mechanism conveying the bursters was in the caustic bath. The current design will expose an overhead transfer mechanism to less caustic (only through vapors and splashes) but will still operate in a harsh environment.

The steel burster tubes from projectiles are unaffected by the caustic in the COINS. To enhance reaction rates, burster tubes are sheared into three pieces by the burster size-reduction machine to provide more surface area for the reaction of tetryl with caustic. The burster sections are then loaded in the COINS baskets, and the burster explosive is separated in the hot caustic bath within the COINS process.

The fuze assemblies and bursters from rockets are sent to the COINS process after processing in the RDM. Although the COINS process is not designed to complete the hydrolysis of the energetic materials, it is designed to reduce the explosive hazard associated with the processing of bursters. A sump pump transfers the resulting energetics slurry from the COINS to the EPDR for complete hydrolysis. The sump pump is designed to also transport any solids that may accumulate at the bottom of the COINS trough. The metal parts remaining in the COINS baskets are pulled from the caustic bath, washed, and loaded into large bins for thermal treatment in the TRBP. Some energetic material is expected to remain on the metal parts.

M36 and M34 bursters from rockets are placed in COINS without shearing. These bursters require less exposure time for melting and reaction of the Composition B explosive than the tetrytol in projectile bursters. The energetic material in these rocket bursters is fully extracted from the burster metal and plastic hardware during the COINS processing.

The M417 fuze assemblies from rockets are transferred to the COINS process from the RDM. The aluminum rocket body and the zinc body of the RDX booster will dissolve in the COINS, allowing access to and reaction of the RDX booster and lead charges. The stab detonator and lead azide are expected to remain intact through the COINS process. These materials will be deactivated in the TRBP process. Explosivity tests were performed during the EDS II program to assess the impact of small charges being present in the TRBP during the thermal treatment of the solid waste (Eco Logic, 2001f). These tests measure the margin of safety. Pressure and temperature excursions were measured in experiments with 56 pounds of energetic material in a full-scale TRBP. The results indicate no significant design or safety issues.

Hydrolysis of Agent and Energetics (Area 200)

The Army has established the efficacy of agent hydrolysis at both laboratory and pilot scales, and the Army's program was reviewed by the ACW I Committee (NRC, 1999). That committee found that hydrolysis could decompose the agent with destruction efficiencies of 99.9999 percent, and the ACW II Committee has no reason to modify the previous finding. Eco Logic's agent hydrolysis process is a straightforward application of the Army's data, and the committee has no special concern about its efficacy.

Significant hydrolysis will take place in the COINS bath and in the propellant collection reactor (PCR) in advance of transfer to the EPDR. The batch processing time in the schedule is sufficient to accomplish complete hydrolysis within the EPDR without including the hydrolysis that takes place in the COINS and PCR. The PMACWA-sponsored EDS test program for hydrolysis of energetics demonstrated that the control of foaming, offgas emissions, temperature, and rate of heat release, as well as the materials handling for hydrolysis reactors on the scale of the EPDR, can be accomplished safely and effectively (see Chapter 2). No explosion hazard is anticipated to exist following the treatment in the EPDR. As discussed in Chapter 2, coprocessing of tetrytol and M28 propellant should be investigated further because of indications that picric acid and, possibly, lead picrate form during the hydrolysis reaction.

Supercritical Water Oxidation (Area 300)

The design and operation of the transpiring-wall SCWO reactor developed by Foster Wheeler were covered extensively in previous reports (NRC, 1999, 2001b). Of primary concern for this report is the performance of the SCWO system in the tests that were ongoing at the time this report was being prepared. As previously reported, several problems were encountered during the 500-hour GB campaign completed just before the data deadline for this report (Foster Wheeler, 2001). A review of the more significant challenges and operating problems experienced during testing, and their impact on the committee's assessment of the Eco Logic process, follows:

1. The problems initially encountered with the oxygen/nitrogen system were eventually corrected, and the system subsequently supported test operations without a single incident. Assuming the instability problems (see below) encountered when operating on oxygen can be resolved, oxygen generated from liquid oxygen appears to be a suitable oxidant.
2. The difficulty in initiating oxidation using propylene glycol as the initiating fuel was attributed to the lower reactivity of propylene glycol in com-

parison to kerosene. The committee believes that substituting kerosene for propylene glycol as the initiating fuel was a logical step to take.

3. The technology provider resolved the loss of reaction caused by the less-than-optimum reactivity of propylene glycol by continuing the flows of the initiating fuel (kerosene) and start-up hot water during EDS II test operations. The committee believes that the ratios of the various feeds may still not be correct, and that further adjustment may be necessary to resolve this instability problem.
4. There were a few incidents of pressure relief valve actuation, especially during troubleshooting of the instability problem. The committee believes that resolution of the instability problem will alleviate this problem.
5. The most significant issue, the occurrence of periodic spikes in hydrocarbon and CO measurements in the gaseous effluent, will require further testing to identify the cause and provide a remedy for the problem (see Figure 4-3). The committee considers this problem to be very serious. Resolution must be obtained before the transpiring-wall SCWO process can be seriously considered for full-scale application. The technology provider believes that salt formation and periodic sloughing off of this salt from the downstream piping is a possible cause of or contributor to the instability problem. This may or may not be the case. If the technology provider is correct, then salt formation is another issue to be addressed.
6. The several bumplike deformations and pinhole-size leaks observed in the upper region of the upper liner were attributed to the previously mentioned oxygen instability problem. The committee sees this discovery of deformations and tiny leaks as an issue, but not one so serious as to preclude implementation of the Foster Wheeler SCWO in a full-scale plant.
7. The committee concurs with Foster Wheeler's conclusion that in spite of general corrosion in the top 5 inches of the upper liner after the 500-hour test, the upper liner would have a life of at least 500 hours when processing GB hydrolysate.
8. Eco Logic must remove aluminum from the feed to the SCWO and intends to use a process based on General Atomics' aluminum removal technol-

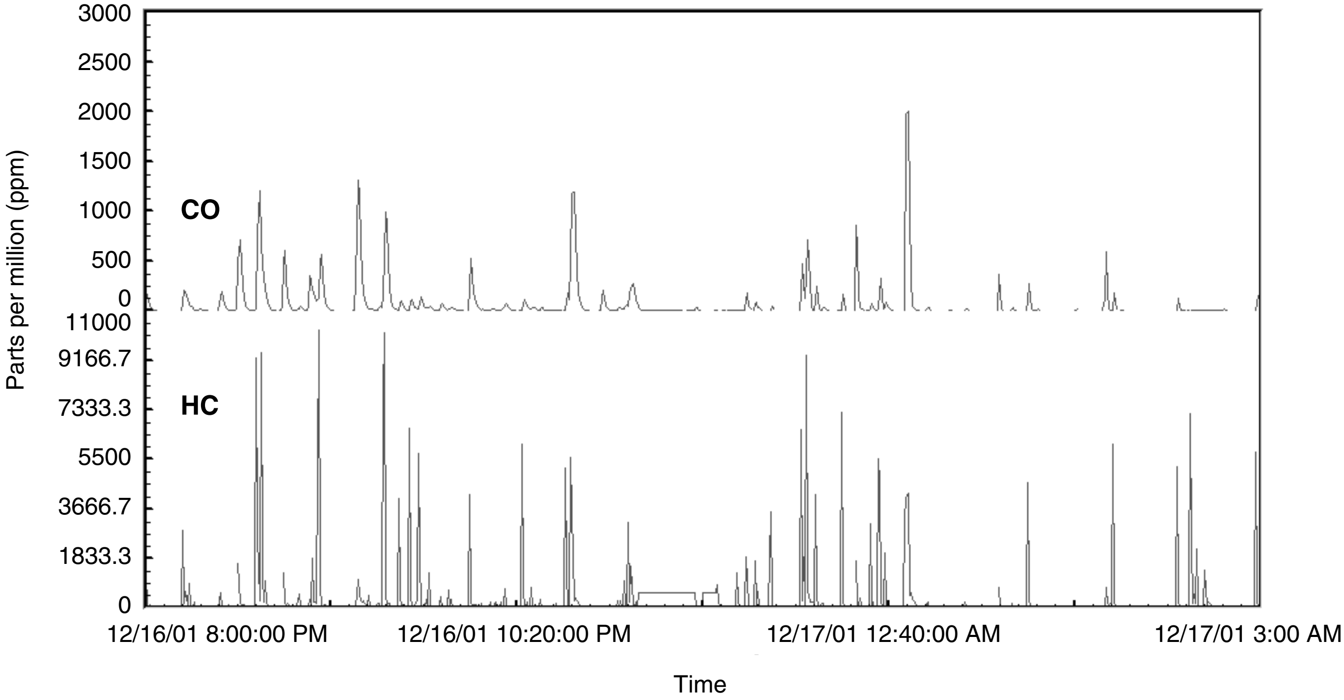

FIGURE 4-3 CO and HC spiking profiles during transpiring-wall SCWO operation. SOURCE: Crooker and Ahluwalia (2002).

ogy. However, Eco Logic does not have rights to use that technology, so it must develop and demonstrate its own technology.[3]

Overall, it is this committee's opinion that problems remain with Foster Wheeler's current transpiring wall SCWO system that may prevent the application of this SCWO technology in a full-scale facility.

Gas-Phase Chemical Reduction (Area 400)

Two changes to the design of the GPCR™ system were made by Eco Logic during the preparation of the EDP (Eco Logic, 2001a). First, a gas scrubber system step has been added to remove the toxic phosphine gas that would be formed during the GB disposal campaign. Second, hydrogen sulfide that would be formed during the mustard agent and VX disposal campaigns will be removed. This will require one or both of the following modifications: (1) minor enhancements to the projectile disassembly trains in Area 100 to maximize the removal of mustard agent from the projectiles and (2) optimizing conditions for removal or adsorption/absorption of hydrogen sulfide in the existing Area 400 scrubber. Eco Logic is considering the addition of another packed-bed scrubber operating with a caustic/potassium permanganate scrubbing solution to remove additional hydrogen sulfide and phosphine (Eco Logic, 2001a).

The GPCR™ system was tested in the Demo II program (NRC, 2001b). Key results were as follows:

- The GPCR™ process was able to achieve 5X decontamination of carbon, PCP-spiked wood, DPE suit material, firing tubes, and HD mortar casings. However, the first run with DPE suit material was stopped prematurely because the reactor input line plugged.
- Reactor product gases were effectively treated in the two-stage scrubber system and product gas burner. However, Eco Logic concluded that process modifications might be necessary to control hydrogen sulfide and phosphine in the offgas from the reactor. Also, the liner in the product gas burner showed evidence of corrosion.
- The gas, liquid, and solid process streams from the GPCR™ process were analyzed for specified chemical constituents and physical properties, as well as for the presence of agent, CWC Schedule 2 compounds, and other hazardous, toxic compounds. For the tests that included chemical agents, the scrubber solution and scrubber filters were completely characterized. However, the gas sampling streams for the runs with agents could not be analyzed, and DREs could not be established for VX, GB, and mustard agent. Earlier tests of the GPCR™ system conducted at Aberdeen Proving Ground, Maryland, in 1996 were able to destroy VX to a DRE of 99.999999 percent (NRC, 1999).

These results led to the analytical methods development program conducted during the ACWA EDS II program and discussed earlier in the chapter. The key analytical problems cited above that were encountered during the Demo II program now appear to have been adequately addressed and, for the most part, resolved (Brewer et al., 2001). MINICAMS can now be used on the product gas stream, although it would be preferable if these instruments could handle higher concentrations of hydrogen sulfide and phosphine. A bubbler system can be used in place of the DAAMS tubes. However, continued refinement and verification studies are recommended.

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

Eco Logic analyzed stack gas from combustion of the GPCR™ product gas in the product gas burner. The stack gas was found to meet the boiler and industrial furnace (BIF) emission standards (40 CFR 266.1). However, the EPA recently promulgated new maximum achievable control technology (MACT) standards for hazardous waste combustion that are more stringent than BIF standards. Eco Logic must determine whether the emissions from the product gas burner can meet the MACT standards, not just BIF rules. The GPCR™ product 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 would therefore be regulated under RCRA.

Dioxins were detected in the GPCR™ product gas in tests with pentachlorophenol (PCP)-spiked wood,

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[3] Douglas J. Gray, Vice President, Engineering, Eco Logic International, personal communication to Darren Dalton, PMACWA, February 4, 2002.

DPE suits, and butyl rubber (NRC, 2001b). 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 test with neat GB, the product gas contained 0.01 to 0.06 percent phosphine. As noted previously in the section on methods development testing, phosphine can interfere with the measurement of GB. Based on results from the EPA's toxicity characteristic leachate procedure, stabilization would be necessary only for solid wastes derived from DPE suit material, because the cadmium and lead criteria were not met by the treated dunnage in some tests (NRC, 2001b).

The operation of the smoking rooms needs to be addressed further. It is not clear that the misting process employed in the rooms and the associated carbon filters will remove the smoke generated from the contents removed from the TRBPs. If the smoke is not adequately managed, a visible plume will be emitted from this part of the facility. Because the oxygen supply is limited, carbon monoxide formation can be expected, but this appears to not have been addressed in the design.

Utilities (Area 500)

All Area 500 equipment is off-the-shelf units and storage tanks. There should be no problem providing this equipment.

Materials Handling (Area 600)

The materials-handling scheme uses a series of charge cars and conveyors similar to the baseline system to move materials throughout the facility. However, the loaded TRBP bin can weigh up to 20,000 pounds, and the charge car and conveyor carrying capacity must be increased relative to that of the trays used in the baseline system design. Conveyors on the upper floor are modified from baseline system specifications only in length and/or width, whereas conveyors on the lower floor are modified to accommodate the heavy TRBP bins. The drained projectile bodies are placed in trays similar to those in the baseline system. These changes from baseline should not unduly affect the performance or safety of materials handling.

The shredders should not be difficult to procure and operate since the end product is not severely reduced in size (8 to 10 inches for dunnage and 4 inches for metal and fiberglass).

ASSESSMENT OF INTEGRATION ISSUES

Component Integration

Eco Logic's schedule shows destruction of the Blue Grass stockpile within the CWC treaty extension deadline of April 27, 2012 (Eco Logic, 2001a). Meeting this deadline means that the overall process must achieve the required throughput levels and process availability. This, in turn, demands that the throughput and availability of each process step, in combination with equipment redundancy and sufficient buffer storage capacity between the individual process steps, maintain the specified destruction rate. Integrating the individual processing steps requires effective process monitoring and control to ensure that appropriate materials are fed to each step and that all materials discharged from the plant meet safety and environmental specifications. In addition, attainment of the required process availability depends on the durability of all materials of construction, and on the effectiveness of the plant operating and maintenance force.

The Eco Logic technology package is a combination of continuous and batch processing steps with buffering between some of the steps. Eco Logic has designed the process and specified the size and number of the components to be able to process the entire Blue Grass stockpile in 91 weeks, operating the plant 24 hours per day, 7 days per week, with an overall plant availability of 75 percent (Eco Logic, 2001a). Eco Logic assumes a systemization period for the facility of 15 months. Munitions containing different agents would be handled in separate campaigns.

Process Operability

There are areas of the Eco Logic technology package where important process operability questions exist. The first is Area 300, where the operability of the Foster Wheeler transpiring-wall SCWO reactor remains a significant and incompletely resolved problem. The transpiring-wall design is meant to minimize both corrosion and salt deposition, which would plug the reactor. The most recent test results, summarized above, show that effective and reliable operation of the SCWO reactor was not demonstrated, and additional development work on this operation is needed.

Maintenance Issues

The reliability and long-term operability at stable operating conditions of the current transpiring-wall SCWO reactor by Foster Wheeler remain unproven. The EDS II test results from the processing of GB hydrolysate showed that the reactor could be kept online for tens to hundreds of hours without shutdown in the later phase of the testing. However, this test was not performed under stable operating conditions, and at stable operating conditions, the tests might have been more or less favorable. If problems of corrosion and salt deposition occur, as they did with other SCWO reactors, or if liner cracks develop, as occurred during Demo II testing, significant maintenance and downtime would be required. In particular, the time required for disassembly, reassembly, and pressure sealing, followed by pressure testing of a high-pressure SCWO reactor, could be considerable. Moreover, fabrication of a new transpiring-wall liner is expensive.

New TRBP doors and door mechanisms, designed by El Dorado Engineering, are planned (Eco Logic, 2001c). These doors must withstand high temperatures and temperature cycling (as the TRBP cycle time is 42 hours or less) while maintaining a tight seal to prevent hydrogen leakage and, perhaps, agent leakage, in a closed environment. Since this is a newly designed system, the maintenance requirements have not yet been determined.

The Area 200 hydrolysis system for metal parts treatment involves moving parts-containing baskets through a corrosive environment consisting of hot (90°C) NaOH solution. Although the drive system for these baskets is now designed to be outside the bath, the committee nonetheless believes this still to be high-maintenance equipment. No data are available to assess the maintenance that will be required.

The maintenance for other parts of the process appears to be similar to that for other complex chemical plants.

Monitoring and Control Strategy

Areas 100 through 500 will be controlled with independent systems. The technology provider intends to pursue the selection of a common hardware standard for each area to facilitate integration and reduce overall system requirements for spare components. Area 600 will be controlled remotely by an operator using video and a remote conveyor actuator. The independent control systems are connected by a common bus, which communicates critical parameters between areas to control material flow and to provide data to the safety system.

The independent control for Area 100 is based on the reverse assembly machines of the baseline incineration system, which use an Allen Bradley PLC platform with direct operator control of the munitions loading, with solids output to Area 400 and liquid output to Area 200. Eco Logic plans to use equivalent Siemens PLCs.

Area 200 control uses a batch controller. Each reactor is monitored and controlled separately. Level sensors and control valves meter the agent, energetics, or propellant along with sodium hydroxide into the empty reactor vessel. Fluid density is used to monitor and control the caustic concentration in the reactor vessel.

Area 300 is controlled using a distributed control system (DCS). The DCS monitors and controls all aspects of the SCWO process, including the ignition system, the reactor pressure, the pressure drop across the transpiring wall, the reactor axial temperature profile, the effluent system, and the evaporation/crystallization system. Each of these control functions is accomplished using a network of pressure, flow, temperature, and analytical sensors linked to control valves through DCS control loops. The measurements of reactor pressure and the pressure differential across the reactor liner are especially important since they determine when shutdowns are needed. Reactor pressure and temperature measurements are important because they can indicate unstable operation that causes incomplete reaction.

A DCS is also used to control Area 400. The DCS monitors and controls the cycling of the TRBPs, the preheaters, the GPCR™ reactors, the scrubbers, and the product gas handling system. Each of these subsystems is controlled with a series of control loops tied to the individual control functions through input devices linked to output devices via control algorithms within the DCS.

Area 600 also uses many baseline system machines that are controlled with a DCS.

Each of the independent controllers is linked to a human-machine interface (HMI) network. Dedicated HMI operator stations are assigned to each area, with an appropriate number of redundant stations providing backup in case of maintenance or breakdown. A development station permits tooling changes between campaigns to be carried out at the same time as systemization is carried out on the individual areas. A redundant set of historical data collection stations ensures that all of the data are collected and stored for both regulatory and process optimization purposes. A supervisory station permits overall monitoring of the process status without disturbing the individual area process control operators.

Thus, the systems used to monitor and control the process are conventional. Process materials (with the exception of agents and energetics), temperatures, and pressures used in this technology package are commonly used in other industrial applications, where they are routinely monitored and controlled. The usual collection of equipment for monitoring temperature, pressure, level control, flow, and other parameters normally measured in a chemical plant is used. The analytical procedures to be used to monitor certain streams will be new, and they present the greatest uncertainty.

Process Safety

In its original report, the ACW I Committee observed the following about the Eco Logic technology package (at the time known as the Lockheed Martin Integrated Demilitarization System) (NRC, 1999):

- The TRBP and GPCR™ reactors are high-temperature, low-pressure hydrogen processes, and leakage of hydrogen from them is a safety concern.
- The most significant worker safety issue will probably be maintenance of the conveyor system through the caustic bath of the COINS.

The three safety sections in the 1999 ACW I Committee report are updated in the following three sections (NRC, 1999).

A preliminary hazards analysis (PHA) was prepared as a part of the EDS design package in accordance with MIL-STD-882D (DoD, 2000) and OSHA 29 CFR 1910.119 (Eco Logic, 2001a). The PHA was prepared in conjunction with the engineers responsible for the EDS design package and was used by the Eco Logic engineers to identify several design measures for reducing risk. The committee believes that design solutions should be used whenever possible, particularly early in the design process, and that administrative solutions (e.g., training and procedures) for reducing risks should be minimized in favor of engineering design changes. The Eco Logic philosophy appears to be consistent with that of the committee. The PHA appears to have been conducted in a satisfactory manner, although it will have to be updated as the design progresses.

Worker Health and Safety

In the earlier ACW I Committee's report on the Eco Logic design, the most significant worker safety issue was expected to be maintenance of the COINS conveyor system by personnel in DPE suits (NRC, 1999). The conveyor system was to operate immersed in a hot caustic solution. The ACW II Committee's Demo II report noted the COINS process had been changed to provide for an overhead chain conveyor system to move the immersed baskets in the caustic bath (NRC, 2001b). The committee remains concerned that the COINS will require significant maintenance by personnel in DPE suits and finds that efforts to minimize maintenance should continue.

The ACW I Committee was also concerned that the GPCR™ reactors operate in a hydrogen atmosphere and generate methane and other gaseous hydrocarbons that could burn or explode in the presence of air (NRC, 1999). The ACW II Committee's Demo II report noted that, based on the Demo II test results, the designs and procedures for safe operation of the high-temperature TRBP and GPCR™ reactors could be prepared in the relatively short time allowed for construction of the equipment, systemization, and training of Army operators (NRC, 2001b). However, the report called for a preliminary hazards analysis to seriously evaluate the use of hydrogen in an enclosed area. PHA analyses and the design to date appear to adequately address not only the leakage of hydrogen from the TRBPs and GPCR™ reactors but also the inadvertent upstream introduction of oxygen into the feed for the TRBPs and GPCR™ reactors. Early in the ACWA process, Eco Logic commissioned a safety study on this issue (Prugh, 1998). Many of the recommended safety measures were either already in use by Eco Logic or have since been implemented. The remaining recommendations are expected to be implemented in a more detailed design that goes beyond that in the EDP. The use of high-temperature hydrogen within a building will remain a concern for the committee until a detailed quantitative risk assessment (QRA) shows the final design to be of very low risk.

Two changes in the SCWO system have had a significant impact on safety. The switch from kerosene to a mixture of kerosene and propylene glycol as a feed additive should reduce operational risks, while the change from air to oxygen will introduce a new hazard. Although the committee knows of no previous accidents that would raise safety questions unique to the transpiring-wall SCWO reactor, it has expressed general concern about the use of oxygen in SCWO reactors (NRC, 2001b). PHA analyses and the design to date appear to adequately address the issue at this stage of design. The use of oxygen, particularly pressurized oxygen, while safely done elsewhere in the chemical industry, will remain an area of concern for the committee until a detailed QRA shows the final design to be of very low risk.

Eco Logic proposes to grind M28 propellant in a water environment. The ACW I Committee had expressed the following 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.

EDS II tests showed that grinding under water would prevent propellant ignition even in the presence of metal parts (Eco Logic, 2001i).

The committee expects that energetics from fuzes and burster assemblies will be present in the feed to the TRBP under normal operating conditions. EDS II testing showed that decomposition of these energetics will occur in a controlled manner and that explosive or detonation events are very unlikely (Eco Logic, 2001f).

As the design becomes more detailed, the PHAs will have to emphasize the safety of maintenance workers. The design should be configured to minimize the number of maintenance activities in contaminated areas to reduce worker risk and to ensure that workers in DPE suits can access the areas easily and safely. This precaution could also reduce the waste streams arising from DPE suits and decontamination solution used during maintenance activities.

Public Safety

Accidental releases of agent or other regulated substances to the atmosphere or the groundwater are unlikely. The primary cause of a significant release of material containing agent or other regulated substances would be a disruptive explosion. The disassembly step provides for early separation of energetics and agent. Subsequent destruction of agent and energetics is performed in low-temperature, low-pressure processes, thereby avoiding significant stored energy and reducing process hazards. Secondary treatment of agent-free streams does involve high-temperature and, in the case of SCWO, high-pressure processes. The likelihood of a significant explosion is expected to be very small for the full-scale facility, whose design process is understood to include a QRA. A PHA conducted by the technology provider revealed no events with unacceptable or undesirable public safety risks.

Human Health and the Environment

The impact of normal operation of the Eco Logic technology package on human health and the environment was addressed in the ACW I Committee report (NRC, 1999). That report's conclusion—that the health and environmental impacts are minimal—and the supporting information it contains are unchanged.

Effluent Characterization

The committee's report on Demo II testing of the Eco Logic technology package found as follows (NRC, 2001b):

> *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 offgas.
>
> *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.

EDS testing has resulted in adequate resolution of gaseous waste stream characterization issues, as discussed earlier in this chapter. The concentrated brine product from the evaporator blowdown stream has not been characterized, but this is planned. Solid wastes have been shown to be adequately decontaminated to a 5X condition.

Effluent Management Strategy

The proposed strategy is discussed in earlier sections of this report and in NRC (1999). The strategy appears reasonable and is expected to protect public health and the environment.

Off-site Disposal Options

Dunnage. Experience at the baseline system facilities at Johnston Atoll and Tooele, Utah, has shown that only a tiny fraction of dunnage is contaminated with agent. Uncontaminated dunnage from these two stockpile locations is being disposed of off-site by commercial waste management facilities (McCloskey, 1999; U.S. Army, 1998). Off-site management of uncontaminated dunnage is also planned for both the Newport and Aberdeen sites. Off-site management of dunnage from Blue Grass would greatly reduce the on-site processing requirements and greatly simplify process integration by eliminating the need for size reduction and GPCR™ treatment of the dunnage. Off-site disposal of other agent-free waste streams, such as hydrolysates, may be possible, thereby reducing the number or size of treatment steps.

Brines. Brines produced from air pollution control processes at the baseline facility at Tooele, Utah, are currently being shipped off-site for disposal by commercial waste management facilities.

Environmental Compliance and Monitoring

The committee's earlier report on demonstration testing found as follows (NRC, 2001b):

> The product gas from the GPCR™ process does not meet the EPA syngas requirements because of high benzene and polyaromatic hydrocarbon content.

The finding remains valid, as discussed earlier in this chapter. The permitting of the process is expected to require special efforts with the EPA to establish the appropriate basis for the use of the product gas.

ASSESSMENT OF OVERARCHING TECHNICAL ISSUES

Steps Required Before Implementation

The most significant need is to demonstrate that the SCWO reactor is ready to be commercialized. Effective and reliable operation over an extended period of time remains to be demonstrated. Additional energetics grinding tests with a more powerful, modified grinder are also needed. These are described in the section "Propellant Grinder Tests" under Objective 5.

FINDINGS AND RECOMMENDATIONS

Findings

Finding (Blue Grass) EFKE-1. The occurrence of frequent spikes in hydrocarbon and CO emissions in the gaseous effluent from the transpiring-wall SCWO reactor is a serious problem that must be resolved before the Eco Logic technology can be implemented.

Finding (Blue Grass) EFKE-2. Energetics from fuzes and burster assemblies will probably not be destroyed in COINS, and downstream equipment must be able to handle this situation. Explosivity tests conducted by the technology provider showed that these materials decompose in the TRBP environment rather than explode or detonate and that the TRBPs will accommodate the decomposition of these explosive materials.

No explosive or detonation events occurred during explosivity testing (Eco Logic, 2001f). The heating of energetic materials in an environment that simulated the Eco Logic process caused the materials to decompose in a controlled manner, with temperature and pressure profiles within the normal design and operating range of full-scale TRBP operations.

Finding (Blue Grass) EFKE-3. The grinding test achieved most of its objectives, particularly those related to safety. Upon completion of the grinding test, questions remained about the design of the feed system and the size of the grinder and motor needed for the full-scale system.

A grinding test program was performed using a grinder modified to operate with water flooding (Eco Logic, 2000i). The program included grinding of low-density polyethylene as a propellant simulant and the grinding of actual M28 propellant. Particle size distribution, throughput estimates, and operational performance of the grinding machine were observed and evaluated. No mishaps or accidents occurred. It was determined that the equipment could grind M28 propellant grains to pieces with diameters not exceeding $^1/_4$ inch. Simulated antiresonance rods and igniter cables were included in the grinding operations. Three of the four antiresonance rods were fed vertically and passed through the grinder without being size-reduced. Because the grinder used in the test was unable to grind the propellant simulant when it was fed in a horizontal position, the testing program was only a partial success.

Finding (Blue Grass) EFKE-4. The process for energetics hydrolysis proposed by Eco Logic is sufficient to destroy energetic materials to an extent that makes the explosion hazards manageable.

The energetics from the RDM and from the propellant grinder operation, as well as those exposed during the COINS operation, are completely hydrolyzed in Area 200 within the PCR and the EPDR. Significant hydrolysis will take place in the COINS bath and the PCR before the energetics are added to the EPDR. The process schedules are sufficient to allow for the complete hydrolysis within the EPDR without considering any hydrolysis that occurred in the COINS and PCR. Any unexposed energetic remaining in the fuzes and bursters following the COINS operation will be destroyed in the TRBP, which can withstand the explosion of the small amounts of energetic materials that remain (see Finding EFKE-2).

Finding (Blue Grass) EFKE-5. To prevent scale buildup in the SCWO reactor due to the presence of aluminum in the hydrolysate feed, Eco Logic must remove the aluminum from the feed stream to the SCWO reactor.

Eco Logic intends to use a process that is based on the aluminum removal technology of General Atomics. However, Eco Logic does not have rights to the General Atomics process and must obtain or develop such a technology. A 500-hour run with GB hydrolysate was completed, and a draft report was issued (Foster Wheeler, 2001). To simulate the use of the General Atomics process to remove aluminum from the hydrolysate, the concentration of aluminum was increased from zero to that expected from this process. This was done part way through the campaign and had no apparent adverse impact.

Finding (Blue Grass) EFKE-6. The RDM, while grounded in baseline system technology, has been modified considerably, and its engineering and operation have not been demonstrated. In particular, there is a potential for problems with the effective positioning and cutting of the rocket. The separation of propellant from the rocket motor case may also be problematic if the cut results in burrs, rolled edges, or other deformations.

Finding (Blue Grass) EFKE-7. The abatement of smoke from the TRBP smoking rooms and the formation and management of carbon monoxide do not appear to have been adequately addressed.

No information was available to the committee on the quantity and composition of the offgases from the processed TRBP bins when they are in the smoking rooms. It is not clear that the design will provide adequate abatement for the smoke, and no provision appears to have been made for abatement of the carbon monoxide. The misting process employed in these rooms and the associated carbon filters may not remove the smoke generated from the contents removed from the TRBPs. If the smoke is not adequately managed, a visible plume whose composition is unknown but that could contain hazardous air pollutants will be emitted from this part of the plant.

Finding (Blue Grass) EFKE-8. The Eco Logic process for handling energetics will cause lead-containing M28 propellant to mix with tetrytol when rockets with tetrytol boosters are treated. The combination might lead to the formation of lead picrate.

Recommendations

Recommendation (Blue Grass) EFKE-1. Eco Logic should conduct additional grinding tests for the final design of the feed system to specify the size of the grinder and motor for the full-scale plant.

Recommendation (Blue Grass) EFKE-2. The PCR stream should not be mixed with the COINS effluent until lead picrate formation can be ruled out.

Recommendation (Blue Grass) EFKE-3. The RDM is on the critical path for the schedule of the facility, so its further development should be undertaken as soon as practical to minimize risk to the overall facility design, process integration, and operations schedule.

Recommendation (Blue Grass) EFKE-4. The gaseous effluent from the smoking rooms must be characterized and appropriately controlled.

5

General Atomics Technology Package

DESCRIPTION OF THE PROCESS

The General Atomics Total Solution (GATS) technology package proposed for the Blue Grass Army Depot is based on and is very similar to the design General Atomics originally proposed for the treatment of assembled chemical weapons early in the ACWA program (NRC, 1999). General Atomics is the sole developer of the GATS process, including the designs of all of the munitions-processing and dunnage-processing equipment. The balance of the plant design and site infrastructure was prepared by the Parsons Infrastructure and Technology Group.

The specific GATS technology design evaluated by the committee is for the treatment of the particular inventory of agent-filled munitions stored at Blue Grass (i.e., M55 GB and VX rockets, 8-inch GB projectiles, and 155-mm VX and H projectiles). These munitions are described in Table 1-3. The GATS process is designed to treat agent, energetic materials, metal parts (including munitions bodies), dunnage (e.g., wooden pallets and packing boxes used to store munitions), and nonprocess waste—the last-mentioned includes plastic demilitarization protective ensemble (DPE) suits; the carbon from DPE suit filters and plant heating, ventilating, and air-conditioning (HVAC) filters; and miscellaneous plant wastes.

The following discussion of the GATS process is based on Figure 5-1, which identifies the steps of the GATS process designed for a facility at Blue Grass Army Depot (General Atomics, 2001a).[1]

Disassembly of Munitions

Steps 1, 3, and 4 of the GATS process (Figure 5-1) incorporate comparatively minor modifications to existing baseline system procedures for the reverse assembly of munitions. These procedures were used at the baseline incineration system disposal facilities at Johnston Atoll (in the Pacific Ocean) and Tooele, Utah, where the Army has accumulated more than 10 years of experience in their operation. Rockets are processed by two rocket shear machines (RSM), each operating at the rate of 10 rockets per hour. The rockets are first punched and drained of agent, then flushed with hot water to remove any gelled agent. The flush operation is an addition to the GATS process. After agent removal, the rockets are sheared by the RSM into nine segments to access the fuze, burster, and rocket motor propellant. The sheared segments are transferred to the energetics rotary hydrolyzer (ERH).

Not all the projectiles in storage at Blue Grass Army Depot contain explosive burster charges. Those projectiles with bursters (155-mm HD rounds) are pro-

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[1] Note that the step numbers for the Blue Grass design have been modified and reordered from those identified for the GATS process designed for Pueblo Chemical Depot and given in NRC (2001a).

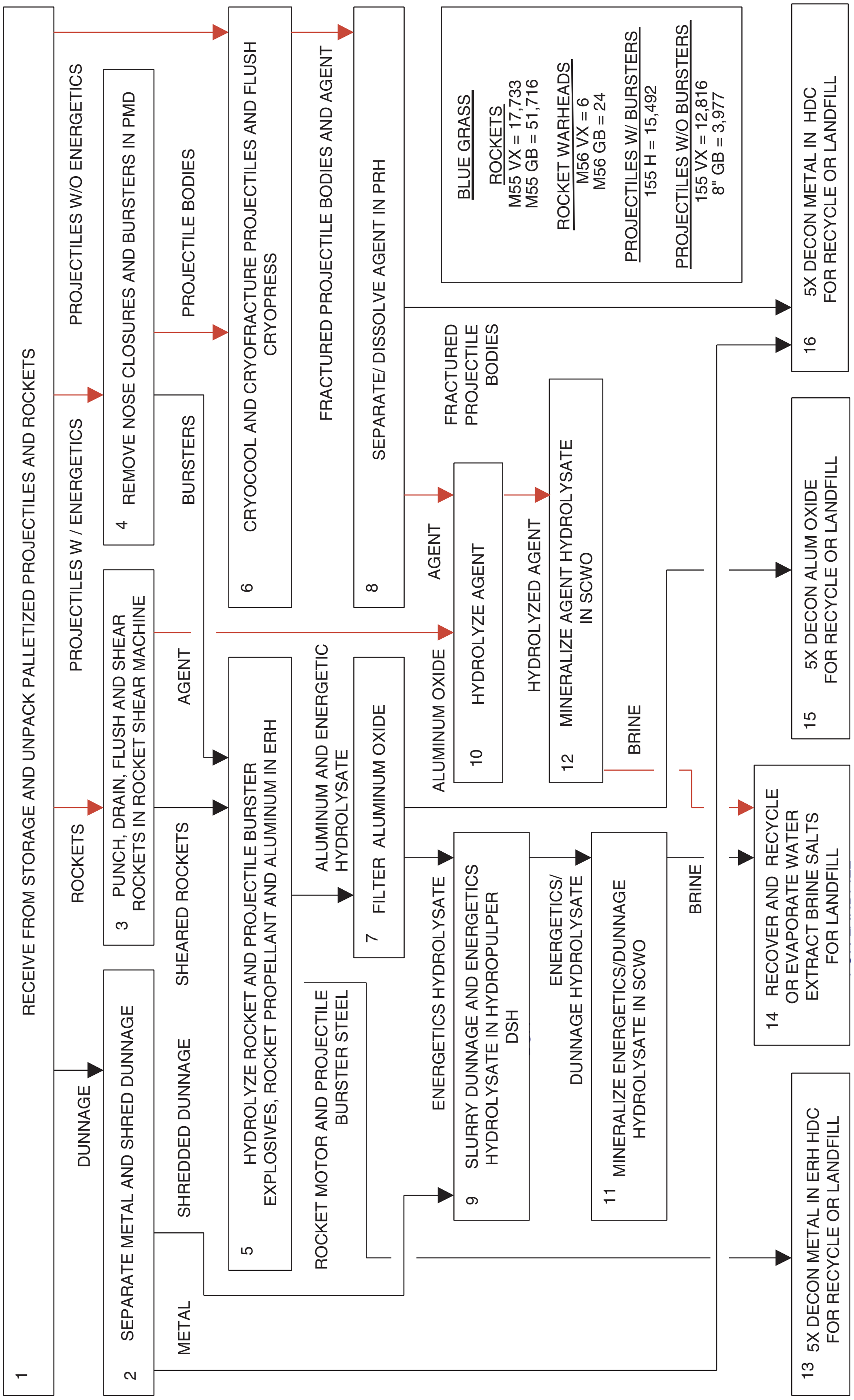

FIGURE 5-1 GATS Blue Grass block flow diagram. SOURCE: General Atomics, (2001a).

cessed through a single projectile mortar demilitarization (PMD) machine, which removes the whole bursters. Unlike the GATS process described in the ACW II Committee's report for Pueblo, the bursters removed from the munitions are not sheared as in the baseline system but are transferred directly to the ERH (NRC, 2001a; General Atomics, 2001a). Projectiles without burster charges do not go through the PMD. There are no propellant materials for the projectiles at Blue Grass.

Dunnage Separation, Shredding, Grinding, and Slurrying

Step 2 is the separation, shredding, and grinding of dunnage and nonprocess wastes. The dunnage shredding and handling system (DSHS) incorporates three subsystems to process (1) wood, originating mainly from pallets, (2) plastic and rubber from DPE suits, and (3) the activated carbon from respirators and from the plant's numerous carbon filters. The design is based on the assumption that all dunnage and nonprocess wastes are contaminated with agent. The organic materials (e.g., wood, paper, rubber, plastic, metal-free DPE suit material, and spent carbon) are reduced in a series of steps to a particle size of less than 1 mm. The resulting dry material is placed in storage bins from which it is fed into the two commercial hydropulpers, described in Step 9. The resultant slurried material is subsequently transferred via a grinding pump to the high-pressure pumps that feed the SCWO reactors. The wood, plastic and rubber, and spent carbon materials are processed in three separate equipment lines, but the respective fine slurry materials are mixed with energetics hydrolysate to make feed for the SCWO reactors.

Wood shredding is itself a four-step process. Pallets and similar dunnage materials are sheared in a low-speed shredder. This is followed by a Komar reducing screw feeder, which also equalizes the downstream flow. The dunnage materials are then further size-reduced in a hammer mill. Metal components, such as nails, are removed by magnetic head pulleys both after the low-speed shredder and after the hammer mill. The metal goes to the heated discharge conveyor (HDC) (Step 16) for 5X decontamination. In the fourth step, the milled wood with the metal removed is ground to less than 1 mm in a micronizer whose design includes a baghouse for dust control.

DPE suits are treated by the following steps. Metal parts are manually cut and removed from each DPE suit when the worker is cut out of the suit upon exiting a Category A area. The metal fittings are sent for 5X decontamination to the HDC that treats metal munition parts from the projectile agent removal system (Step 16). The metal-free DPE suit material is fed to a two-stage size-reduction system. In this operation, the material is first shredded to less than 10 mm in a dedicated granulator. Tests showed that further mechanical size-reduction in the granulator was ineffective because the polyvinyl chloride suit material melted rather than tearing. Therefore, after granulation, the DPE material is reduced to less than 1 mm in a cryomicronizer with a liquid-nitrogen-cooled screw feeder. The micronized wood and DPE are mixed together with energetics hydrolysate and spent decontamination solution in hydropulpers (Step 9) and processed through the SCWO reactors of the energetics hydrolysis system.

The spent activated carbon from plant HVAC filters and from respirators is first heat-treated in a heated, helical screw conveyor to drive off agent and is then reduced to less than 0.5 mm in a wet spent carbon grinder. Offgas from the processing of the carbon will be treated by the gas treatment system of either the projectile rotary hydrolyzer (PRH) or the ERH. The micronized carbon is mixed with water in the hydropulper (Step 9) and separately processed through the SCWO reactors of the energetics hydrolysis system.

Hydrolysis of Energetic Materials

In **Step 5**, energetic materials and associated metal parts from the PMD machine and RSM operations are sent to the ERH, a long, steam-jacketed rotating cylinder with internal spiral flights and lifting flights where the hydrolysis of energetics begins. The hydrolysate from the ERH then goes to continuously stirred tanks in which the hydrolysis is completed. The design for Blue Grass calls for two ERH systems.

Table 5-1 lists design parameters for the ERH (and the PRH discussed in Step 8). Hot water and sodium hydroxide solution, along with the energetic materials and associated metal parts, are fed into the ERH at one end and flow concurrently through the rotating cylinder. The liquid in the ERH drum is maintained at a minimum depth of 24 inches.

Hydrolysis of the energetic materials by the caustic leaves only small pieces of residual energetics, as shown during the EDS tests. The energetics feed rate is maintained to keep the explosive loading in the ERH to

TABLE 5-1 Key Design Parameters for GATS ERH and PRH

Parameter	ERH	PRH
Residence time (hr)	5	1
Drum diameter (ft)	8	6
Drum length (ft)	50	40
Flight (ft/pitch)	Spiral, 2.5	Spiral, 2.5
Nominal rotational speed (rotations/hr)	4	16
Operating temperature	105°C (bursters), 120°C (rockets)	194°F (90°C)
Operating pressure	Negative 0.5-inch water column to ambient	Negative 0.5-inch water column to ambient
NaOH (dry weight)		
(lb/lb propellant)	6.5 (7.3 × stoichiometric)	
(lb/lb explosives)	0.83 (1 × stoichiometric)	
(lb/lb aluminum)	1.482 (1 × stoichiometric)	
Liquid flow direction	Cocurrent	Countercurrent

SOURCE: Adapted from General Atomics (2001a).

less than 17.5 pounds TNT equivalent; the system is designed to safely contain an accidental detonation of this size. The solids residence time is approximately 5 hours, a time that was shown during Demo I and EDS testing to be sufficient for the essentially complete hydrolysis of rocket propellant in the original aluminum casing. Because of their large size and the metal casing, these chunks of propellant are considered by the designers (and by the committee) to represent worst-case conditions for processing by the ERH.

The ERH has two types of flights, spiral and lifting. The spiral flights transport material axially along the cylinder. The lifting flights slowly agitate the hydrolyzing solution and the solids. The drum is steam-jacketed to maintain the ERH contents at the desired temperature for the particular energetic materials being processed. The ERH is operated as a single-temperature-zone reactor when processing parts from projectiles and as a two-temperature-zone reactor when processing rocket parts.

For projectile processing, the temperature is maintained along the entire ERH length at 110°C. Because this is above the melting temperature of the TNT in the bursters, hydrolysis occurs rapidly in the caustic solution. The hydrolysis solution is maintained at 8 M NaOH at the feed inlet, and the feed rate is controlled to match the feed rate of the projectile bursters.

For rocket processing, the ERH is divided into two zones. The first part of the reactor, nominally 10 feet, is initially fed 4 M (14 weight percent) NaOH with the temperature kept at 105°C to accommodate the relatively quick dissolution of the aluminum and burster material. The residence time in the first 10 feet of the reactor is 1 hour. To handle the slower hydrolysis rate of the propellant, the remaining 40-foot section of the reactor—the second zone—is operated at high caustic concentrations and temperatures. Additional NaOH solution (50 weight percent) is added to the ERH through a pipe 8 feet from the feed inlet to raise the caustic concentration to 13 M (37 weight percent). The second zone is kept at 120°C.

At the discharge end of the ERH, the slurry is drained through a perforated section into a sump. The solid materials, which include unreacted fuze components, segments of fiberglass and steel casings from rocket motors, small quantities of residual rocket propellant, and steel burster tubes and nose closures, are lifted out of the solution by the spiral flights and fed into a chute leading directly onto the HDC, discussed in Step 13, for 5X decontamination.

The slurry from the ERH sump is pumped to one of two continuously stirred reactors (two for each ERH, four for the plant) to complete the hydrolysis, if necessary. Because the sodium hydroxide dissolves any aluminum present in the munitions, converting it to aluminum hydroxide, the aluminum hydroxide is prevented from clogging downstream components by neutralizing the completely reacted hydrolysate with hydrochloric or sulfuric acid, causing the dissolved aluminum to form a precipitate, which is then filtered (Step 7). The hydrolysate is sent to holding tanks to await secondary treatment in the SCWO reactors.

Air is drawn through the ERH to remove fumes, including particulate, mist, and the hydrogen produced from the hydrolysis of aluminum in the munitions, and to dilute the offgas to well below the lower explosive limit (LEL). The principal constituents of the ERH gas stream are these:

- ventilation air (room air), which is used to dilute hydrogen in the ERH
- nitrogen, which is used to purge the HDC
- water vapor, which evaporates from the hydrolysate in the ERH drum
- organic gases that evolve during the hydrolysis process, including nitroglycerine and nitrosodiphenylamine

The vent gas from the ERH flows through an electrically heat-traced duct (to prevent condensation) into an electric heat exchanger that raises its temperature to 260°C (500°F) and then into a CATOX unit to destroy residual organic contaminants. The gas is then cooled to between 12°C and 15°C (60°F) in a scrubber/condenser that removes additional contaminants. The gas then flows through an induced draft fan, discharging into the plant's HVAC system.

The pressure in the ERH is continuously monitored at the vent gas exit duct of the ERH drum to ensure that it operates at subatmospheric pressure. In addition, the hydrogen gas concentration is measured in the ERH drum vent gas exit duct to ensure that hydrogen gas concentration remains below 50 percent of its LEL. Signals from these monitors are used to control the ventilation gas flow by changing the speed of the induced draft fan.

Cryofracture of Munitions

Step 6 of the GATS process is the projectile agent removal system. After the energetic materials and associated metal parts have been removed and sent to the ERH, the agent cavity of the munition body is accessed by a cryofracture process. Cryofracturing involves first embrittling the munition by cooling it in a liquid nitrogen bath (77 K, –321°F, –196°C) and then transferring it via an overhead robot to a hydraulic press that cracks open the agent cavity, thereby exposing the frozen, solidified, or gelled agent and agent heels. The cracked, frozen agent and munitions go to the PRH (Step 8), which is discussed below.

The cryocooling bath is modeled after commercial food-freezing tunnels. Key design parameters for each cryofracture train are given in Table 5-2. Projectiles and mortars (minus energetic components) are conveyed from the PMD machine ECR to the cryobath loading station in a horizontal orientation. A cryobath loading robot places each round onto a moving link belt that conveys the munitions completely submerged through the liquid nitrogen bath. The residence time in the liquid nitrogen is sufficient to freeze the munition and associated agent to the temperature of liquid nitrogen. At the discharge end, the belt lifts the munitions out of the bath, and the unloading robot places it onto the anvil of the cryopress (hydraulic press).

TABLE 5-2 Key Design Parameters for Each GATS Cryofracture Train

Parameter	Value
Cryocooling conveyor (two trains)	
Dimension (ft)	25 (L) × 5 (W) × 4 (H) approx.
Max speed (ft/min)	0.75
Capacity	20 munitions (in each bath)
Liquid N_2 capacity (gal)	2,000 (in each bath)
Munition residence (min)	40, in bath
Cryofracture press (one train)	
Tonnage (tons)	500 (24-inch stroke)
Cycle time (sec)	30
Ventilation air flow (lb/hr)	4,800
Liquid nitrogen usage boil-off	1 lb liquid nitrogen/lb munition + 400 lb/day
Flush water per fracture (gal)	1.0

SOURCE: Adapted from General Atomics (2001a).

The press base is a tilt table that sends the cracked munitions into a discharge chute, which in turn sends both the metal and frozen agent into the feed chute of the PRH. Residual material, including any melted agent, is flushed from the cryopress with hot water into the PRH along with the remaining munitions components. Test data appear to indicate that the cryopress operation provides good removal of the agent from the munition body and improves the accessibility of the hydrolysis solution in the PRH to the metal surfaces of the agent cavity (General Atomics, 1993).

The cryofracture system for cracking chemical weapons was developed and tested by General Atomics for the Army during a test program that is discussed later in the chapter. Munitions-processing bay-bridge robots, as used in the baseline system, have been fitted with new end effectors for loading and harvesting munitions from the cryobath and transferring them into the cryopress.

Aluminum Precipitate Filtration

In **Step 7**, after the energetics hydrolysate from the continuously stirred tank hydrolysis operation has been pumped into a holding tank, acid is added to precipitate aluminum, and the hydrolysate is filtered through an automatic filter press to remove precipitated aluminum compounds. The liquid effluent goes to the dunnage hydropulper (Step 9). The filter cake from the press is sent to an electrically heated screw conveyor (Step 15) for 5X treatment.

Projectile Rotary Hydrolyzer

In **Step 8**, accessed frozen agent is hydrolyzed with hot caustic (when processing GB and VX) or hot water (when processing HD), and agent-contaminated metal parts from the cryofracturing step are washed in one of two PRHs. The PRHs are smaller but similar in function and construction to the two ERHs (see Table 5-1). The liquid flow in the PRH is countercurrent to the flow of solids. The PRHs are externally steam heated to maintain the temperature at the desired level.

The drum of the PRH is fitted with a spiral flight and lifting flights to transport and mix the munition fragments axially along the drum from feed to discharge. A stationary shell of thermal insulation encloses the drum. Depending on the agent being processed, either caustic (GB and VX) or hot water (HD) is introduced at the discharge end and flows countercurrent to the stream of solids. The liquid, which dissolves the frozen agent and/or agent heels, is discharged through a screen at the feed end of the PRH, separating the solution from the freshly introduced metal fragments. At the discharge end, the lifting flights lift the metal fragments out of the solution onto a chute leading to an HDC, which is distinct from but similar to the HDC to which ERH materials are discharged.

The PRH hydrolysate is discharged to a stirred tank, where the hydrolysis of agent is completed. Air is drawn through the PRH to remove volatile materials, and the gaseous effluent is passed through a CATOX, scrubber, and carbon filters prior to release to the plant's HVAC system. The solids, consisting of the washed munitions parts, go into the HDC that treats metal parts from the projectile agent removal system (Step 16), where they are decontaminated to a 5X level.

Dunnage Hydropulping

In **Step 9** of the GATS process, the dry, size-reduced dunnage materials and nonprocess wastes from Step 2 are slurried with energetics hydrolysate in preparation for feeding to one of the SCWO reactors. Following the removal of precipitated aluminum compounds by filtration in Step 7, the energetics hydrolysate is transferred to one of two hydropulpers. Spent decontamination solution used in various decontamination operations in the plant also goes to the hydropulper tanks.

In the hydropulpers, the ERH hydrolysate fluid is mixed with the dry material produced from shredding and micronizing the organic dunnage and other waste materials to produce a slurry. Additional water or a dilute solution of NaOH is added as needed to adjust water content, neutralize any residual agent, and otherwise adjust the slurry to meet the feed requirements of the SCWO reactors. Proprietary additives are used to ensure that the solids remain in suspension and that the slurry can be reliably pumped and processed in the SCWO reactor system (Step 11). The hydropulper tanks are continuously stirred and periodically sampled prior to transfer of their contents to an energetics hydrolysate slurry storage tank, from which the slurried dunnage and neutralized energetics are pumped to the SCWO reactors.

Completion of Agent Hydrolysis

In **Step 10**, the agent drained from rockets or the PRH hydrolysate solution (and any residual agent) is transferred to one of the four batch reactor vessels of the projectile agent hydrolysis system. These vessels, equipped with high-shear agitators and containing caustic solution (or water when processing agent HD), complete the hydrolysis of the agent drained from rockets or the remaining agent in the PRH hydrolysate solution. The hydrolysate then is stored in the vessel pending verification of agent destruction. These reactor vessels are similar in design to the ones that will be used in the hydrolysis of bulk mustard agent at the Aberdeen Chemical Agent Disposal Facility. See Table 5-3 for key design parameters of the neutralization system tanks.

Treatment by Supercritical Water Oxidation

Step 11 of the GATS process is treatment of the hydropulped dunnage slurried with energetics hydroly-

TABLE 5-3 Key Design Parameters for the GATS Projectile Agent Hydrolysis System

Mustard (HD) agent hydrolysis
- Eight 6,428-lb, 15-wt% HD agent /water batches per day . . .
- Following confirmation of agent destruction, HCl is neutralized with sodium hydroxide.
- Batch hydrolysis at 194°F
- Reaction time 1½ hr
- Total batch time 5½ hr

Sarin (GB) agent hydrolysis
- Eight 5,205-lb, 7-wt% GB agent /NaOH batches per day . . .
- Batch hydrolysis at 120°F
- Reaction time 2 hr
- Total batch time 5 hr

VX agent hydrolysis
- Three, 5,104-lb, 22.7-wt% VX agent /NaOH batches per day . . .
- Batch hydrolysis at 194°F
- Reaction time 5 hr
- Total batch time 11 hr

SOURCE: This table was reproduced from General Atomics (2001a). The committee has omitted the chemical equations because they were not balanced and some formulae were inaccurate.

sate by SCWO. **Step 12** is the treatment of agent hydrolysate by SCWO. The final GATS design for Blue Grass includes five SCWO reactors (General Atomics, 2001a). The basic SCWO reactor is a retort-style pressure vessel with injectors at the upper end to feed the wastes, air, and a supplementary fuel. Isopropyl alcohol is used as a supplementary fuel during start-up or when needed to maintain the temperature.

Early tests of the SCWO reactors (discussed below) indicated significant corrosion and plugging. Plugging problems were solved by procedural modifications that include periodic flushing with clean water at slightly subcritical conditions. The corrosion is mitigated by inserting various composite liners that resist the corrosivity of the different feed streams.

The EDS II tests (discussed later in the chapter) showed that titanium backed by Hastealloy C-276 provides adequate corrosion resistance for treating tetrytol hydrolysate, M28 propellant hydrolysate, and slurried dunnage in these two hydrolysates. It also showed that C-276 alloy has sufficient corrosion resistance for treating VX hydrolysate, given the short duration of the VX disposal campaigns at Blue Grass. When treating HD and GB hydrolysate, corrosion was severe for all materials. Thus, the design calls for using an additional sacrificial titanium wear liner that is thick enough to withstand 500 hours of operation (assuming a corrosion rate equal to that measured during the EDS II testing). Although the corrosion rate is high, the liner is designed so that no replacement is necessary during processing of the comparatively small quantities of H in the Blue Grass stockpile. Processing the far greater amounts of GB in the stockpile would require a changeout of the sacrificial titanium wear liner, whose thickness during EDS was sufficient to withstand 120 hours of operation. Therefore, during the GB campaign, the technology provider has scheduled each reactor to be periodically removed from service under a conservative preventive maintenance program that would include replacement of the wear liner and the full-length removable liner after 110 hours.

The different liner configurations proposed for use at Blue Grass are shown in Figure 5-2. The first three liner configurations (left to right) shown are the reference designs proposed for the GATS SCWO reactors at Blue Grass on which all of the operations are based. The two rightmost designs are tentative improvements in the liner system. General Atomics proposes to investigate these backup designs if subsequent tests confirm their improved performance, or if they are needed for other reasons. Test results to date on these two "product improvement" designs are noted later in this chapter, but these liner configurations were not considered by the committee as part of its evaluation of the current GATS SCWO system design.

Thus, the final design as evaluated consists of a liner system composed of an Inconel 600 insulating barrier over a Hastelloy C-276 removable sleeve. The Hastelloy C-276 sleeve is mounted in the reactor by bolting the flange on the top of the sleeve between the cylindrical body of the reactor and the reactor head. Water-cooled seals are used in this joint. A full-length, cylindrical removable liner is inserted into the Hastelloy C-276 sleeve assembly. The two are separated by spacers, which also hold the removable liner in place while allowing some differential expansion and contraction. The liner during the disposal campaigns when tetrytol and M28 propellant hydrolysates and slurried dunnage are treated is a commercially available titanium pipe. When processing VX hydrolysate, the liner is made of Hastelloy C-276 pipe. When processing HD and GB hydrolysates, a titanium pipe is inserted as an additional sacrificial wear liner into the

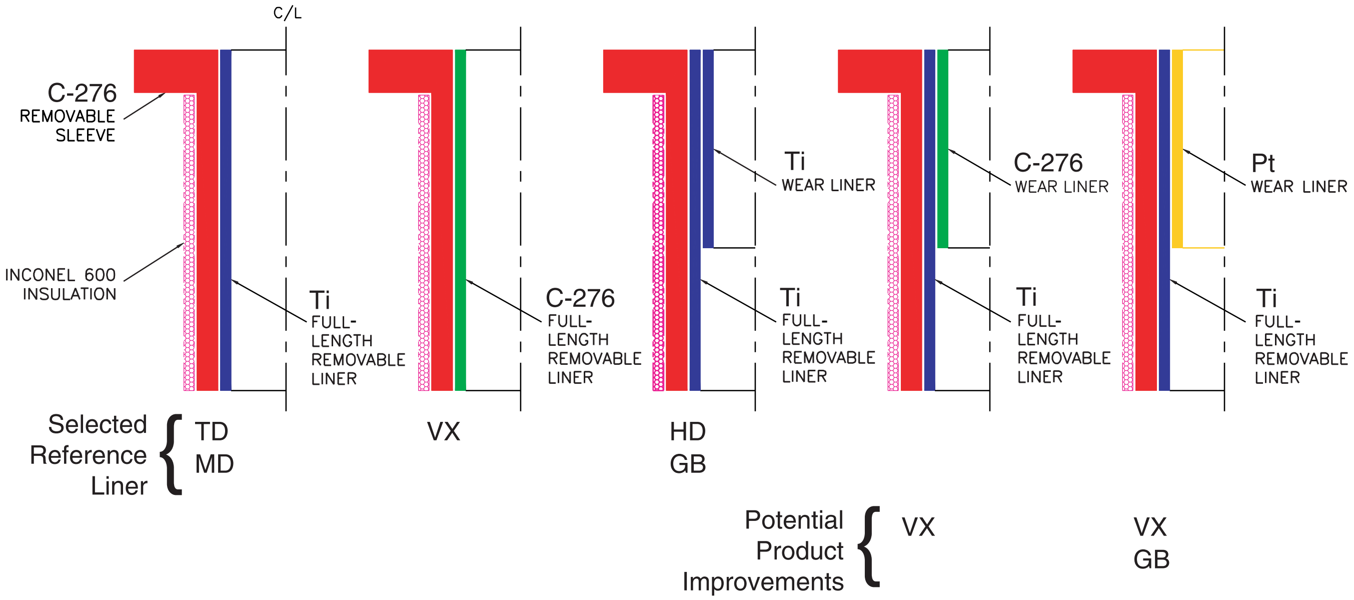

FIGURE 5-2 GATS SCWO liner materials and configuration. SOURCE: General Atomics (2001a).

full-length titanium liner at the top portion of the reactor, where the corrosion has been found to be the worst. In summary, all of the GATS composite liner systems consist of three major components: (1) an Inconel 600 insulating barrier, (2) a Hastelloy C-276 outer sleeve liner, and (3) a full-length, removable liner made of titanium or Hastelloy C-276. In addition, for HD and GB hydrolysates, a short-wear liner is inserted into the removable liner.

The GATS design calls for five SCWO reactors. Three of the SCWO units are dedicated to processing the energetics hydrolysate slurry and two to processing agent hydrolysate. The three SCWO units for processing energetics hydrolysate slurry have titanium liners that are expected to last for the duration of operations at a Blue Grass facility. The two agent hydrolysate SCWO units have liner types and change-out frequencies appropriate for the specific agent. With two SCWO units used for agent hydrolysate, one unit is always available as a spare.

Table 5-4 gives the specifications for the SCWO reactors. The reactors operate at approximately 650°C (1,200°F) and 3,400 psig. These conditions are well above the critical temperature and pressure of water. The oxidizer is either pressurized ambient air or a synthetic air consisting of a mixture of oxygen and nitrogen at a 21:79 volume ratio, delivered at a feed rate that is 20 percent in excess of the stoichiometric requirement.[2] Isopropyl alcohol and water are used to adjust the waste feed to the proper heating value. The isopropyl alcohol is added as an auxiliary fuel whenever needed to maintain an autogenous[3] feed mixture to the SCWO reactor.

During system start-up, an electric preheater and the isopropyl alcohol feed are used to heat the reactor to the desired operating temperature. When the reactor is at operating temperature, the preheater is turned off, hydrolysate flow is initiated, and auxiliary fuel and water flows are adjusted to control the temperature at the set point.

The fluid discharged from the SCWO reactor passes through a water-cooled heat exchanger and staged-pressure gas-liquid separation equipment. Noncondensable gases, mostly nitrogen, carbon dioxide, and the excess oxygen, are monitored and filtered before release to the environment via the plant HVAC HEPA filter and activated carbon adsorption system. Liquids are monitored and transferred to the brine recovery area. If fluid does not meet release specifications, it is returned to a storage tank for off-specification product and reprocessed in the SCWO reactors.

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[2] The equipment that will be used had not been specified at the time this report was prepared.

[3] Autogenous means that the heat released from the oxidation reaction is sufficient to maintain the reactor temperature so that no external heat is needed to maintain the process temperature.

TABLE 5-4 SCWO System Design Parameters

Equipment Component	Requirement (Normal Operating Conditions)
Reactor	18.4-in. I.D. × 221 in. long (process dimensions) 3.25-in. thick vessel (1 1/4 Cr, 1/2 Mo) steel
Removable liner, outer support sleeve	¼-in. C-276
Removable liner, full length, inner corrosion-resistant sleeve	¼-in. C-276
Removable liner assembly in upper reactor zone	Multiple liner options depending on feed
Process oxidant	Air
Water feed pump (to preheater)	6 gpm, 3,800 psi
Hydrolysate feed pump	15 gpm, 3,800 psi
Quench pump	32 gpm, 3,800 psi
Auxiliary feed pump	3 gpm, 3,800 psi
High-pressure oxygen system	3,100 lb/hr (for synthetic air)
High-pressure nitrogen system	12,800 lb/hr (for liner purge and synthetic air)
Hydrolysate tank	10-hr holdup, 25% free space, 12,000 gal
Water tank	4-hr holdup, 25% free space, 2,400 gal
Auxiliary fuel	750-gal tank
Transfer pumps	4 pumps
Start-up preheater	600 kW steam generator, 1,000°F, 3,800 psi
Cooldown heat exchanger	22 million Btu/hr, U-tube heat exchanger
Gas pressure letdown	Redundant control valves and pressure regulators
Liquid pressure letdown	Redundant capillaries and control valves

NOTE: Two SCWO units are for processing energetics hydrolysate (slurried with dunnage), two SCWO units are for processing agent hydrolysate, and one SCWO is available for either service; five interchangeable trains total.

SOURCE: Adapted from General Atomics (2001a).

As previously noted, the three-component basic liner is fitted into the SCWO reactor pressure vessel by bolting the flange of the Hastelloy C-276 sleeve liner (the horizontal component in Figure 5-2) between the SCWO reactor pressure vessel and the reactor head. A small flow of nitrogen gas is maintained in the gap between the liner and the pressure vessel. The nitrogen flow insulates the reactor wall, keeps corrosive fluids from touching the reactor wall, and prevents binding of the liner assembly to facilitate its removal. With this design, the liner can be periodically replaced to compensate for corrosion, and the pressure vessel can be fabricated from low-alloy steel with a relatively thin corrosion-resistant coating.

Treatment of Metal Parts

In **Step 13**, solids from the ERH pass through a chute to an HDC; in **Step 16**, metal parts from the PRH pass through another chute to a similar but different HDC. The HDCs are electrically heated and purged with nitrogen. The materials in each HDC are heated to 1,000°F (538°C) and held at temperature for at least 15 minutes. The conditions are sufficient to decontaminate the solids to a 5X level or to cause residual energetic materials that might remain in the solids to decompose. The solids leaving the HDCs are cooled and disposed of off-site. Offgases from the HDCs in Steps 13 and 16 are passed through the respective gas treatment systems of the ERH or PRH before being discharged to the plant ventilation system.

Water Recovery and Salt Disposal

In **Step 14**, the brine from the four SCWO reactors is concentrated using evaporation/crystallization equipment to reclaim the water and generate solid salt cake for off-site disposal. The evaporation/crystallization step has been eliminated from the designs for the bulk agent disposal facilities at the Newport, Indiana, and Aberdeen, Maryland, sites, and brine from the baseline system facility at Tooele, Utah, is being sent off-site rather than processed in the facility's brine reduction area. It is the committee's understanding that the off-site disposal of SCWO liquid effluent and the elimination of this step is being investigated for Blue Grass as well.

The brine recovery system (BRS) receives the effluents from the SCWO units processing agent hydrolysate and energetics hydrolysate slurry and separates the water for recycle from the salts for disposal. The BRS consists of a reverse osmosis system, a regenerative heat exchanger, a brine concentrator, a crystallizer, and solids separators. The BRS converts the SCWO effluent, which is approximately 2 to 4 weight percent salt solution (primarily sodium sulfate, sodium fluoride, sodium chloride, and sodium monophosphate), to a salt cake and a high-purity water that is recycled into the process as needed. Unneeded water is discharged.

Decontamination of Aluminum Precipitate

In **Step 15**, the precipitated aluminum salts produced in Step 7 are decontaminated. This step is necessary to ensure that the precipitate meets 5X decontamination requirements before being sent off-site for disposal. The 5X decontamination is to be done in a dedicated electrically heated screw conveyor.

Design and Throughput Basis for the GATS

The GATS process is designed to process the stockpile of chemical munitions stored at Blue Grass Army Depot (shown in Table 1-3). It treats the entire assembled munition, including agent, energetics, munition casing, and associated packaging and processing waste materials. The quantities of these materials in need of processing are listed in Table 5-5.

TABLE 5-5 Waste Materials to Be Processed per Munition

Material	Amount
Wood	4.33 lb per rocket 8.55 lb per 8-in. projectile 12.67 lb per 155-mm projectile
Decontamination solution	5 lb per round
DPE suits	0.15 lb per round
Carbon	0.3 lb per round
Waste oil	0.3 lb per round
Miscellaneous metal	0.4 lb per round
Hydraulic fluid	0.4 lb per round

SOURCE: Adapted from General Atomics (2001c).

The GATS process is designed to coprocess projectiles and rockets containing the same type of agent to minimize processing time. Three campaigns are planned: one for coprocessing GB projectiles and GB rockets, one for coprocessing VX projectiles and VX rockets, and one for HD projectiles alone.

Five sets of mass and energy balances have been produced: (1) GB projectiles and rockets coprocessing, (2) GB rockets alone (after all GB projectiles are gone), (3) VX projectiles and rockets coprocessing, (4) VX rockets alone (after all VX projectiles are gone), and (5) HD projectiles alone. Processing rates have been selected, as shown in Table 5-6.

The duration of the processing campaigns for munitions disposal is set by the processing rates of the two RSMs and the one PMD machine, with the other processing equipment sized to match these rates. The RSMs are operated during the GB and VX rocket campaigns. The PMD machine extracts bursters during processing of HD projectiles only. It acts only as a conveyor during the GB and VX campaigns, with no operation on the projectiles. The RSM operating rate is 10 rockets per hour per machine, a 6-minute cycle during which one rocket is brought into the ECR, punched and drained, and flushed. During the same 6 minutes, a second rocket is moved from the punch-drain-flush station to the RSM, is sheared into nine segments, and then is transferred to the ERH. This processing rate is well within the demonstrated processing rate for the RSM in the baseline incineration system. Experience at Johnston Atoll Chemical Agent Disposal System (JACADS) with the RSM shows typical rates of 18 to 22 rockets per hour per RSM (without flushing).

The maximum operating rate of the PMD machine is specified at 50 projectiles per hour. This rate is used only for the 310 processing hours scheduled for 155-mm HD projectiles. In the 72-second cycle, the PMD machine loads one projectile on the rotating table, while in the same period removing the nose closure from a second projectile, the fuze well cup and burster from a third, and unloading a fourth projectile from the table to the projectile agent removal system. Experience with the PMD machine at the Johnston Atoll baseline system facility shows that the theoretical maximum operating rate of 144 projectiles per hour has never been achieved. Maximum rates of 48 rounds per hour were used for 155-mm HD and 155-mm VX processing. Two-thirds of the rounds were processed at rates exceeding 30 rounds per hour, and more than one third at rates exceeding 40 rounds per hour. Lessons learned during baseline system operations at Johnston Atoll are expected to improve the rate and availability of the PMD machine.

TABLE 5-6 Munition Processing Rates and Durations

Munition Name	Number (rounds)	Processing Rate (rounds/hour)	Processing Time (hours)	Processing Days (at 12 hours/day)
M55 GB rocket	51,417	20	2,571	214
8-inch GB projectile	3,977	10	398	Coprocessed with GB rockets
M55 VX rocket	17,733	20	886	74
155 mm VX projectile	12,816	20	641	Coprocessed with GB rockets
155 mm HD projectile	15,492	50	310	26
Total	101,435			314

SOURCE: Adapted from General Atomics (2001a).

A GATS facility for Blue Grass would operate 24 hours per day, 46 weeks per year. According to the General Atomics EDP, the equipment for munitions processing, energetics and agent extraction, and hydrolysis is assumed to operate productively 72 hours per week (averaging 12 hours per day, 6 days per week). SCWO equipment is assumed to operate productively 132 hours per week (averaging 22 hours per day, 6 days per week), but with only 50 percent utilization (i.e., 100 percent spare capacity is provided). The GATS process design is sized on the assumption that a Blue Grass facility overall will only be operating productively 38 percent of the time, i.e., 38 percent availability.

The requirement for 38 percent online availability means an offline allowance of 62 percent of the time, which can be used for equipment maintenance and repairs. The required capacity factor of 38 percent sets a very low reliability requirement for the equipment and provides a margin for combined equipment failure rates within a processing line.

With the selected RSM and PMD machine rates, all equipment and processes are sized to achieve the required average weekly output of 3,600 155-mm H projectiles, 1,440 155-mm VX projectiles, 720 8-inch GB projectiles, or 1,440 rockets (VX or GB) per week. If the actual average operating hours achieved during the munition processing campaigns exceed 72 hours per week, the plant would complete operations ahead of schedule.

The GATS process components are sized so that all material processing can be balanced on a weekly basis. For example, all agent hydrolysate and energetics/dunnage hydrolysate produced during a given time period would be completely processed by SCWO during a similar time period. Buffer storage at the front end of each process is sized to prevent minor upsets in flow from affecting total throughput. The ERH and PRH operate on a cycle that follows the operation of the RSMs and PMD machine or cryofracture steps. Hydrolysate and decontaminated metal parts emerge from the projectile agent removal system and ERH steps after the design residence time—from 2 to 6 hours. Hydrolysate is collected in buffer storage tanks so that the SCWO operations can be independent of the other parts of the process.

The 16 unit operations shown in Figure 5-1 are evaluated by the committee in subsequent sections of this chapter. For clarity, some secondary unit operations (such as CATOX units) are not shown in Figure 5-1. The GATS design anticipates the movement of munitions from storage to the MDB using modified ammunition vans (MAVs). Transport will be in two steps: first to the container handling building (CHB), and then to the unpack area (UPA) in the MDB.

INFORMATION USED IN DEVELOPING THE ASSESSMENT

Engineering-Design-Related Documents

In December 2001, General Atomics issued a final draft of the EDP for a full-scale pilot plant implementing its technology package at Blue Grass Army Depot (General Atomic, 2001a). The EDP includes technical descriptions and data, drawings, a preliminary hazard analysis, and cost and schedule analyses. The committee used it as the primary source of information for this assessment. Other EDP-related documents used in the assessment include the initial draft of the EDP and the study plan and test reports for the EDS submitted by General Atomics (with addendum) (General Atomics, 2000a, 2001c, 2001d). Committee members also attended design review briefings by technology provider team members, who discussed and clarified intermediate plans and activities associated with the development of the facility design (General Atomics, 2001b, 2001e, 2001f). Assessments of the General Atomics technology package in earlier NRC reports were also considered (NRC, 1999, 2000a).

The engineering design drawings and associated documentation for the proposed facility provided to the committee were very extensive. The committee concentrated its efforts on evaluating the following critical components of the GATS process design that had been identified as potential concerns in earlier NRC reports (NRC, 1999, 2000a, 2001d):

- the long-term reliability of the SCWO system
- the advantages and disadvantages of cryofracture over baseline technology as a means of accessing the agent in the munitions
- the ability of the rotary hydrolyzers (both the PRH and the ERH) to process their respective feed materials in a reasonable time and with acceptable safety and reliability

Overview of Engineering Design Studies and Tests

The committee's review of test data and documented design developments took place concurrently with ongoing testing by the technology provider. Thus, most of the data were available only in draft form. Some information was provided to the committee orally through briefings by the PMACWA and by the technology providers. In developing its assessment, the committee used draft and final reports of the various tests as well as weekly updates on the progress of the testing.

In addition, the committee obtained from General Atomics two compact disks containing a compilation of test reports on the use of cryofracture as a means to access the agent in munitions (General Atomics, 1993). General Atomics had investigated and designed cryofracture systems for the Army from 1982 through 1993. In these tests, General Atomics had processed 3,695 explosively configured agent-simulant-filled projectiles, rockets, and mines through cryofracture. All of those tests successfully breached the agent cavity in the munition. The tests also successfully breached the energetics cavities, but these results are not relevant to the Blue Grass design, which calls for removal of the energetics prior to cryofracturing.

Engineering Tests

General Atomics conducted an extensive series of tests on selected components of the GATS design to gather necessary design data. The tests began with Demo I and then proceeded to the EDS I phase. The Demo I tests showed proof of concept for the basic SCWO system as an effective means to treat hydrolysate (NRC, 2000a). The Demo I tests also identified problems with corrosion and plugging that would need to be resolved in a full-scale system. The tests also gathered data for the evaluation of the ERH, DSHS, and SCWO components of the GATS process. The results of the ERH and DSHS tests, as well as initial EDS results of SCWO testing, were discussed in earlier NRC reports (NRC, 2000a, 2001a).

The Demo I tests were performed on three unit operations of the GATS: (1) a batch version of the energetics rotary hydrolyzer, (2) a dunnage shredding and reducing system, and (3) a SCWO reactor. The tests on the first two systems were by and large highly successful. They identified some design problems, which could be resolved with comparatively simple design modifications. During the EDS phase, the dunnage shredding and slurrying system and the ERH were further tested and demonstrated. These tests, which included the shredding, micronizing, and slurrying of spent activated carbon, wood pallets, and DPE material, were successful. The EDS testing of the SCWO system totaled 5,773 hours of operation.

Supercritical Water Oxidation Testing

The EDS II phase of the ACWA program included a number of tests of the General Atomics' SCWO system (General Atomics, 2001g, 2001h, 2001i, 2001j). Extensive testing and development of the SCWO system has been the subject of a number of test programs sponsored by PMACWA and other units of the Army engaged in chemical demilitarization activities. That testing was in turn reviewed and evaluated by several NRC committees in earlier NRC reports. The General Atomics' SCWO system has undergone the following test stages:

- *Engineering-scale test (EST).* The EST addresses the application of General Atomics SCWO technology for the disposal of VX hydrolysate from the processing of bulk VX stored at the Newport, Indiana, site. This work was sponsored by the Army's Product Manager for Alternative Technologies and Approaches. NRC reports pertaining to the use of SCWO at Newport and EST testing were issued by the Committee on Review and Evaluation of the Army Chemical Stockpile Disposal Program (NRC, 1998, 2000b, 2001d). These tests showed that corrosion was a major problem that needed to be resolved before full-scale system design was possible (NRC, 2001d).

- *Demonstration I*. This phase of the ACWA program included a series of tests to validate that the proposed General Atomics technology package (GATS), which included use of SCWO, was suitable for disposal of assembled chemical weapons. The ACW I Committee's evaluation of the results of these tests is given in NRC, 2000a. This set of tests further clarified the corrosion problem.
- *EDS testing*. This is the series of tests conducted to support the engineering design studies General Atomics has put forth for use of the GATS technology at Pueblo Chemical Depot and Blue Grass Army Depot. Tests of the GATS SCWO system during the EDS phase of the ACWA program are listed in Table 5-7 and are discussed below. Additional details concerning the earlier SCWO system testing during the EDS phase can be found in NRC (2001a, 2001c).

The Demo I and EST tests demonstrated that although SCWO was an effective method for destroying the organic materials found in agent and energetics hydrolysates and in dunnage, the reactor was subject to two problems—suitable materials of construction and effective transport of solids. The Demo I tests with the SCWO reactor, conducted on HD hydrolysate simulant in a reactor with a platinum corrosion liner, showed corrosion rates as high as 10 mils per day. The tests also showed that titanium might be suitable as an alternative sacrificial liner for this application.

TABLE 5-7 Major SCWO System Test Campaigns Conducted in Support of GATS Design Considered by the Committee

Test	Test Dates	Total Hours of Testing	Reference
EDS (early testing)	10/4 to 12/17/00	332[a]	General Atomics, 2001h
Fuels operation		892[b]	General Atomics, 2001b
HD	1/3 to 1/29/01	1,172	General Atomics, 2001a
TD	3/17 to 4/14/01	897	General Atomics, 2001g
MD	5/22 to 6/29/01	751	General Atomics, 2001h
GB	8/9 to 9/11/01	934	General Atomics, 2001i
VX corrosion	9/12 to 10/19/01	201	General Atomics, 2001d, 2001j
VX	10/19 to 11/12/01	976	General Atomics, 2001a
Carbon	11/26 to 12/2/01	153	General Atomics, 2001a
Total hours processing wastes		5,416	
Total hot hours		5,773	

[a] Hours included in the total hours below.
[b] Not actually testing.

Key:

Carbon	Micronized carbon slurry
EDS	Engineering design study testing
HD	HD hydrolysate
TD	Tetrytol hydrolysate slurried with shredded and micronized dunnage (palettes, DPE suits, gloves, etc.)
MD	M28 rocket propellant and burster hydrolysates slurried with shredded and micronized dunnage that has had the hydrolyzed aluminum removed by filtration. These tests included the results of aluminum filtration tests. This test also demonstrated a modified chemistry for controlling solids transport through the reactor without the use of an additive that degrades the titanium reactor material.
GB	GB hydrolysate
VX	VX hydrolysate

The EDS tests shown in Table 5-7 generated much of the data that were used to design and specify the current GATS SCWO system. Based on this new information, the committee reassessed the SCWO system's performance and operability. The most important results of EDS SCWO testing are as follows:

- The SCWO system destroys all organic materials in the feed to a better than 99.9999 percent DRE, with hydrolysis previously also having destroyed the agent in the agent hydrolysate feed to better than 99.9999.
- Gaseous discharges from the SCWO system are of low volume and contain only very low (generally below levels of detectability) concentrations of hazardous materials, including chlorodibenzodioxins and chlorodibenzofurans.
- No problems were encountered in over 1,600 hours of testing during the treatment of slurries of dunnage and hydrolysates of tetrytol or M28 propellant, as shown in Table 5-7.
- The pumps of the SCWO system worked well, and no unacceptable plugging occurred.
- The removable SCWO reactor liner system developed for use at a Blue Grass facility (the leftmost liner of Figure 5-2) appears to have performed well with energetics hydrolysates and HD agent hydrolysates. The corrosion rate of titanium is higher for VX hydrolysate than for GB hydrolysate, which in turn is higher than for HD hydrolysate. A Hastelloy C-276 liner gave much better results with VX hydrolysate and has now been included in the design for the VX disposal campaign.
- The plugging problems that were encountered were controlled by reducing the temperature below the critical point of water and flushing the system for 2 hours every day.

Energetics Rotary Hydrolyzer Testing

The ERH testing was described in the NRC report on EDS I testing (NRC, 2001a). The tests were conducted at the Chemical Agent Munitions Disposal System test site in Utah. They consisted of rate-of-hydrolysis tests for the following feed materials:

- tetryl, tetrytol, and Composition B bursters in 8 M NaOH at 105°C (220°F) (done as part of Demo I testing)
- single 4-inch rocket motor segments in 12 M NaOH solution at 110°C (230°F)
- single 8-inch rocket motor segments in 12 M NaOH solution at 110°C (230°F)
- multiple rocket motor segments in 12 M NaOH solution at 110°C (230°F)
- single and multiple rocket motor segments in 12-14 M NaOH solution at 120°C (248°F)

The results of the ERH tests were as follows (General Atomics, 2001k):

- The rate-limiting step in the operation of the ERH is the hydrolysis of propellant, not the hydrolysis of explosives.
- Single 4-inch rocket motor segments of M28 propellant can be hydrolyzed at 110°C (230°F) in 5 hours with less than 5 grams of propellant remaining.
- Single 8-inch rocket motor segments of M28 propellant can be hydrolyzed at 110°C (230°F) in 5.5 hours with less than 5 grams of propellant remaining.
- As many as eight 4-inch rocket motor segments (one complete rocket) of M28 propellant can be hydrolyzed at 110°C (230°F) in 7 hours with less than 30 grams of propellant remaining, or in 7.5 hours with less than 5 grams of propellant remaining.
- As many as eight 4-inch rocket motor segments (one complete rocket) of M28 propellant can be hydrolyzed at 120°C (248°F) in 3 hours with less than 30 grams of propellant remaining, or in 3.5 hours with no propellant remaining.
- The negative-draft pollution-abatement system with condensing scrubber and mist and particulate filters effectively captured nitrosodiphenylamine and other fugitive emissions.

In the above tests, the ERH was operated in a batch mode rather than a continuous mode. The tests investigated the effect of caustic concentration and process temperature on the rate of reaction. The tests were focused mainly on rocket segments to optimize the processing conditions for M28 propellant. Results from the EDS tests were used for sizing the full-scale ERH and establishing the residence times of the munitions in the hydrolysis solution. Test data had been generated during Demo I for the energetics associated with the 155-mm projectiles stored at Blue Grass. Additional energetics hydrolysis testing is described in Chapter 2.

Dunnage Shredding and Handling System Testing

The following is a summary of previously reported testing of the DSHS conducted during EDS I. Originally from the committee's report on the technology options for the Pueblo site, the text has been adapted for applicability to the Blue Grass site (NRC, 2001a).

EDS testing of the DSHS was performed on full-scale equipment. Although the smallest commercially available shredding equipment was used, it was apparent that its capacity is substantially greater than what would be required at a Blue Grass disposal facility. The only problems encountered during the testing related to the mismatch between the operating rates of the various wood shredders in the train, which caused pulsing in the overall feed through the system. During the EDS testing, a Komar reducing screw feeder was added immediately downstream of the low-speed shredder to even out the flow. This addition appears to have corrected the problem.

The tests on DPE suit material and wood addressed size-reduction and material-transport problems identified during the Demo I testing (NRC, 2000a). DPE suit and butyl rubber simulant materials were shredded in a dedicated granulator, cryogenically cooled in a cryocooler with an internal screw conveyor, and reduced in a cryocooled hammer mill. No materials contaminated with agent were tested.

EDS testing of cryogenic micronization of DPE suit material was completed at Pulva Corporation facilities in Saxonburg, Pennsylvania. General Atomics provided PMACWA-supplied feed materials, guidelines for sampling, a test plan, and operating procedures. Pulva Corporation provided the test facilities, cryogenic equipment, operating personnel, utilities, sieves and sieve stack agitator, equipment cleanout, and product transport to the DSHS test site at Dugway Proving Ground, Utah. Pulva engineers specified the appropriate operating conditions to reduce rough-granulated DPE suit material to less than 1 mm at a process rate of 70 lb/hr.

EDS testing for micronization of granulated activated carbon was completed at Bematek Systems facilities in Beverly, Massachusetts. General Atomics provided PMACWA-supplied feed materials, guidelines for sampling, techniques for drying carbon slurry, a test plan, and operating procedures. Bematek provided the facilities, wet-milling process equipment, operating personnel, utilities, sieves, sieve stack agitator, equipment cleanout, and product transport to Dugway Proving Ground. Bematek engineers specified the appropriate operating conditions to reduce the size of spent granulated activated carbon to less than 0.5 mm at a process rate of 30 lb/hr.

In spite of a few minor operating problems, the DSHS tests appeared to have been successful. All materials, pallets, carbon, and DPE suit material were reduced to the size specified for feeding to the SCWO system; the metal removal devices appear to have performed well, and fugitive dust appears to have been controlled. The size reduction of the DPE suit material was of special interest because the technology for heavy polymeric composites is comparatively new.

The workup and EDS granulation testing demonstrated that DPE suit material could be successfully granulated to less than 10 mm in General Atomics' existing granulator at Dugway Proving Ground. The granulated DPE suit material was then shipped to the Pulva Corporation facilities, where it was successfully size-reduced in Pulva's cryogenic micronization system. Approximately 177 pounds of DPE suit material was micronized during six test runs.

ASSESSMENT OF PROCESS COMPONENT DESIGN

With the exception of the PMD machines and the SCWO system, all of the components in the unit operations of the GATS process are commercially available. In its assessment of the General Atomics' EDP for a GATS facility at Blue Grass, the committee addressed the following issues:

1. Does the available information identify any insurmountable technical obstacles to implementation of the unit operations, or is there a fatal flaw that would keep the facility from working properly?
2. Would additional research or testing at the smaller scale improve the full-scale plant's performance or safety?
3. Is the technology ready for implementation at the next step up—the full-scale plant.

The committee's assessment is based on the recognition that the Blue Grass disposal facility will be a one-of-a-kind, short-term (16-month duration) operation. The committee further recognizes that while additional research would always result in some process improvement, all work that involves agent poses risk and requires containment structures. Construction of larger containment structures for testing becomes equivalent to building the plant itself; hence, additional testing at a larger scale is not an option.

Disassembly of Munitions (Steps 1, 3, 4, 6)

Steps 1, 3, and 4. The Army has accumulated extensive experience with the PMD machinery, which has been used at two baseline incineration system disposal facilities. Although these machines experienced a number of operational problems in the past, they appear to have matured and are an acceptable method of separating energetic components from assembled chemical weapons. In the baseline system, however, PMD machines are used to prepare munitions for incineration. The GATS process (and other ACWA technologies being investigated) proposes using them in slightly different ways. The GATS PMD machine operation is the same as the baseline operation for removing fuzes, bursters, and miscellaneous parts. The components for transporting the disassembled parts to their destinations are different, but the changes can be accommodated by well established engineering methods such as modifying the number of cuts to a rocket.

The rocket disassembly machine (RDM) has been modified slightly to include washout of agent heels. While this is a new step, it is not critical for this technology, because the sheared rocket sections are fed to the PRH, where any remaining agent will be hydrolyzed.

In **Step 6**, the projectile agent removal system, cryofracturing is used to access the agent cavity of projectiles. In its report on EDS I for the Pueblo site, the committee had concerns about the safety of the procedure and the added process complexity of this step relative to traditional means of accessing the agent cavity, such as cutting (NRC, 2001a). One concern was the risk to maintenance personnel from exposure to agent that might accumulate in the cryobath when the bath was warmed up (e.g., during cleaning) or from agent being pumped back into the nitrogen storage tank when the bath was being emptied. Another concern stemmed from a general consensus that little was to be gained from introducing new technology when existing reverse-assembly technology seems to work satisfactorily.

In response to these concerns, General Atomics compiled reports of a series of tests conducted between 1982 and 1993, when 3,695 explosively configured agent-simulant-filled projectiles, rockets, and mines were opened by cryofracture (General Atomics, 1993). These reports demonstrated that cryofracture appears to be effective and that no leakage of agent simulant was noted. Furthermore, freezing the munition and agent would eliminate the effervescent foaming sometimes experienced when the agent cavity of mustard agent munitions was accessed (ACWA, 2001). Based on this information, the committee now believes that cryofracture is a sufficiently mature technology to offer a means of resolving this problem during disposal operations at Blue Grass.

Hydrolysis of Energetic Materials (Steps 5, 7, and 15)

Energetics Rotary Hydrolyzer

Step 5, the ERH, is unique to the GATS process. To the best of the committee's knowledge, a system such as this has never been used to hydrolyze solid energetic materials. Although the ERH should prove to be workable, a number of engineering issues will have to be addressed before it can be used in a full-scale disposal facility at Blue Grass. The issues still pending following EDS testing are discussed below.

First, the committee is concerned that for the processing of agent-contaminated energetic materials there would have to be verification that no detectable agent is present in the hydrolysate that leaves the ERH. If the complex chemical soup constituting the energetics hydrolysate interferes with analysis for agent, downstream operations (including the high-pressure SCWO) would have to be operated in a Category A area rather than the planned Category C area. This would increase the complexity of the SCWO system and pose additional operating and maintenance challenges.

Second, the committee believes ERH testing performed on a batch reactor or a single-chamber/flight reactor simulating one chamber of the cascading system proposed for the full-scale ERH can adequately simulate the kinetics of energetics hydrolysis, but it cannot simulate the mechanical behavior of the overall system. For example, if the energetic materials contain a granular component that does not hydrolyze, it could accumulate behind the flights of the initial chambers in a full-scale, cascading ERH. According to General Atomics, no such unhydrolyzed energetics were detected during Demo I and EDS testing, and the ERH design should ensure that loose solids (e.g., small metal parts or cuttings) move forward and do not accumulate (Spritzer, 2000). However, a gummy or sticky intermediate reaction product could cause problems that would necessitate a shutdown and manual removal. This situation (or any other maintenance inside the ERH) would be a challenge to maintenance personnel. Enough energetic material could even accumulate to create a hazardous condition. In summary, the committee believes that a full-scale ERH operated in a batch mode would likely offer satisfactory performance comparable to that of the test unit and would be preferable to an untested design employing a continuous-flow operation.

Aluminum Precipitation and Decontamination

Step 7 of the GATS process is the precipitation and filtration of aluminum compounds from the energetics hydrolysate. This step was tested during the EDS, and it appears to have performed well.

Step 15 is the 5X decontamination of the aluminum precipitate in either a separate furnace or an HDC.[4] The heating of a wet filter cake is, in the experience of committee members, very likely to release large quantities of particulate matter that must be captured and controlled. Similar problems are found in the thermal treatment of soil. Heating of soil causes the moisture in the soil to evaporate and to entrain substantial quantities of dust (Troxler et al., 1993). All thermal treatment systems require substantial air pollution control systems to control the dust (Ayen et al., 1994). It is expected that heating of the aluminum oxide would generate similarly large amounts of dust. Thus, the system design should provide for capturing and controlling the dust.

Processing and Treatment of Dunnage and Energetics Hydrolysate (Steps 2 and 9)

Step 2 of the GATS process is the separation, shredding, and grinding of the dunnage and other nonprocess waste until all solid material is reduced to granules less than 1 mm in size. These powdered solids are then mixed with energetics hydrolysate and other liquid wastes in a hydropulper to create a slurry to feed to the SCWO reactor.

It is critical that the feed to the hydropulper be reduced to a fine powder in the slurry feed stream to the SCWO reactor. Micronization of DPE suit material was successful during the EDS II testing; the desired particle sizes were produced in a single pass. The technology appears to have been validated.

Step 9 is the slurrying of the size-reduced dunnage and nonprocess waste with energetics hydrolysate in preparation for feeding to the SCWO reactor. As discussed above, the size reduction and slurrying equipment in the GATS design is commercially available and commonly used in many types of processing. The tests performed to date have shown that the materials can be shredded to the desired size and then slurried to a consistency that allows them to be processed in a SCWO reactor.

Agent Hydrolysis and Treatment of Metal Parts (Steps 8, 10, 13, and 16)

Projectile Rotary Hydrolyzer

In **Step 8** of the GATS process, accessed frozen agent is hydrolyzed and agent-contaminated metal parts from the cryofracturing step are decontaminated by hydrolysis in one of two PRHs. The PRH is similar in design to the ERH, and its application for hydrolysis of agent and munition body fragments from the cryofracturing process appears to be reasonable. In essence, the committee believes a PRH operated in a batch processing mode is an adequate mixing system to hydrolyze agent and wash the metal munition parts.

Step 10 is the completion of hydrolysis of the liquid agent drained from rockets or in the hydrolysate solution that drains from the PRH. The committee considers this system, which uses continuously stirred chemical reactors, to be a well-established technology for hydrolysis of chemical agent.

Treatment of Metal Parts from Rotary Hydrolyzers

Step 13 of the GATS process is the heating of metal parts from the ERH to 1,000°F (538°C) for at least 15 minutes by the HDC to decontaminate them to the 5X level. **Step 16** of the GATS process is the similar treatment of the metal parts from the PRH in a separate HDC. The two units are similar in design and function. The committee did not identify any difficulties in these steps.

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[4] The equipment that will be used had not been specified at the time this report was prepared.

Treatment of Hydrolysates and Dunnage by Supercritical Water Oxidation (Steps 11, 12, and 14)

Step 11 is treatment by SCWO of the energetics hydrolysate mixed with hydropulped dunnage. **Step 12** of the GATS process is treatment of the agent hydrolysate from the continuously stirred reactors by SCWO. The two steps are evaluated collectively.

The Blue Grass design incorporates changes based on over 4,900 hours of testing with waste and nearly 5,800 "hot hours" (either waste or only fuel) of operation of SCWO systems similar to that proposed for Blue Grass. Table 5-7 lists the major test campaigns for the GATS SCWO system that were conducted in support of this design and that were considered by the committee. This time of operation may be compared to the 16 months of operation encompassing a total of 11,311 SCWO reactor hours estimated to be necessary at Blue Grass (General Atomics, 2001a). As previously noted, the Demo I and EST tests showed that SCWO was an effective method for destroying the organic materials found in the agent and energetics hydrolysates and in the dunnage at Blue Grass, but that the SCWO reactor was subject to two basic problems: effective transport of solids and identification of suitable materials of construction.

The solids transport problem is caused by the use of sodium hydroxide for neutralization of agent and the presence of chlorine, sulfur, fluorine, or phosphorus in the agents. These materials lead to the formation of various inorganic compounds that must be flushed out of the SCWO system periodically. These inorganic compounds generally dissolve in liquid water (at subcritical conditions) but precipitate under the supercritical conditions of the SCWO reactor.[5] The behavior of the precipitates is complicated because some melt at SCWO conditions and others remain solid. Based on the test results, the committee concluded that periodic flushing of the system and other operating procedures that General Atomics developed during the testing should prevent accumulation of solids and plugging of the reactor during operation. However, the committee is still concerned that such a system has never been tested at larger scale.

The corrosion of the reactor walls (as exemplified in Table 5-8) further complicates the solids problem because the products of corrosion include metal salts or oxides that are insoluble in both subcritical and supercritical water. Also, the warheads of rockets, the end caps from shipping tubes, and other munition parts are made of aluminum, which forms a hydroxide in the caustic and exacerbates the solids handling problems. General Atomics has developed proprietary additives and routine flushing procedures that successfully manage the solids transport problems. Unfortunately, one of the additives increased the corrosion rate, especially that of titanium.

Based on the new information from the tests listed in Table 5-7, the committee reassessed the SCWO system's performance and operability. Ultrasonic testing after the tetrytol hydrolysate-dunnage and M28 hydrolysate-dunnage tests showed that the full-length titanium liner (leftmost liner of Figure 5-2) had excellent liner life with no visible or measurable degradation of the liner (<3 mils) after the extended-duration tests of these materials. The SCWO treatment of the micronized dunnage (with metal removed) slurried with energetics hydrolysate was of concern to the committee until it received the EDS test reports. The EDS tests showed that the SCWO system can successfully process slurries of organic materials, even if they contain small quantities of tramp metal. The SCWO testing of energetic hydrolysate slurried with shredded and micronized dunnage was successfully demonstrated. No liner changes appear to be necessary for the treatment of the energetics hydrolysate-dunnage slurry during the full SCWO campaign.

As discussed below, corrosion remains a problem with hydrolysates of mustard agent, GB, and VX. However, because the duration of both the VX and H disposal campaigns is short, no liner changes will be required. Only the SCWO treatment of GB will require liner change-outs. For the titanium liner configuration (i.e., the middle configuration in Figure 5-2), the two SCWO reactors treating GB hydrolysate will each require six liner changeouts. A Pt/Ti hybrid liner (the rightmost configuration in Figure 5-2) promises to reduce the number of change-outs to one per reactor during the GB disposal campaign.

The problem in the treatment of the two organophosphorus agents appears to be associated with the phosphorus they contain. The phosphorus present as a phosphate in the reactor appears to attack the protective TiO_2 film that coats the titanium metal. Once this film is removed, the exposed Ti metal is attacked by water and oxygen. Oxygen forms more TiO_2, which in turn is

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[5] Some of these salts would dissolve under "high pressure" supercritical conditions, but these conditions require pressures that are substantially above those of the SCWO reactors of the GATS design.

TABLE 5-8 Corrosion of Titanium Liners During GATS EDS Workup Tests with HD Hydrolysate

Date	Event	Fuel Hours	Feed Hours	Total Hot Hours
10/4/00	Start Ti rolled-sheet liner #1 (Grade 7, 0.030 in. thick).	0	0	0
10/5/00	Small hole in liner about 2 in. below nozzle tip. Two patches each several inches square with wall thinning of about 10 mils.	10	23	33
11/1/00	Start Ti rolled-sheet liner #2 (Grade 7, 0.030 in. thick).	0	0	0
11/2/00	Ti pit depth ~10 mil. General thickness loss <~1 mil.	6	33	39
11/12/00	~2,000 small pits counted with maximum depth ~10 mil. Switch to new liner for 5% HD hydrolysate simulant tests.	16	145	161
11/14/00	Start repaired Ti rolled-sheet liner #1.	10	23	33
11/17/00	Switch to testing of Grade 2 pipe liners.	17	85	102
11/18/00	Start Ti pipe liner #1 (Grade 2, 0.110 in. thick).	0	0	0
11/19/00	Pitting noted 16 in. below nozzle tip. Maximum depth ~10 mil.	2	20	22
11/22/00	Pitting from 6 to 18 in. below nozzle tip. Maximum depth ~20 mil. Maximum general corrosion ~5 mil.	7	47	54
11/30/00	Maximum general corrosion ~50 mil, primarily in top 3.5 in. below the nozzle tip. Maximum pit depth ~20 mil.	10	105	115
12/1/00	Start inverted Ti pipe liner #1 (Grade 2, 0.110 in. thick).	10	105	115
12/5/00	Maximum corrosion ~85 mil, a bit less than 1 mil/hr. Corrosion primarily in top 3.5 in. below the nozzle tip.	21	200	221
12/12/00	Ti pipe liner #1 returned to original orientation.	27	237	264
12/17/00	Ti pipe liner #1 broken during removal from Hastelloy sleeve.	38	294	332

SOURCE: Adapted from General Atomics (2001a).

attacked by phosphate. Water forms both TiO_2 and TiH_2. In fact the most depleted part of the corroded liner was found to have been completely converted to titanium hydride (General Atomics, 2001a).

The titanium hydride does not appear to cause any problems. It seems to hold up well, and the 20 percent swelling caused by the hydride formation seems to lock the titanium into place. According to General Atomics, the rate of phosphorus attack is reasonably linear (General Atomics, 2001f). Thus, a thicker liner will have a longer life. For example, the values for liner life (shown by hours per reactor) in Table 5-9 are computed by assuming that a thicker (0.5-inch-thick) liner will be destroyed at the same rate as was the thinner liner during the tests. The committee believes that this assumption is reasonable in the absence of firm data; however, the assumption does introduce a level of uncertainty about the actual life of the liners for the disposal campaigns for GB munitions at Blue Grass.

Because there are relatively few VX munitions at Blue Grass, total operation with VX hydrolysate shown in Table 5-9 is projected to be only 216 hours split between two reactors, i.e., 108 hours per reactor. Even under the worst corrosion conditions, the VX hydrolysate disposal campaign will take less time than the liner lifetime experienced (with the much thinner liner) during EDS testing.

The GB disposal campaign will require 1,522 reactor hours split between two reactors. This will require six liner changes per reactor, or twelve liner changes over the total 16-month operating life of the plant. The number of reactor liner changes shown in Table 5-9 assumes that each reactor's life is only 761 hours, divided by seven liners, or about 110 hours. In the opinion of the committee, this still constitutes a very large amount of maintenance This liner life, which is used by General Atomics as the basis for its design, is shorter than the 120-hour projected liner life that was based on the EDS test results. This shorter projected life and the extra SCWO reactor provide additional margins of safety in the overall design. The General Atomics EDP for Blue Grass addressed the uncertainty surrounding SCWO liner life in two ways. First, the EDP design includes an extra SCWO system that can be put online if the number of liner changes is larger or the time required to perform liner changes longer than anticipated. Second, extra time has been built into the operating schedule to compensate for the uncertainties.

The 500-hour tests were conducted on a SCWO reactor with an internal diameter of approximately 4 inches. The full-scale system will have an internal

TABLE 5-9 Liner Lifetime and Replacement Calculations for Blue Grass Facility SCWO Reactors

Feed	Liner Material	Total Time in Reactor (hr)	No. of Reactors	Time per Reactor (hr)	Maximum Corrosion Rate	Mils Corroded	No. of Liner Change-outs[a]
GB hydrolysate	Ti/Ti	1,522	2	761	2.5 mil/hr	1,903	6 (per reactor)
GB hydrolysate improvement[b]	Pt/Ti	1,522	2	761	<0.5 mil/hr (for Ti)	380	1 (per reactor)
Energetics from GB munitions/dunnage	Ti	6,943	3	2,164	<1 mil/100 hr	22	0
VX hydrolysate	C-276	216	2	108	0.4 mil/hr	43	0
Energetics from VX munitions/dunnage	Ti	2,399	3	800	<1 mil/100 hr	8	0
H	Ti/Ti	229	2	115	0.5 mil/hr	58	0
Energetics from H munitions/dunnage	Ti	116	3	39	<1 mil/100 hr	0	0

[a] Blue Grass liner change-outs—all titanium and C-276 liners 0.5-in. initial wall thickness, change-out when 0.2 in. remaining.

[b] Platinum (0.030-in. thick) wear liner has shown good performance after 128 hr of testing with GB and VX hydrolysate simulants. Pt corrosion is negligible and Ti corrosion rate is reduced to <0.5 mil/hr, resulting in one Ti liner change per reactor with GB hydrolysate at Blue Grass.

SOURCE: Adapted from General Atomics (2001a).

diameter of approximately 18 inches and will be 18 feet long. Because this is a substantial increase in size, the impact on the corrosion rate, plugging by precipitates and other solids, and other performance factors cannot be reliably assessed until the scaled-up SCWO unit is operated. In conclusion, the next step in the application of a SCWO system at Blue Grass is the actual construction and testing of one full-scale unit under actual conditions. In the committee's opinion, the information that additional testing on a smaller scale would provide would only be of marginal value for the construction and operation of the full-scale system.

Water Recovery and Salt Disposal

In **Step 14** of the GATS process, the brine from the SCWO reactors is concentrated for water recovery and generating a solid salt cake for off-site disposal. Although the EDP did not include specific design parameters for this unit operation, it is existing technology, and assuming that the SCWO reactor produces an effluent with the very low organic content called for in design specifications, appropriate concentration/crystallization equipment is commercially available. The committee notes that other chemical weapons demilitarization facilities have eliminated this processing step and suggests that similar changes be evaluated for the Blue Grass facility.

ASSESSMENT OF INTEGRATION ISSUES

The issues surrounding integration of General Atomics' technology package at the current stage of design were addressed in the ACW II Committee's earlier report for EDS I, which addressed ACWA technology options for disposal of the Pueblo stockpile (NRC, 2001a). The committee's analysis is equally applicable to a GATS facility at Blue Grass and is presented again below with some updating and adaptation for Blue Grass.

Component Integration

Destruction of the Blue Grass stockpile in time to meet the CWC treaty deadline will require that the availability and throughput of each processing step, along with redundant process trains and sufficient buffer storage capacity between individual processing steps as necessary, result in the specified destruction rate. General Atomics has conducted a detailed throughput analysis that takes into account intermediate storage capacity. On the basis of this analysis, General Atomics has verified that planned throughput rates can be achieved. Proper training of plant operating and maintenance personnel is important, because their effectiveness contributes to process availability.

General Atomics has designed the GATS process and sized the equipment to process the Blue Grass stockpile in 16 months (General Atomics, 2001a). The output rate from the reverse-assembly PMD system determines the size and number of units for all downstream process equipment. Because the GATS PMD equipment is similar to the equipment used in the baseline incineration system, the operating experience from baseline facilities led General Atomics to conclude that a throughput rate of 50 rounds per hour per machine would be attainable. General Atomics has estimated that the long-term average capacity for the GATS design for Blue Grass (actual throughput per year/maximum theoretical throughput per year) is 38 percent. To meet this availability, two PMD machines are required to handle the Blue Grass stockpile. The size and number of the rest of the General Atomics process equipment is designed to match the throughput of the PMD operation. For example, two SCWO reactors are used to treat the downstream agent hydrolysate, and two more SCWO units are used to treat the micronized dunnage and energetics hydrolysate waste stream.

Integrating the individual processing steps requires effective process monitoring and control to ensure that appropriate materials are fed to each processing step and that all materials discharged from the plant meet all safety and environmental specifications. Monitoring and control of the integrated facility will be based primarily on the methods used in the baseline system. The overall monitoring and control system consists of the basic process control system (BPCS), the emergency shutdown system (ESS), and PLCs (programmable logic controllers) for individual equipment units. The BPCS is composed of microprocessor-based controllers for monitoring and control. The ESS is a dedicated safety system of PLCs or microprocessor-based controllers that provide protective logic and shutdown capability. The means of controlling machines throughout a GATS facility are similar to those used for the baseline system machines (i.e., sequence-enabled functions with position switches).

Most of the monitoring instruments specified in the GATS design package are simple and reliable, having been used extensively in the chemical industry. Control valves and monitors for temperature and pressure, as well as distributed control systems and PLCs, have also been widely used in industry.

Process Operability

The operability of the SCWO reactors remains a significant issue. The reactors' operating conditions are set to balance competing conditions for minimizing plugging by salts and minimizing liner corrosion. That is, the conditions for good salt transport (and hence minimal plugging) are also the conditions that cause maximum corrosion. Conversely, operating conditions with minimum corrosion are conducive to the precipitation of salts, which can cause plugging. General Atomics has approached this problem by (1) the use of a proprietary additive to improve salt transport and (2) designing the SCWO reactors with a removable sacrificial liner that would be replaced at regularly scheduled intervals. This combination, along with careful monitoring and control of temperature, pressure, additive feed rates, and other operating conditions, reduces the severity of the salt plugging and corrosion problems, but not sufficiently. The committee believes the SCWO system will still be very difficult to operate, especially at full scale (see also the section on maintenance issues, below).

Monitoring and Control Strategy

As discussed in previous NRC reports, except for the monitoring of corrosion and salt plugging, discussed above, the GATS process does not require any unusual monitoring or control systems (NRC, 1999, 2000a). The process control strategies consist of straightforward monitoring of pressure, flow rate, and temperature by well-established methods and equipment.

General Atomics believes that monitoring the turbidity resulting from suspended titanium dioxide in the reactor effluent will effectively monitor corrosion rates. Monitoring the turbidity of the effluent gives a good indication of the instantaneous corrosion rate, which can be used to ensure that operating conditions remain within the desired range. However, the decision to shut the process down for liner replacement would be facilitated if the extent of corrosion could also be monitored. This could be done by adapting one of various probe designs available commercially or developed in previous SCWO studies (Macdonald and Kriksunov, 2001). Another simple method would be to measure the electrical conductivity of the thermocouple well.

Maintenance Issues

The EDS testing clearly showed that successful operation of the SCWO system requires an aggressive, proactive maintenance program to replace (1) the thermocouple well and (2) the titanium liner after every 120 hours or so of operation.

Replacement of internal components of the SCWO reactor is a time-consuming, elaborate procedure that involves cooling the system, flushing with clean water, manually removing the pressure head from the reactor, manually removing the liner, inverting the liner or replacing it with a new liner, reassembling the reactor, and restarting the system. General Atomics has performed this procedure more than 100 times on reactors with test-size (3- to 4-inch ID) liners during the EDS and other SCWO test programs (Hong, 2001). During the EDS tests, the shutdown/start-up procedure required an average of 7 hours (3 to 11 hours) to complete.

However, maintenance has been performed only on comparatively small test-size SCWO reactors. The SCWO reactors proposed for the Blue Grass facility are approximately 18 inches in diameter and 18 feet long. Thus, the length of the liner to be removed is considerably greater than the length of liners removed to date. The head at the top of an 18-inch reactor will not only be larger, but it will also have to be considerably thicker to withstand more than 20 times the force from internal pressure. Thus, the head on the full-scale reactor will be heavier and bulkier and will have more and larger bolts to be removed and replaced than the small SCWO test reactors. It will also have a larger sealing surface that will have to be set and pressure tested. In the committee's experience, proper pressure sealing of equipment is very time consuming. Consequently, the time and effort required to change the liner are likely to be much greater than those required in the EDS and Demo I tests.

Process Safety

The ACW I Committee concluded in its original report that there were "no unusual or intractable process safety problems" associated with the GATS process (NRC, 1999). However, in a subsequent evaluation of the Demo I test results, some aspects of process design for which safety needs to be addressed were identified (NRC, 2000a). General Atomics also acknowledged these safety design requirements in its report on the Demo I test results (General Atomics, 1999a):

- Modify the design to incorporate equipment for removing precipitated aluminum compounds to minimize aluminum-caused salt plugging and associated maintenance. (For Blue Grass, aluminum removal requirements are minimal because the only aluminum in the munitions is expected to be in fuze assemblies.)
- Control volatile organic vapors generated in the ERH to prevent the accumulation of explosive mixtures in the ERH offgas system and to minimize the maintenance requirements for removing condensed organics from fugitive emissions entering the ERH explosion containment cubicle.
- Incorporate safety features to preclude dust explosions in the DSHS.

All of these concerns have been addressed in the EDS design package to the extent possible at this design stage (General Atomics, 2001a).

During the information-gathering phase of this report, the committee learned of an occurrence on December 2, 2000, involving backflow of fuel into the liquid oxygen feed line for the General Atomics SCWO reactor being tested in Corpus Christi, Texas, for treatment of VX hydrolysate at the Newport site (PMACWA, 2000). This backflow caused an overpressure (possibly in excess of 5,000 psig) and permanent expansion of part of the liquid oxygen feed line. The overpressure is believed to have come from the oxidation of fuel in the oxygen line. An earlier occurrence (July 14, 2000) involving the oxygen feed line caused a release of oxygen through the relief valve, which caused a grass fire from melting metal components of the relief valve.[6] The fire was attributed to removal of the high-pressure and high-high-pressure shutdowns from the pump circuit control and the use of stainless steel rather than Monel metal for the pressure relief valve. Based on these incidents, the committee inferred that the General Atomics SCWO system would continue to be vulnerable to fires if pure oxygen and nitrogen were used to produce synthetic air for the SCWO reactors. The committee notes that the final preliminary hazards analysis (PHA) in the EDP for Blue Grass recommends redundant oxygen sensors to protect against high oxygen levels in the synthetic air mixture (General Atomics, 2001a). Use of compressed air, however, would completely eliminate this hazard, and General Atomics notes in its summary of the PHA that it may consider using compressed air to reduce costs.

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[6] Bernard Bindel, safety engineer, PMCD, personal communication on March 26, 2001.

As a part of the EDS design package, General Atomics prepared a PHA in accordance with MIL-STD-882C (DoD, 2000). The committee's review of the final PHA and the Hazard Tracking Recommendations and Resolution (HTRR) log, indicates that the PHA and the HTRR make good use of engineering changes to reduce risk and rely primarily on procedural or administrative controls where the actions of operator and maintenance personnel are a threat. The PHA considered hazards in three categories: agent release, personnel injury, and system damage. This multiattribute approach provides increased assurance of achieving safety goals for preventing agent release and protecting personnel because it also suggests ways to make the system operate more reliably, thereby requiring less repair and maintenance. The final EDP PHA for Blue Grass confirms some committee concerns and recommends measures to address them. For example, a cover is recommended for the cryobath, with venting of vapors through a CATOX system when evaporating liquid nitrogen for purposes of bath cleaning and repair. Similarly, recommendations in the PHA include added instrumentation and equipment redundancy to reduce the chance that hydrogen gas will accumulate in the ERH and its vent gas.

The PHA appears to have been conducted in a satisfactory manner at this stage of design. Of course, it will have to be updated as the GATS design progresses to completion for use in construction.

Worker Health and Safety

The conclusions regarding worker health and safety in the ACW I Committee's original and supplemental reports are still valid (NRC, 1999, 2000a). The primary hazardous materials used during the destruction of agent and energetics are sodium hydroxide, liquid and gaseous oxygen, liquid and gaseous nitrogen, and methane (natural gas) for boiler fuel. Sodium hydroxide will be delivered in concentrated liquid form (50 weight percent) and diluted with water to produce the required weaker solutions. This corrosive caustic is handled safely in similar quantities and concentrations throughout the chemical industry and should not be unusually hazardous in the GATS process. Liquid and gaseous oxygen, liquid and gaseous nitrogen, and methane are also handled routinely and safely in many industries and do not present an unusual hazard to workers.

The committee's review of the PHA reveals extensive recommendations for assuring worker safety. This result supports the committee's earlier conclusion that an adequate level of worker safety would be provided for in the design and the procedures that would be used for facility construction and operation.

Public Safety

Accidental releases of agent or other regulated substances to the atmosphere or the groundwater system are extremely unlikely. Caustic scrubbing and activated carbon filtration are used on all gaseous process streams. Based on experience with the baseline facilities, these measures should provide a reasonable level of safety. Hold-test-release systems are not provided for gaseous effluents, but the scrubbing and filtration scheme, combined with the standard automatic continuous air monitoring system (ACAMS) monitors used at baseline facilities, should provide adequate protection for all gaseous process effluents. The facility HVAC design is similar to the design at existing baseline facilities, where air flows from clean areas to potentially contaminated areas and then through high-efficiency particulate air (HEPA) and activated carbon filters before release to the atmosphere.

The primary cause of a release of agent or other regulated substances would be the explosion of a munition or the rupture of a pipe or vessel, but the likelihood of such events should be extremely small. This conclusion is based on the committee's review of the General Atomics EDP for Blue Grass and the understanding that the PMCD will require a comprehensive quantitative risk assessment (QRA) for the final facility design to ensure acceptable levels of risk. A QRA, which is much more detailed than the PHA performed at this stage of design, is a risk management tool during actual operation of the facility and forms a basis for evaluating proposed changes in design or operation in accordance with Chemical Stockpile Disposal Program (CSDP) risk-management policies and procedures (PMCD, 1996, 1997).

QRAs are typically developed in parallel with the completion of the facility design and construction. However, because design-based solutions to high-risk hazards can be implemented more easily during the design stage than during the construction stage, early implementation of the QRA process can be advantageous. The later in the design that a QRA is prepared, the greater the tendency to rely on procedural and administrative solutions, which often complicate operations and are less effective than design modifications.

TABLE 5-10 500-hr Test Effluent Quality

	HD	TD	MD	GB	VX (200 hr)
TOC (liquid) (ppm)	≤1.3	<1	≤1.9	<2.6	<1
TCLP metals (liquid) (ppm)	Meets criteria except for 5 ppm Cr in feed	Meets criteria	Meets criteria	Cr = 0.7, Ti = 0.6 (?)	Ni = 50, Cr = 13
CO (gas) (ppm)	<10	<10	<10	<10	<10
NO_x/ SO_x (gas) (ppm)	<1/<1	<1/<1	<1/<1	5.2/0.2	Not analyzed
Dioxins/furans	200 times below EPA MACT standard	Not analyzed	72 times below EPA MACT standard	Not analyzed	Not analyzed

NOTE: HD, HD hydrolysate; TD, tetrytol hydrolysate slurried with shredded and micronized dunnage (palettes, DPE suits, gloves, etc.); MD, M-28 rocket propellant and burster hydrolysates slurried with shredded and micronized dunnage that has had the hydrolyzed aluminum removed by filtration; GB, GB hydrolysate; VX, VX hydrolysate.

SOURCE: Adapted from General Atomics (2001a).

Human Health and the Environment

The environmental impact of the proposed GATS process appears to be minimal. All handling and processing of agent will be conducted indoors in sealed rooms that are vented through HEPA and carbon filters. Liquid and solid waste streams will be relatively small and manageable and will be subjected to hold-test-release procedures.

Effluent Characterization

The liquid effluent, which consists of water from the evaporator/crystallizer used to produce the solid filter cake produced by the brine-recovery operation, should not pose a significant hazard to human health or to the environment. While the evaporator/crystallizer system has not been tested yet, the composition of the water and solid filter cake can be readily determined from an analysis of the SCWO liquid effluent. As shown in Table 5-10, the liquid effluent is essentially free of organics. The source of the chromium and nickel that were found in some of the effluents is generally believed to be corrosion products from the SCWO reactor components. These elevated levels of metals indicate that the solid filter cake will need to be treated (e.g., by stabilization) prior to disposal in a hazardous waste landfill.[7]

Much of the water recovered from the evaporator/crystallizer is recycled for use in the process.

As also shown in Table 5-10, the gaseous effluents contain very low concentrations of hazardous constituents. Furthermore, the flow rates of the gaseous effluents from the SCWO reactor are very low. Thus, the mass emissions of hazardous air pollutants (concentration times gas flow rate) from the SCWO reactor are extremely low. These emissions are further cleaned by passing them through carbon filters. This combination should ensure that the total impact on health and the environment of the SCWO system is minimal. This must be confirmed through formal testing, including comprehensive, full analytical scans that indicate the quantities of a large number of compounds of environmental concern,[8] and by fully developed site-specific health and ecological risk assessments for the process.

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[7] There is a common misconception that the absence of hazardous constituents in a waste makes it nonhazardous. In fact, any waste that is produced by a hazardous waste treatment process such as this is legally hazardous unless it goes through a lengthy legal "delisting" process. The effluent from the Newport Chemical Weapons Destruction Facility's SCWO system has been delisted by the Indiana Department of Environmental Management.

[8] EPA analyses are done with 8000 Series methods, especially those using gas chromatography/mass spectrometry scans (e.g., methods 8260B, "VOCs by GC/MS," and 8270C, "Semi-VOCs by GC/MS").

Completeness of Effluent Characterization

The liquid and solid effluents are well characterized. As the ACW I Committee noted in its original and supplemental reports, the gaseous process emissions will have to be characterized for health risk assessments and environmental risk assessments required by EPA guidelines (NRC, 1999, 2000a). These results, along with the results of analyses of metals emissions (including chromium VI), can be used to assess the environmental impact of a facility through accepted risk-assessment methods (EPA, 1998).

Effluent Management Strategy

The proposed strategy appears to be reasonable and should protect public health and the environment.

Off-site Disposal Options

Dunnage. Experience at the baseline system facilities at Johnston Atoll and Tooele, Utah, has shown that only a tiny fraction of dunnage is contaminated with agent. Uncontaminated dunnage from these two stockpile locations is being disposed of off-site by commercial waste management facilities (McCloskey, 1999; U.S. Army, 1998). Off-site management of uncontaminated dunnage is also planned for both the Newport and Aberdeen sites. Off-site management of dunnage from Blue Grass would greatly reduce the on-site processing requirements and greatly simplify process integration by eliminating the need for size reduction and SCWO treatment of the dunnage. Off-site disposal of other agent-free waste streams, such as hydrolysates, may be possible, thereby reducing the number or size of treatment steps.

Brines. Brines produced from air pollution control processes at the baseline facility at Tooele, Utah, are currently being shipped off-site for disposal by commercial waste management facilities.

Environmental Compliance and Permitting

The combination of technologies in the General Atomics technology package is not expected to lead to problems with environmental compliance or permitting. All process waste streams except the SCWO offgas will be evaluated prior to release to confirm that they are either free of regulated substances or that these substances are at acceptable concentrations. The SCWO offgas will be released to the environment via the plant HVAC HEPA and activated carbon filters.

The committee considered the small amounts of PCBs that are suspected to be present in rocket motors and concluded that these amounts are far too small to pose an environmental concern. No data on PCBs were provided in any of the tests that the committee evaluated. Any small amounts of PCBs that may be present from rockets treated would end up in one of the solid waste streams and be at a concentration well below regulatory limits.

ASSESSMENT OF OVERARCHING TECHNICAL ISSUES

Overall Engineering Design Package

The EDS test results with the PRH, the ERH, and their HDCs appear to warrant proceeding with these GATS components. The method for treating dunnage and energetics also has been reasonably tested. Corrosion of the SCWO reactor liner and other internal components is high but manageable. All issues about whether the GATS process would be able to destroy the munitions stored at Blue Grass within a reasonable period of time appear to have been resolved. In conclusion,

- The GATS process meets the whole-plant availability requirement of 38 percent.
- Readily available, inexpensive titanium grade 2 is a suitable SCWO reactor liner material for TD and MD, and with the addition of a titanium wear liner, as shown in Figure 5-2, it is suitable for processing GB and HD.
- Readily available Hastelloy C-276 is a suitable SCWO reactor liner material for VX.
- Improvements have been identified and demonstrated that may afford a lower corrosion rate for SCWO liners and improve effluent quality for all chemical agents, including GB (see the two rightmost liner configurations in Figure 5-2).
- SCWO system pressure and temperature control are well maintained.
- Suitable salt transport has been achieved for all feeds to SCWO system reactors; a salt flush is required not more than once a day.

- The SCWO system achieves high organic destruction efficiency.
- The SCWO system is safe.

Steps Required Before Implementation

The following additional testing/piloting activities should be carried out in parallel with facility design, construction, and permitting activities:

- Consider a design change to replace manufactured air (made from liquid oxygen and nitrogen sources) with high-pressure air compressors for the SCWO oxidants.
- Consider a separate feed conveyor for GB and VX projectiles that do not contain energetics. The conveyor would bypass the PMD area and feed projectiles directly into the projectile agent removal system.

Additional Testing/Piloting

The following additional testing/piloting activities should be considered in parallel with the facility design, construction, and permitting activities. None of these suggestions are critical to the success of the present operation:

- Develop an agent sampling and analysis protocol for trace amounts of agent in energetics hydrolysate.
- Develop a pilot-scale ERH, and demonstrate and verify the materials flow.
- Develop and test a full-scale SCWO reactor system to verify performance parameters, including preventive maintenance operations.
- Develop and test other SCWO liners to further reduce liner preventive maintenance requirements.

The initial evaluation of the GATS process by the ACW I Committee identified the following steps required for implementation (NRC, 1999). These steps are reevaluated below.

> Conduct tests of the cryofracture process to ascertain if it provides better access to the agent cavity in projectiles and mortars than the baseline disassembly process.

The cryofracture data that were provided resolve this issue. Cryofracture also prevents the effervescent foaming of agent.

> Sample and analyze air emissions from the demonstration system. The air emissions will have to be measured to a level of detail and accuracy that can be used for HRAS [health risk assessments] and environmental risk assessments required by EPA.

Subsequent testing that included extensive analyses of air emissions revealed no obvious concerns. The tests consistently showed extremely low levels of organic contaminants in all SCWO effluents. Final determinations of safety and environmental acceptability can only be made through a formal risk assessment process that will occur as part of the permitting program.

> Verify that energetic materials encased in metal (e.g., rocket or other munitions fragments) will be hydrolyzed.

The EDS testing appears to demonstrate that energetic materials encased in metal can be hydrolyzed successfully, although some questions remain about the completeness of the hydrolysis. During the Demo I testing, fuzes were observed to have “popped” on the HDC, indicating that they may not have been completely hydrolyzed. However, the level of popping appears to have been within the design specifications of the HDC for such events.

> Ascertain how well the SCWO process can handle high-solids materials (shredded dunnage).

SCWO testing was conducted using energetic hydrolysate slurried with shredded and micronized dunnage. The tests showed very low corrosion rates with an inexpensive titanium liner and excellent organic destruction efficiency. The committee concluded that these tests were successful.

> Determine erosion and corrosion behavior of the components of the SCWO system.

The EDS testing and other testing cited in this report has provided design information on the erosion and corrosion of SCWO components. The data, which appear to be reliable, show that corrosion rates are acceptable for the energetics-dunnage slurries, which are the largest streams to be treated at Blue Grass. The results do confirm component corrosion when processing GB, VX, and HD hydrolysate. As discussed earlier under maintenance issues, the corrosion appears to present a significant but not unmanageable maintenance burden for operators and maintenance personnel. Table 5-11 summarizes the status of recent developments concerning the processing of various waste streams by the SCWO reactor.

TABLE 5-11 Status of Recent Developments on Treatment of Various Waste Streams by SCWO

Waste Stream	Problem	Approach	Recent Developments
HD hydrolysate	Platinum corrosion and mechanical instability	Use of a titanium reference liner with extended change-out frequency for a given full-scale reactor every 500 hr or longer	Titanium liner showed corrosion rates compatible with 500 hr of operational life. Other test data indicated that a platinum liner (per Configuration 5 in Table 5-2) would be sufficiently corrosion resistant to not need change-out during HD hydrolysate disposal at Blue Grass.
GB and VX hydrolysate	Titanium corrosion and embrittlement	For GB: Extend titanium reference liner change-out frequency using multiple reactors or implement Pt wear liner product improvement For VX: Adopt Hastelloy C-276 reference liner or implement wear liner product improvement	Problem is being managed through an aggressive preventive maintenance program.
Tetrytol hydrolysate/ dunnage slurry	Operational difficulties encountered while processing mixed dunnage containing carbon	Processing the carbon separately with internal baffles in SCWO reactor to ensure sufficient residence time; alternatively, decontaminating carbon to 5X using one of several processes (e.g., heated discharge conveyor or Al filter cake dryer)	153 hr of successful testing processing slurried carbon separately using modified SCWO reactor; concluded on 12/2/01.
M22 propellant hydrolysate/ dunnage slurry	Aluminum precipitate caused problems in SCWO reactor and downstream components	Implementation of improved filtration technique and verification of correct SCWO feed composition by sampling and analysis	Treatment of hydrolysate slurry, including filtration of aluminum compounds, was successfully tested in MD runs (see also Table 5-1).

FINDINGS AND RECOMMENDATIONS

Findings

Finding (Blue Grass) GA-1. The GATS process appears to have reached a level of maturity sufficient for construction of a full-scale facility at Blue Grass, notwithstanding the issues raised in the subsequent findings.

The testing that has been conducted has shown that the General Atomics SCWO system is a high-maintenance operation; however, the level of maintenance that is required for the application of the SCWO system in particular, and the GATS process in general, in a Blue Grass facility is not beyond the ability of well-trained operators and maintenance personnel.

Finding (Blue Grass) GA-2. In the committee's opinion, the number of SCWO reactor liner changes required to process the stockpile at Blue Grass, while high, is manageable.

Finding (Blue Grass) GA-3. The committee still has some concern about possible problems when scaling up from the existing GATS SCWO reactor to the full-scale design.

Finding (Blue Grass) GA-4. Although the General Atomics cryofracture process was not demonstrated during the ACWA program, the committee believes the technology to be sufficiently mature for implementation in a Blue Grass facility.

This finding is based on the results of the tests conducted between 1982 and 1993, during which time 3,695 explosively configured, agent-simulant-filled projectiles, rockets, and mines were opened by cryofracture.

Finding (Blue Grass) GA-5. The GATS process for handling energetics within the ERH will result in the mixing of a lead-containing propellant (M28) with tetrytol when rockets with tetrytol bursters are treated.

This combination might lead to the formation of lead picrate.

Finding (Blue Grass) GA-6. The ERH was tested in a batch mode; however, the design proposed for both the ERH and PRH for Blue Grass is operated in a continuous mode. The resulting equipment is an ERH 50 feet long and a PRH 40 feet long. In the opinion of the committee, this equipment may prove difficult to maintain.

Recommendations

Recommendation (Blue Grass) GA-1. The hydrolysis streams for the processing of propellant and burster materials must be kept separate until the formation of lead picrate has been ruled out.

Recommendation (Blue Grass) GA-2. The technology provider should consider that the ERH and PRH units for a full-scale GATS process at Blue Grass be batch units similar in design to those tested.

6

General Findings

In this chapter, the committee presents its general findings on the three alternative technology packages that have been proposed for Blue Grass. The general findings should be considered with the caveat that various ACWA EDS tests were not completed in time for the committee to review the final results and that some process steps remain to be demonstrated on a pilot scale. Also, following its statement of task, the committee limited its evaluations to separate assessments of the total solutions proposed by AEA, Eco Logic, and General Atomics for destroying the assembled chemical weapons at Blue Grass Army Depot. That is, the committee has neither compared the proposed alternatives with one another or compared them with the Army's baseline incineration system.

General Finding 1. The reverse assembly of munitions, followed by water or caustic hydrolysis of nerve or mustard agents and associated energetic materials, is a mature, safe, and effective method for initial treatment of the chemical weapons stored at Blue Grass Army Depot. It is ready for immediate implementation for the neutralization of energetics and agents. However, the resulting hazardous streams must be treated further before they are released to the environment.

This finding is based on the test results presented earlier in this report for the EDS hydrolysis of energetic materials and on the effectiveness of the modified baseline reverse assembly and agent hydrolysis processes discussed in this and previous NRC reports (NRC, 1999, 2001a). Because hydrolysis destroys agents and active energetics, the resulting hydrolysates pose a lesser hazard than the assembled chemical weapons.[1] This finding is independent of any subsequent secondary treatment steps that will be necessary to render the hydrolysates environmentally acceptable. That is, agent and energetics could be hydrolyzed even before the secondary treatment process is selected. However, further treatment is necessary to reduce hazardous waste concentrations to a level sufficiently low to allow for final disposal. Additional treatments will also be required for dunnage and the metals parts generated in the reverse assembly process.

General Finding (Blue Grass) 2. Several of the unit operations of the technology packages have process streams that are not unique to the chemical weapons stockpile. These streams, which include dunnage, brines from water recovery, and hydrolysates, could potentially be treated at existing off-site treatment, storage, and disposal facilities. Off-site treatment of these wastes would simplify the overall technology and facilitate process integration by eliminating the need for

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[1] All tests of agent destruction were performed with field-grade chemical agents produced at approximately the same time as the agents loaded into the chemical weapons. Thus, any complications arising from stabilizers, other additives, or degradation products were accounted for during testing.

further development of individual process steps in the secondary treatment processes and their integration into a whole system. It might also simplify design requirements to meet safety concerns.

All of the process streams that could be treated offsite have compositions similar to waste streams routinely treated by commercial industrial waste treatment facilities. Thus, they could be transported by standard commercial conveyance to commercial facilities that are appropriately permitted to receive the waste.

General Finding (Blue Grass) 3. Based on the results of the PMACWA-sponsored test program on the hydrolysis of energetic materials, the committee believes that hydrolysis can be used safely, effectively, and on the scale necessary for the destruction of the energetic materials contained in assembled chemical weapons at Blue Grass, provided that lead-containing propellants are not processed with tetrytol until the possibility of lead picrate formation can be eliminated.

The test plan that was executed at HAAP demonstrated that the hydrolysis process for the energetic materials of interest successfully destroyed more than 99.7 percent of the energetics treated. Over 9,500 pounds of energetic materials were destroyed while encountering only the types of problems normally expected during the implementation of a new facility and process. The suite of tests performed at Los Alamos National Laboratory (LANL) and at HAAP covered a range of reaction temperatures and caustic concentrations. Under all these conditions, the destruction of energetics was carried out safely and effectively.

Effective destruction of energetic materials during the test program is interpreted by the committee to mean that the products resulting from destruction no longer pose an explosion or detonation hazard. The committee notes, however, that the hydrolysate produced and the gaseous decomposition products are not benign. Gases such as ammonia and oxides of nitrogen are evolved, and cyanide or other toxic substances may be present in the hydrolysate. However, a variety of conventional methods for treating the offgases are available.

The fate of lead during energetics hydrolysis in the presence of picrate is not understood. Until more information becomes available on the speciation of lead and the solubility of lead picrate under the conditions of hydrolysis, the possibility of solid lead picrate formation cannot be dismissed. As a precautionary measure, lead-based propellants should be processed separately from tetryl and tetrytol, eliminating the possibility of lead picrate formation.

General Finding (Blue Grass) 4. Based on the results of EDS testing and the engineering design package, the committee believes that the AEA SILVER II™ total solution technology package could eventually destroy the assembled chemical weapons stored at the Blue Grass Army Depot. However, the immaturity of a significant number of processes in this electrochemical destruction technology could threaten PMACWA's achievement of its objectives for weapons destruction. Considerable additional development and demonstration must be completed before this technology can proceed to a full-scale facility. There is also concern that the ever-increasing complexity and frequent configuration changes that have occurred in the AEA technology package to date are indicative of the immaturity of the process. The approach employed by AEA has been to solve problems as they arise by incorporating additional steps that are modifications of previously demonstrated technologies. The technology provider infers the success of these new steps based on previous demonstrations in other applications but has not generated the data to show that the modifications will result in a safe, effective, and reliable integrated system for assembled chemical weapons destruction.

The AEA SILVER II™ technology process does accomplish the following:

- It destroys chemical agent to a 99.9999 percent DRE and energetic materials to a 99.999 DRE by mediated electrochemical oxidation, provided that the following safeguards are observed:
  —hydrocyclones are used on all slurry feed streams,
  —high-shear vortex mixers are used,
  —piping design minimizes dead legs, and all system equipment and piping use trace heating to prevent precipitation of energetic intermediates, and
  —a second set of electrochemical cells is used to polish the anolyte to the required destruction level.
- It produces acceptable slurry compositions for processing burster energetics and rocket propellants.
- It safely treats process offgases using a combina-

tion of thermal treatment, catalytic oxidation, activated carbon filters, and treatment of the munitions demilitarization building (MDB) HVAC system ventilation air through activated carbon filter media prior to release.

However, the committee notes that the effectiveness of some process steps has not been demonstrated:

- Removal of energetics from rockets using the new tube-cutting technology in a modified baseline RDM, and achievement of agent-free energetics for processing by SILVER II™ electrochemical cells in a Category C area.
- Integrated operation of the primary and polishing anolyte circuits with a common catholyte circuit.
- Effective control of the flow of slurry mixture to a large number of parallel flow paths. (Currently, flow control has been demonstrated on only three parallel paths, whereas the full-scale application would have up to 432 parallel flow paths in the catholyte feed circuit.)
- Reliable operation of the electrochemical cell membrane, particularly for agent-containing slurries. (Currently, cell pressures and chemistry must be carefully controlled, and it is unclear if the membranes will work well in prolonged exposure to organophosphate-containing slurries.)
- Demonstration of a method to control the movement of fluorine and assure that all elements of the system exposed to fluorine-containing liquids and vapor streams can operate without excessive corrosion and maintenance.
- Decontamination of metal parts and fuzes to a 5X condition in the metal parts treater (MPT).
- Decontamination of dunnage and DPE suits to a 5X condition in the proposed, but as yet untested, enclosed-augur version of the dunnage treatment systems (DTS) and successful management of dioxins and furans in the offgas from this process.

General Finding (Blue Grass) 5. Stable operation of the ELI-Eco Logic/Foster Wheeler SCWO system at the design conditions has not yet been demonstrated. The SCWO system for treatment of hydrolysates in EDS II testing exhibited frequent spiking in hydrocarbon and carbon monoxide concentrations in the offgas. This issue must be resolved before implementing the Eco Logic process at Blue Grass. If it is resolved, the committee believes that the Eco Logic package could provide an effective and safe means for destroying the assembled chemical weapons. However, the following design features still require validation:

- Design of the overhead conveyor system for the caustic bath should be tested to demonstrate satisfactory reliability.
- Removal of aluminum from the feed to the SCWO reactor. At this time, Eco Logic has not proposed an aluminum removal technology.
- Smoke abatement from the thermal reduction batch processor (TRBP) smoking rooms and the measurement and management of carbon monoxide and other products of incomplete combustion generated in these rooms. (These processes were not adequately addressed in the EDP.)

The Eco Logic technology does accomplish the following:

- It disassembles munitions by a modified baseline reverse-assembly technology.
- It destroys chemical agent to a 99.9999 percent destruction and removal efficiency (DRE) and energetic materials to a 99.999 DRE.
- It effectively treats solid and gaseous residues in a gas-phase chemical reduction (GPCR™) system and decontaminates solids to a 5X condition.
- It destroys by SCWO the hydrolysates and slurries that result from upstream processing.
- It adequately treats the low volumes of offgases produced in the process, including those from the SCWO reactors, through catalytic oxidation and activated carbon adsorption systems.

The committee also points out the following:

- Eco Logic's munitions reverse-assembly process starts with the baseline system technology, but modifications have been made. The rocket dismantling machine (RDM) operation is complex and differs in several aspects from the baseline operation. Further development of this process is needed.
- A testing program, described in Chapter 4, showed that the M28 propellant grinding could be done safely. However, additional testing with a larger, modified grinder is required to demonstrate any design modifications and to determine grinder and motor sizes for the full-scale plant.

- The Demo II tests generally confirmed the performance and efficacy of the GPCR™ process for the treatment of the waste streams from the process. However, problems with the agent detection method hampered a full evaluation of the process, and some concerns were raised about the selection of materials of construction for the processing agent.

EDS testing has provided additional design data, but the results have not yet been used in operating systems.

General Finding (Blue Grass) 6. Based on the results of the EDS testing and the EDP, the committee believes that the General Atomics technology package is an effective and safe method for the destruction of assembled chemical weapons at the Blue Grass Army Depot. The GATS SCWO system appears to have reached a level of maturity where construction and testing of a full-scale reactor to treat agent hydrolysate is the next logical step. Tests of the SCWO system have shown that the SCWO reactor requires frequent scheduled maintenance; however, the level of maintenance estimated by General Atomics for the Blue Grass application is manageable by well-trained operators and maintenance personnel. The committee still is concerned about possible problems associated with the extent of scale-up that will be needed for the SCWO reactor. Another concern is the proposed operation of the full-scale ERH and PRH in a continuous rather than batch mode and the maintenance difficulties that would result.

The General Atomics technology does accomplish the following:

- It disassembles munitions by a modified baseline disassembly that removes the agent from the projectile bodies by cryofracture.
- It destroys chemical agents to a 99.9999 percent DRE by hydrolysis.
- It destroys fuzes by processing first in the ERH and then in the HDC.
- It destroys energetic materials by hydrolysis.
- It provides effective 5X-level decontamination for munition bodies using an electrically heated HDC.
- It destroys by SCWO the hydrolysates and slurries that result from this processing.
- It adequately treats the offgases produced in the process, including those from the SCWO reactors, through a CATOX unit and activated carbon adsorption systems.

It is expected that weapons processing at Blue Grass will require no SCWO liner changes for the SCWO reactors treating energetics hydrolysates and dunnage slurried in energetics hydrolysates. For each of the two operating reactors used to treat agent hydrolysates, the technology provider has scheduled six liner changes that are estimated to be necessary for treatment of GB hydrolysate (i.e., 12 liner changes total for the GB campaign) over the 16-month operating life of the plant. During periods of steady operation with GB hydrolysate, based on six liner changes per reactor, each liner will be replaced after 110 hours of operation. It appears likely that liner change-out procedures in the full-scale system will encounter more problems than were encountered in the smaller test reactors used to date.

Although no evidence was found of material hanging up in the ERH during the batch testing conducted to date, this is, in the committee's opinion, a distinct possibility during the longer operating runs of a full-scale system in a continuous mode. The energetics rotary hydrolyser (ERH) is 50 feet long and the projectiles rotary hydrolyser (PRH) is 40 feet long in the Blue Grass design. Should some form of blockage or other problem occur inside cylinders of this length, maintenance would be extremely difficult, especially considering that energetics and/or agent might be present. The tests demonstrated that the ERH and PRH appear to work well in a batch mode. Replacing the continuous-flow hydrolyzers proposed for Blue Grass with larger versions of the batch hydrolyzers used during the Demo I and EDS testing would appear to eliminate this risk and simplify system operation.

General Finding (Blue Grass) 7. As the ACW I Committee observed, the unit operations in any of the three technology packages have never been operated as total integrated processes (NRC, 1999). As a consequence, a prolonged period of systemization will be necessary to resolve integration issues for the selected technology as they arise, even for apparently straightforward unit operations.

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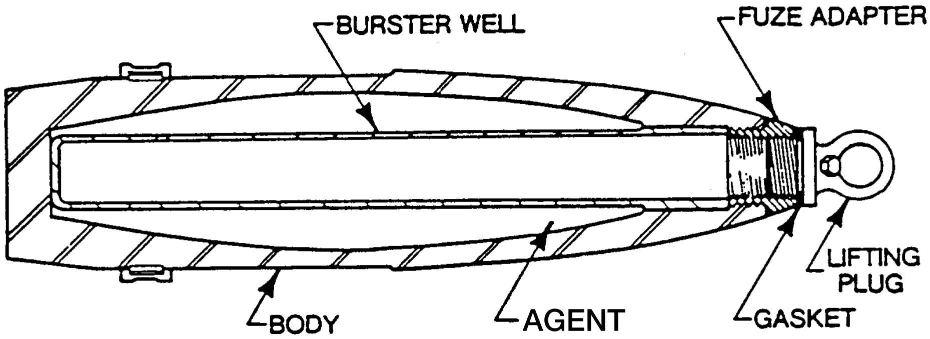
APPENDIXES

Appendix A

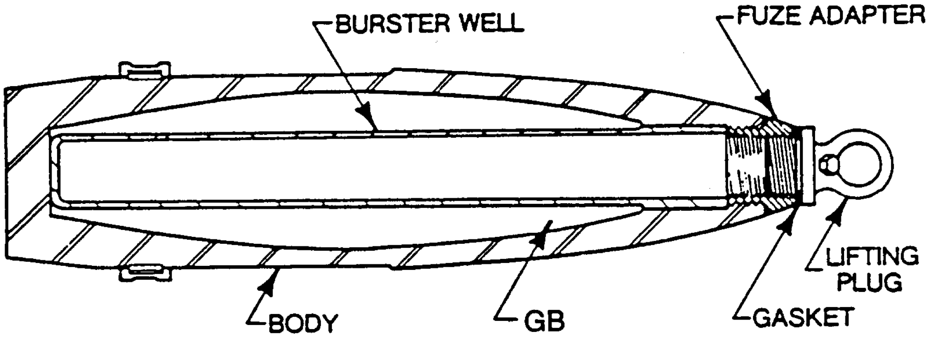
Description of Munitions in the Blue Grass Army Depot Stockpile

Figures A-1 through A-3 are cutaway drawings of the 155-mm shell, the 8-inch howitzer, and the 115-mm rocket. Information is also included on the size, weight, energetics, and packaging of each projectile.



	M110	**M121A1**
Length	26.8 in.	26.7 in.
Diameter	155 mm	155 mm
Total weight	98.9 lb	98.9 lb
Agent	H/HD	VX
Agent weight	11.7 lb	6.0 lb
Fuze	None	None
Burster	M6	M71
Explosive weight	0.41 lb	2.45 lb
Suppl chg explosive	None	None
Explosive weight	N/A	N/A
Propellant	None	None
Propellant weight	N/A	N/A
Primer	None	None
Packaging	8 rounds/ wooden pallet	8 rounds/ wooden pallet
Materials		
Shell	Steel	Steel
Burster well	Steel	Steel

FIGURE A-1 A 155-mm howitzer projectile, M110, H/HD; M121A1, VX. The drawing typifies projectiles of this sort. SOURCE: U.S. Army (1983).

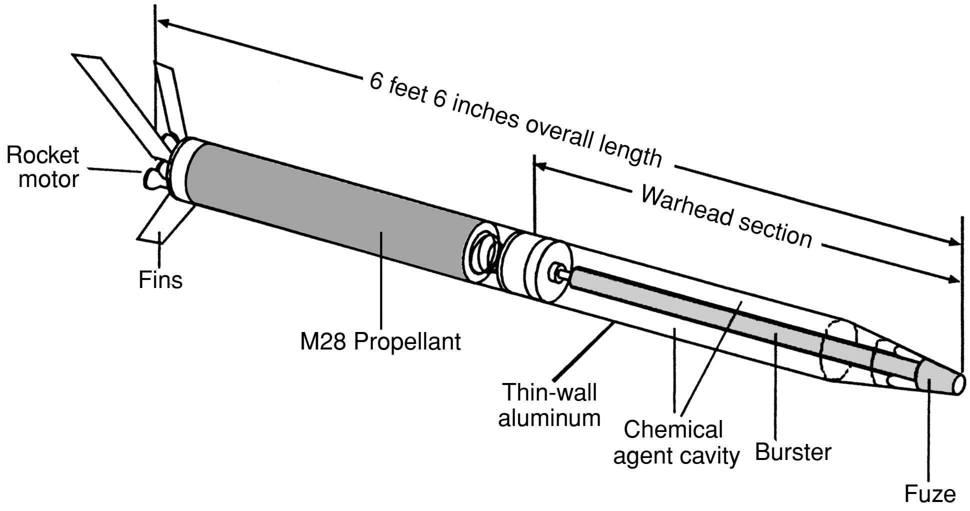


	M426
Length	35.1
Diameter	8 in.
Total weight	203 lb
Agent	GB
Agent weight	14.5 lb
Fuze	None
Burster	M83
Explosive	Comp B4
Explosive weight	7 lb
Suppl chg explosive	TNT
Explosive weight	0.3 lb
Propellant	None
Propellant weight	N/A
Primer	None
Packaging	6 rounds/ wooden pallet

Materials

Shell	Steel
Burster well	Steel

FIGURE A-2 An 8-in. howitzer projectile, M426, GB. The drawing typifies projectiles of this sort. SOURCE: U.S. Army (1983).



	H521	H520
Length	78 in.	78 in.
Diameter	115 mm	115 mm
Total weight	57 lb	57 lb
Agent	GB	VX
Agent weight	10.7 lb	10.7 lb
Fuze	M417	M417
Burster	M34, M36	M34, M36
Explosive	Comp B	Comp B
Explosive weight	3.2 lb	3.2 lb
Propellant	M28	M28
Propellant weight	19.3 lb	19.3 lb
Primer	M62	M62
Packaging	15 rounds/ wooden pallet	15 rounds/ wooden pallet

FIGURE A-3 A 115-mm rocket, M55, GB, VX. The drawing typifies a rocket of this sort. SOURCE: Feinberg (2001).

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Appendix B

Committee Meetings, Site Visits, and Information-Gathering Activities

COMMITTEE MEETING 1, JUNE 8-9, 2000

Edgewood, Maryland

NRC Participants

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

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 engineering design studies (EDS) status; tour and inspect the four test sites located 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)

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

Objectives

Visit the following Demonstration II test units: AEA 12 kW SILVER II™ test unit for energetics, AEA 2 kW SILVER II™ test unit for agent, Eco Logic gas-phase chemical reduction test unit.

Visit the following EDS test units: Parsons/Honeywell immobilized-cell bioreactor test unit.

SITE VISIT 2, JUNE 20-21, 2000

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, William R. Rhyne, Stanley I. Sandler, Leo Weitzman. NRC staff member: Harrison T. Pannella.

Objectives

Visit the following Demonstration II test units: Teledyne-Commodore solvated-electron technology (SET) test unit for energetics, SET test unit for agent, and fluid-jet cutting test unit; Foster Wheeler supercritical water oxidation (SCWO) test unit. Visit the following EDS test units: General Atomics SCWO test unit and dunnage shredder/hydropulping system. Tour the baseline incineration-based Tooele Chemical Agent Disposal Facility (TOCDF) and the adjacent chemical stockpile storage area.

SITE VISIT 3, AUGUST 2-3, 2000

Parsons, Inc., Pasadena, California

NRC Participants

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

Objectives

Receive briefings on the Parsons/Honeywell technology design package. Observe working sessions of scientists and engineers discussing preliminary examinations and possible minor modifications of the package.

Committee Meeting 2, August 8-9, 2000

National Research Council, Washington, D.C.

NRC Participants

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

Objectives

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

SITE VISIT 4, AUGUST 17-18, 2000

Arthur D. Little, Inc., Cambridge, Massachusetts

NRC Participants

Committee member: Leo Weitzman. NRC staff member: Patricia P. Paulette.

Objectives

Receive briefings on the General Atomics interim engineering design technology. Observe scientific and engineering working sessions on preliminary examinations and design optimization of the package.

SITE VISIT 5, AUGUST 25, 2000

Illinois Institute of Technology Research Institute, Chicago, Illinois

NRC Participants

Committee member: Leo Weitzman.

Objectives

Observe the Parsons catalytic oxidation (CATOX) unit under consideration for incorporation into the General Atomics technology package and scheduled for EDS testing.

SITE VISIT 6, SEPTEMBER 6, 2000

Aberdeen Proving Ground Edgewood Area, Maryland

NRC Participants

Committee member: Leo Weitzman. NRC staff members: Patricia P. Paulette, Harrison T. Pannella.

Objectives

Attend the integrated product review meeting on the status of the engineering-scale test for the one-tenth-scale General Atomics SCWO test unit planned for treatment of VX hydrolysate at the Newport bulk storage site.

SITE VISIT 7, SEPTEMBER 7, 2000

Aberdeen Proving Ground Edgewood Area, Maryland

NRC Participants

NRC staff members: Patricia P. Paulette, Bruce Braun, Harrison T. Pannella.

Objectives

Attend the joint Program Manager for Chemical Demilitarization/Program Manager for Assembled Chemical Weapons Assessment (PMCD/PMACWA) Industry Day briefings to commence nontechnology-specific acquisition request for proposals (RFPs) activities for the disposal facility at the Pueblo Chemical Depot.

SITE VISIT 8, OCTOBER 10, 2000

Pueblo Chemical Depot, Pueblo, Colorado

NRC Participants

NRC staff members: Patricia P. Paulette, Bruce Braun.

Objectives

Receive a briefing from the site commander of the Pueblo Chemical Depot, tour the storage bunkers, and visit the public outreach office. Attend a meeting of the Pueblo, Colorado, Citizens' Advisory Commission.

COMMITTEE MEETING 3, OCTOBER 19-20, 2000

J. Erik Jonsson 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, Stanley I. Sandler, William R. Seeker, Leo Weitzman. NRC staff members: Patricia P. Paulette, Bruce Braun, Harrison T. Pannella, Chris Jones.

Objectives

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

SITE VISIT 9, OCTOBER 25, 2000

Pine Bluff Chemical Depot, Pine Bluff, Arkansas

NRC Participants

Committee member: Willard C. Gekler.

Objectives

Observe unit operations of the SCWO reactor being tested for use in the destruction of smoke and dye materials.

SITE VISIT 10, OCTOBER 30, 2000

Holston Army Ammunition Plant, Kingston, Tennessee

NRC Participants

Committee members: Ruth M. Doherty, 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.

SITE VISIT 11, NOVEMBER 3, 2000

ACWA Dialogue Meeting, Pueblo, Colorado

NRC Participants

Committee Chair: Robert A. Beaudet. Committee members: Ruth M. Doherty, John A. Merson, Hank C. Jenkins-Smith. NRC staff members: Patricia P. Paulette, Bruce Braun.

Objectives

Provide briefings to the Dialogue on NRC activities concerning the ACWA program and attend the Dialogue meeting.

SITE VISIT 12, NOVEMBER 8-10, 2000

Parsons, Inc., Pasadena, California

NRC Participants

Committee Chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Willard C. Gekler, David S. Kosson (Parsons/Honeywell only), John A. Merson, William R. Rhyne, Stanley I. Sandler (General Atomics only), Leo Weitzman. NRC staff members: Patricia P. Paulette, Harrison T. Pannella.

Objectives

Receive briefings about the Parsons/Honeywell and General Atomics EDS. Results of some EDS testing were also reported.

SITE VISIT 13, NOVEMBER 26-28, 2000

Parsons, Inc., Pasadena, California

NRC Participants

Committee Chair: Robert A. Beaudet. Committee member: William C. Gekler.

Objectives

Attend briefings prepared for the Army ACWA team by the technology providers on the status of preliminary technical reports concerning the Parsons/Honeywell and General Atomics engineering designs. Results of testing for the engineering designs were also reported.

SITE VISIT 14, DECEMBER 7, 2000

Edgewater, Maryland

NRC Participants

Committee members: Joan B. Berkowitz, Leo Weitzman.

Objectives

Attend presentation by General Atomics to PMACWA on the developmental status of its SCWO reactor.

COMMITTEE MEETING 4, DECEMBER 14-15, 2000

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

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 15, JANUARY 4, 2001

ACWA II Dialogue Meeting, Lexington, Kentucky

NRC Participants

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

Objectives

Attend Dialogue meeting and provide an update of the NRC ACWA II Committee's activities.

SITE VISIT 16, 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 working review meeting on energetics hydrolysis testing at Holston and the results of this testing to date. Become informed on the scientific and engineering issues revealed during the test procedures.

COMMITTEE MEETING 5, FEBRUARY 8-9, 2001

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

Objectives

Receive briefings from the PMACWA technical staff concerning the status of EDS I, EDS II, and Demonstration 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.

INFORMATION GATHERING ACTIVITY, MARCH 11-14, 2001

Houston, Texas

NRC Participants

NRC staff member: Patricia P. Paulette.

Objectives

Attend National Association of Corrosion Engineers (NACE) Symposium on SCWO technology as it relates to materials of construction and corrosion reactions in reactors. Arrange to obtain scientific briefing packages and technical papers for distribution to committee members.

COMMITTEE MEETING 6, MARCH 26-27, 2001

Beckman Center, 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, Leo Weitzman. NRC staff members: Bruce Braun, Patricia P. Paulette, Harrison T. Pannella, Gwen Roby, William 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 date and discuss items for the agenda.

COMMITTEE MEETING 7, MAY 9-10, 2001

Washington, D.C.

NRC Participants

Committee Chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Joan B. Berkowitz, Ruth M. Doherty, Willard C. Gekler, Frederick J. Krambeck, John A. Merson, William R. Rhyne, William R. Seeker, Leo Weitzman. NRC staff members: Bruce Braun, Patricia P. Paulette, Mike Clark, Dennis Chamot, Harrison T. Pannella, Gwen Roby, William Campbell.

Objectives

Receive briefings from the PMACWA technical staff concerning the status of the EDS II program activities. Prepare responses to reviewers' comments on the EDS I (Pueblo) report and complete the concurrence draft for the Demonstration II report. Assign writing teams, set goals for interim activities, schedule next meeting date, discuss agenda items. Discuss concept drafts for the Blue Grass, Kentucky, report.

SITE VISIT 17, JUNE 20-21, 2001

Dugway Proving Ground and Deseret Chemical Depot, Utah

NRC Participants

Committee members: Willard C. Gekler, Sheldon E. Isakoff, William R. Rhyne.

Objectives

Inspect and evaluate the following EDS test units: Foster Wheeler and General Atomics SCWO units, Parsons Projectile Washout and Continuous Steam Treatment, and the Eco Logic Propellant Grinding System.

SITE VISIT 18, JULY 17-18, 2001

General Atomics Offices, San Diego, California

NRC Participants

Committee Chair: Robert A. Beaudet. Committee members: Willard C. Gekler, David S. Kosson, Frederick J. Krambeck, Leo Weitzman. NRC staff member: Patricia P. Paulette.

Objectives

Attend a presentation and review of the initial engineering design package developed by General Atomics for the demilitarization of chemical weapons at the Blue Grass, Kentucky, storage site.

SITE VISIT 19, AUGUST 6-7, 2001

CH2MHill Offices, Gainesville, Florida

NRC Participants

Committee Chair: Robert A. Beaudet. Committee members: Ruth M. Doherty, Willard C. Gekler, William R. Seeker. NRC staff member: Patricia P. Paulette.

Objectives

Attend a presentation and review of the initial engineering design package developed by AEA Technologies for the demilitarization of chemical weapons at the Blue Grass, Kentucky, storage site.

SITE VISIT 20, AUGUST 9-10, 2001

Houston, Texas

NRC Participants

Committee Chair: Robert A. Beaudet. Committee members: Richard J. Ayen, John A. Merson, William R. Rhyne, Stanley I. Sandler. NRC staff member: Patricia P. Paulette.

Objectives

Attend a presentation and review of the initial engineering design package developed by Foster Wheeler/ Eco Logic/Kvaerner for the demilitarization of chemical weapons at the Blue Grass, Kentucky, storage site.

COMMITTEE MEETING 8, SEPTEMBER 9-10, 2001

Washington, D.C.

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, Stanley I. Sandler, Hank C. Jenkins-Smith, Leo Weitzman. NRC staff members: Bruce Braun, Patricia P. Paulette, Mike Clarke, Harrison T. Pannella, Gwen Roby.

Objectives

Discuss press conference information for the EDS Pueblo Report and report dissemination. Discuss technology provider reviews attended in October. Integrate writing contributions and continue to develop an initial first full message draft of the EDS Blue Grass Report.

SITE VISIT 21, OCTOBER 10, 2001

Aberdeen Proving Ground Edgewood Area, Maryland

NRC Participants

Committee members: Ruth M. Doherty, Frederick J. Krambeck.

Objectives

Tour the Edgewood CDTF (Chem Demilitarization Training Facility) to become familiar with weapons structures and the complete weapons incineration plant.

SITE VISIT 22, OCTOBER 22-23, 2001

General Atomics Offices, San Diego, California

NRC Participants

Committee Chair: Robert A. Beaudet. Committee members: Willard C. Gekler, Frederick J. Krambeck, John A. Merson, Leo Weitzman. NRC staff member: Patricia P. Paulette.

Objectives

Attend presentations of General Atomics final engineering design package and most recent updates of unit demonstration test results.

SITE VISIT 23, OCTOBER 25-26, 2001

CH2MHill Offices, Gainesville, Florida

NRC Participants

Committee Chair: Robert A. Beaudet. Committee members: Frederick J. Krambeck, Willard C. Gekler. NRC staff member: Patricia P. Paulette.

Objectives

Attend presentations of AEA's final engineering design package and most recent updates of unit demonstration test results.

SITE VISIT 24, OCTOBER 29-30, 2001

Houston, Texas

NRC Participants

Committee members: John A. Merson, William R. Rhyne, Richard J. Ayen. NRC staff members: Patricia P. Paulette, Harrison T. Pannella.

Objectives

Attend presentations of the Eco Logic/Foster Wheeler/Kvaerner final engineering design package and most recent updates of unit demonstration test results.

COMMITTEE MEETING 9, NOVEMBER 15-16, 2001

Washington, D.C.

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, Leo Weitzman. NRC staff members: Bruce Braun, Patricia P. Paulette, Harrison T. Pannella, Gwen Roby, Catherine Lewis.

Objectives

Receive briefings from the PMACWA technical staff concerning the EDS II testing plan and the Blue Grass demilitarization program status. Develop concept draft for the Pueblo letter report. Continue development of the EDS II first full message draft. Conduct a bias discussion update.

SITE VISIT 25, NOVEMBER 19-20, 2001

Arthur D. Little, Inc., Cambridge Massachusetts

NRC Participants

Committee members: Joan B. Berkowitz, Frederick J. Krambeck, Leo Weitzman.

Objectives

Attend presentations of General Atomics final engineering design package and the most recent updates of unit demonstration test results.

SITE VISIT 26, DECEMBER 6, 2001

Avondale Elementary School, Avondale, Colorado

NRC Participants

Committee Chair: Robert A. Beaudet. NRC staff members: Bruce Braun, Patricia P. Paulette, Donald Siebenaler, William Campbell.

Objectives

Present the NRC findings and recommendations from the NRC EDS I—Pueblo report to the Citizens Advisory Commission for Pueblo, Colorado.

COMMITTEE MEETING 10, JANUARY 3-4, 2002

Beckman Center, Irvine, California

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, Stanley I. Sandler, William R. Seeker, Leo Weitzman. NRC staff members: Patricia P. Paulette, Harrison T. Pannella, Gwen Roby.

Objectives

Receive briefings of the most recent demonstration test results from technology providers. Develop responses to reviewer's comments for the EDS I update letter report. Continue text development toward a concurrence draft for the Blue Grass EDS II report.

COMMITTEE MEETING 11, MARCH 14-15, 2002

Washington, D.C.

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, Stanley I. Sandler, Leo Weitzman. NRC staff members: Bruce Braun, Patricia P. Paulette, Harrison T. Pannella, Gwen Roby.

Objectives

Receive briefings on "Operational Risk Assessments for Three Technologies Being Considered for Chemical Weapons Destruction at Pueblo, Colo." Receive update of ACWA Program for Bluegrass. Continue text development toward a concurrence draft for the Blue Grass EDS II report. Set goals and confirm next meeting date and location,

SITE VISIT 27, JUNE 5-6, 2002

ACWA II Dialogue Meeting, Lexington, Kentucky

NRC Participants

Committee Chair, Robert A. Beaudet. NRC staff members: Bruce Braun, Patricia P. Paulette. Harrison Pannella.

Objectives

Attend Dialogue meeting and provide an update of the NRC ACW II Committee's activities.

COMMITTEE MEETING 12, JUNE 13-14, 2002

Washington, D.C.

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, Stanley I. Sandler, Leo Weitzman. NRC staff members: Bruce Braun, Patricia P. Paulette, Harrison T. Pannella, Gwen Roby.

Objectives

Receive an update of the ACWA program and a technical presentation concerning H destruction processes. Develop responses to the reviewer comments on the current report. Lay out concept draft for the Blue Grass letter report.

COMMITTEE MEETING 13, JUNE 20-21, 2002

Washington, D.C.

Participants

Committee Chair: Robert A. Beaudet. Committee members: Richard J. Ayen, Frederick J. Krambeck, John A. Merson, William Rhyne, Stanley I. Sandler, William Seeker, Leo Weitzman. NRC staff members: Bruce Braun, Patricia P. Paulette, Harrison T. Pannella, Gwen Roby.

Objectives

Receive briefings from Foster Wheeler on the results of validation testing of the transpring-wall SCWO and the evaporator/crystallizer. Evaluate the test results and discuss the objectives for the Blue Grass letter report. Complete the outline for the first full message draft and determine writing assignments. Discuss goals and significant dates in the development of the letter report.

Appendix C

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 Chemical Corps 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 the chair of the Chemistry Department from 1974 to 1979. He has served on Department of Defense committees that have addressed both offensive and defensive considerations surrounding chemical warfare agents. He was chair of an Army Science Board committee that addressed chemical detection and trace gas analysis. He also was the chair of an Air Force technical conference on chemical warfare decontamination and protection. He has served on two NRC studies on 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 of 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 in these areas.

Richard J. Ayen received his Ph.D. in chemical engineering from the University of Illinois. Dr. Ayen was formerly a 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 Waste Management's Clemson Technical Center, including treatability studies and technology demonstration for hazardous and radioactive waste. Dr. Ayen has published extensively in his fields of interest.

Joan B. Berkowitz graduated from the University of Illinois with a Ph.D. in physical chemistry. Dr. Berkowitz is currently managing director of Farkas Berkowitz and Company. She has extensive experience in the area of environmental and hazardous waste management, a knowledge of available technologies for the cleanup of contaminated soils and groundwater, and a background in physical and electrochemistry. She has contributed to several EPA studies, been a consultant on remediation techniques, and assessed various destruction technologies. Dr. Berkowitz has written numerous publications on hazardous waste treatment and environmental subjects.

Ruth M. Doherty received a Ph.D. in physical chemistry from the University of Maryland. Dr. Doherty is currently technical advisor for the Research and Technology Department, Naval Surface Warfare Center, Indian Head, Maryland. Since 1983, she has coauthored almost 60 publications in various subjects in the physical chemistry arena, including the chemistry of underwater explosives. Over the past 6 years, Dr. Doherty has conducted over 30 presentations in various aspects of the science and technology of explosives. She has worked extensively in the research and development of energetics materials and explosives with the Naval Surface Warfare Center for over 15 years. She is a member of the editorial advisory board of the journal *Propellants, Explosives and Pyrotechnics*.

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, ABS Consulting, 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, Umatilla, Pine Bluff, Aberdeen, and Newport chemical disposal facilities and project manager for development of facility design criteria for the Johnston Atoll Chemical Agent Disposal system. 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, The American Institute of Chemical Engineers (AIChE), and the American Nuclear Society. He is the author or coauthor of numerous publications.

Sheldon E. Isakoff received his Ph.D. in chemical engineering from Columbia University. Dr. Isakoff is the retired director of the Engineering R&D Division of E.I. du Pont de Nemours & Company. His expertise includes the management of technology. More specifically, he has been engaged in activities encompassing the direction of research and development, market assessment and development, process scale-up, commercial introduction, and leadership of personnel involved in these activities. His expertise also includes materials science and engineering and the development and application of new materials for both industrial and consumer markets. Dr. Isakoff is a former president of AICHE, was elected to the National Academy of Engineering in 1980, and has served on numerous National Research Council committees.

Hank C. Jenkins-Smith is professor of public policy at the George H.W. Bush School of Government and Public Service at Texas A&M University in College Station. He holds the Joe R. and Teresa Lozano Long Chair of Business and Government at the Bush School. He was previously 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. Previously, he was professor of chemical and biochemical engineering at Rutgers. Well known for his expertise in bioremediation, Dr. Kosson has carried out research and published extensively on subsurface contaminant transport phenomena; leaching phenomena; physical, chemical, and microbial treatment processes for hazardous waste; and waste management policy. He served on the NRC Committee on Review and Evaluation of the Army Chemical 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, Dr. Kosson contributed to the decision to use alternative methods of destruction at both the Aberdeen and Newport facilities.

Frederick J. Krambeck received his Ph.D. in chemical engineering from the City University of New York. Dr. Krambeck is a consultant in research and development in petroleum refining, including process and reactor design and development, chemical reaction engineering, online and offline 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. He is also a fellow and member of the board of directors for the American Institute of Chemical Engineers. He has authored or coauthored over 25 patents and 40 other 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. Dr. Merson is currently the department manager for the Stockpile Stewardship Office at Sandia National Laboratories. His prior experience at Sandia has included research, development, and application of energetic materials and components within the nuclear weapons stockpile. He has been responsible for surveillance, chemical compatibility, energetic material characterization, advanced component development and production. He has designed components for DOE, DoD, and NASA programs. Dr. Merson is a member of the American Institute of Chemical Engineers and other professional societies.

William R. Rhyne received a B.S. in nuclear engineering from the University of Tennessee and M.S. and D.Sc. degrees in nuclear engineering from the University of Virginia. Dr. Rhyne is currently an independent consultant and earlier cofounded H&R Technical Associates, Inc., where he remains a member of the board of directors. He has extensive experience in risk and safety analyses 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 has authored or coauthored numerous publications and reports in nuclear and chemical safety and risk analysis areas and is author of the book *Hazardous Materials Transportation Risk Analysis: Quantitative Approaches for Truck and Train*. He is a former 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 graduated from the University of Minnesota with a Ph.D. in chemical engineering. Currently, Dr. Sandler is the Henry Belin du Pont Chair and director of the Center for Molecular and Engineering Thermodynmics at the University of Delaware. His extensive research interests include applied thermodynamics and phase equilibrium, environmental engineering, and separations and purification. Among the awards Dr. Sandler has received are the Warren K. Lewis and Professional Progress Awards from the American Institute of Chemical Engineers and the E.V. Murphree Award of the American Chemical Society. He is a member of the National Academy of Engineering, the author of several books, and has had over 290 technical articles published in recognized journals and conference proceedings. He is a member of the Technical Advisory Board of Aspen Technology, Inc., and editor of the *AIChE Journal*. He is also a member of the Sierra Club.

William Randall Seeker received his Ph.D. in engineering (nuclear and chemical) from Kansas State University. He is the senior vice president and a member of the board of directors of Energy and Environmental Research Corporation. Dr. Seeker has extensive experience in the use of thermal treatment technologies and environmental control systems for managing hazardous waste. He is a member of the Executive Committee of the Environmental Protection Agency's Science Advisory Board. Dr. Seeker has authored over 100 technical papers on various aspects of technology and environment subjects.

Leo Weitzman received his Ph.D. in chemical engineering from Purdue University. He is a consultant with 30 years of experience in the development, design, permitting, and operation of equipment and facilities for treating 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 over 40 technical papers