TESTING UPDATE: Battelle's Acid Digestion Process

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ABSTRACT

Previous briefings have introduced Battelle's Acid Digestion Process™ (ADP), a prototype system, designed to treat recovered munitions containing explosive components and toxic chemicals. Since 2003, Battelle has continued testing the ADP with recovered WWI and WWII era munitions, munition components, and various fills.

This paper will highlight testing done using the ADP equipment since 2003 and will discuss the wide range of toxic chemicals shown to be compatible, explosives and fuzing mechanisms tested, and the recent testing done to treat incendiary and newly developed insensitive munitions.

The breadth of testing had demonstrated that the procedures, process, and equipment, can be applied to other recovered chemical warfare materiel destructions, as well as other UXO. Preliminary studies have also shown that the ADP is capable of destroying many industrial toxic chemicals, smoke, and biological fills. Battelle is currently developing ADP process concepts to treat multiple munitions with explosive components and propellants.

Notes about the presenter

Ed Groth, a program manager with Battelle Memorial Institute, Aberdeen, MD began his Army career at the 259th Ordnance Detachment (Explosive Ordnance Disposal), Savanna Army Depot, Illinois in 1972. His assignments included the US Army in Berlin, Germany, the 45th Ord Det (EOD) at Ft. Polk, Louisiana, the Technical Escort Unit at Edgewood Arsenal, Maryland and culminated in his deploying with the 43rd Ord Det (EOD) of Ft. Knox to Operation Desert Storm. Since leaving the military, he has supported the Program/Product Manager for Non-Stockpile Chemical Materiel in various contractor roles related to mobile destruction systems, including the Munitions Management Device, Explosive Destruction System, and the Rapid Response System. He is currently utilizing his mobile treatment system experience as Program Manager for Battelle's Acid Digestion Process. Mr. Groth has supported projects involving demilitarization of recovered chemical warfare material in Europe and various sites within the continental United States.

Mr. Groth's educational accomplishments include being a distinguished graduate of US Naval Explosive Ordnance Disposal Course, Indian Head Naval Ordnance Station, and a distinguished graduate for US Army Technical Escort Qualification Course, Redstone Arsenal, Alabama. He has also attended various EOD related course at Sandia National Labs, Savanna Army Depot, and Indian Head NOS. He holds a BS degree from Upper Iowa University.

1.0 INTRODUCTION

Battelle has developed a process for demilitarizing chemical warfare agents both in munitions containing explosives and chemical agents, and those stored in steel containers. The treatment method, known as the Acid Digestion Process (ADP), has been shown to be effective with a wide range of chemical agents, biological agents, and explosives. In the context of disposal of chemical or biological agents stored in steel containers or munitions, the ADP does not require cutting, drilling or mechanical disassembly of any kind to access and neutralize the agents. Also, the reactor chamber size, as well as the entire design of the system utilizing the ADP, can be tailored to accommodate a wide variety of container/munition shapes and sizes. In addition, the process can be augmented with additives to minimize emissions, enhance dissolution, or facilitate recovery and disposal of the byproducts. The ADP may be configured as a fixed installation or as a transportable/mobile system.

The ADP is a straightforward chemical decomposition of the entire container/munition and internal chemical fill. The process has been developed to a level of maturity consistent with field demonstration through testing on various container/munition types and chemical agents. In the course of reaching this level of maturity, Battelle has completed several demonstrations consisting of treatment of recovered chemical munitions smoke munitions, and stockpiled conventional projectiles ranging from 75 mm to 120 mm. Throughout development and testing, performance and safety issues have been mitigated through focused design efforts.

2.0 PROCESS DESCRIPTION

The ADP consists of four steps:

- **Step 1 – Loading:** The munitions or hazardous material containers are placed into a sealed reaction vessel. No special preparation of the item is required so handling is minimized.
- **Step 2 – Digestion:** The primary step of the treatment system occurs once the processing tank is filled with an acid solution. This solution accesses the contents of the munitions and metal containers, treats hazardous compounds and separates any non-soluble items into liquid and solid waste products.
- **Step 3 – Off-Gas Treatment:** The gases generated during the digestion step are collected and then treated by a series of wet scrubbers.
- **Step 4 – Process Waste Treatment:** The ADP treats the hazardous items such that the liquid and non-soluble wastes are suitable for safe transport for final disposal by one of several commonly available secondary treatment technologies.

The ADP uses dilute (7 molar) nitric acid to digest the steel container and access the internal chemical and explosive components for treatment or disposal. Nitric acid is such a powerful oxidizer that it does not produce any flammable gases (hydrogen, for example, is oxidized to water). The effectiveness of nitric acid as a digestion solution provides a secondary benefit because of its recognized ability as a decontaminant for chemical agents. The oxidizing power of nitric acid is sufficient to effect reactions with many chemical agents and thus render them neutralized. The aqueous environment also provides for efficient hydrolysis to occur.

While the use of nitric acid as a standard decontaminant is not generally recommended due to its corrosive properties, this corrosiveness does make it the acid of choice for the ADP. During processing, the active ingredients of a container or munition are both accessed and destroyed by the nitric acid solution in a contained environment. Vapors of agent are not released to a room, but are contained in the process and destroyed. Containers or munition bodies do not require further decontamination due to the aggressive action of the acid; if desired, the entire container body can be dissolved.

While nitric acid is an excellent decontaminant for chemical and biological agents (and will dissolve most metals), it does not react with the explosive components allowing the explosive components to be easily collected for reuse, recycle, or disposal.

3.0 TESTING

The following sections detail the testing that has been conducted in support of ADP design and development.

3.1 Explosive Testing

Explosive testing, including drop tests and static discharge tests, was conducted to determine if exposure to nitric acid would sensitize the explosives. Testing has shown that explosives are not sensitized as a result of nitric acid treatment. Table 3-1 lists the energetic materials Battelle has tested for compatibility with the ADP.

Table 3-1. Explosives Tested with the ADP

Primary Explosives	Dinitrobenzene
Lead styphanate	Dinitrotoluene
Lead azide	Picric acid
Mercury fulminate	Hexanitrodiphenylamine
Propellants	**Pyrotechnics**
Black powder	Barium chromate/zirconium
Double base propellant	Flash powder
Secondary Explosives	**Insensitive Explosives**
TNT	HBU-88B
RDX - cyclonite	AFX-757
PETN	PBXN-109

3.2 Fuze Testing

Battelle tested fuze components to determine if there is a potential for causing an armed fuze to function during acid digestion. Testing was conducted with non-electric blasting caps (#8), shotgun shell primers (W290), hand grenade fuzes (M213), and time fuse (M700).

The results of testing are summarized as follows:

Time fuse (black powder) did not ignite as designed after being wetted with nitric acid. When mechanical fuzes dissolved in the acid solution the acid attacked the primers quickly enough to prevent ignition, even when the fuze functions mechanically. Blasting caps could not be detonated in the drop fixture either before or after acid digestion, suggesting that #8 caps are not particularly sensitive to shock. Following digestion, blasting caps did not detonate when ignited as designed. As expected, the aluminum blasting caps did not dissolve. Shock sensitive energetic materials (primers) tested indicated the primer to be less sensitive to ignition/detonation from impact after being wetted with the nitric acid solution.

3.3 Recent Testing/Treatability Studies

Table 3-2 (following page) lists the munitions (including suspect fill material) that have been destroyed via the ADP as part of treatability studies.

Table 3-2. Munitions Treated Via the ADP

Munition	#	Suspect Fill	Comments
Unfuzed Projectile	3	Arsine	Recovered in Washington DC
WWI era fuzes	3	Mercury Fulminate	Fuzes Only
Armed/Fuzed Projectiles	5	Phosgene (CG) Hydrogen Cyanide (AC) Arsenic Trichloride Oleum (FS) Mustard (H) Ethyl Iodoacetate (SK)	Anomaly Occurred*
Unfuzed Mortar	1	HBU 88B	Insensitive Munition

Discussion of Test Anomaly

During the disposal of WWI era recovered chemical weapons, an incident occurred involving a small projectile. In the course of destroying the projectile, the command post unexpectedly lost closed circuit view of the reactor interior. The reactor pressure recorded was just above normal but well below design pressures. The temperature was at the normal operating temperature. Room monitors did not detect any hazardous vapors. Personnel entered the test chamber, inspected the ADP demonstration hardware, and discovered visible damage to the viewport (which still held pressure), and to the camera. Treatment was continued for an additional hour to ensure that the munition had been accessed and the fill treated. After removal of the munition carcass and waste, an additional inspection of the hardware and munition body was conducted.

Following operations, personnel found that rupture discs were intact and hardware damage was limited to the broken view port and the light-weight basket holding the munition. There was no fragmentation damage. The camera had fallen to the floor and was inoperable although had no visible damage. A unrelated hardware problem was discovered, a pump diaphragm was damaged (may have occurred before or after incident).

An inspection of the munition carcass revealed the fuze fragmented into large pieces. The threads were sheared at nose of projectile but the main body was intact. There was no residue visible in munition body.

Battelle believes that the fuze was ejected due to build up of hydrogen from the long term degradation of mustard in the munition. Hydrochloric acid is created, which reacts with steel and produces hydrogen. Dissolution of the threads holding the fuze allowed the forcible ejection of the fuze into the sight glass. The fuze likely had sufficient energy to cause the fuze to detonate when it struck the glass.

It does not appear that an explosion occurred while the fuze was still in the projectile since the top edges of the projectile body did not appear to be bent outwards. The absence of any fragmentation damage to ADP reactor and lack of any evidence of overpressure, implied the booster did not ignite.

3.4 Chemical Compatibility

The breadth of testing conducted to support the disposal of recovered munitions is shown below. The testing shows that treatment of any of the potential fills can be completed using the same basic recipe and equipment, thereby minimizing handling required to non-intrusively characterize each munition. Reduction of this handling step would reduce risk to workers, and increase the throughput for the system. A complete list of chemicals that have been treated will be provided upon request.

Simple lachrymators – 5
Toxic lachrymators – 7
Respiratory Irritants (Toxic) –1
Choking Agents – 2
Blood Agents – 3
Vomiting Agents – 3

Lung Injurants (Simple) – 9
Lung Injurants – 4
Smokes – 6
Vesicants – 5
Solvents – 2

3.5 Treating Insensitive Munitions

Disposal of insensitive munitions is expected to become an issue as these munitions become obsolete or unserviceable. Difficulties have arisen in removing the cast-cured or thermo-cured explosives from the munition casing. The ADP has demonstrated the ability to remove the casing from the explosives and that explosive contamination of the liquid waste did not occur.

3.6 Testing with Phosphorus

Disposal of munitions containing red and white phosphorus also presents challenges. Prior to testing a munition in the demonstration unit, Battelle personnel conducted calorimeter testing and waste analysis to verify that treatment of RP/WP with nitric acid is safe and that waste generated can be recycled or disposed in commercial facilities.

Preliminary testing has shown that the materials are compatible as used in the ADP and that the waste is predominantly phosphates and nitrates which are not reactive. This will allow treatment of UXO or range scrape containing WP or RP in a simple reactor with local disposal of the waste. Testing has also indicated that heats of reaction are well within the design basis used for the demonstration unit, indicating it is safe to treat a full scale munition in the test unit.

4.0 SIMPLIFIED SCALE UP

The current ADP demonstration hardware was meant only for proof of concept testing. A production unit could be fabricated based on the demonstration unit design, while taking advantage of lessons learned and site specific needs. The demonstration hardware is designed for processing a single munition. This approach is applicable for small sites as it is readily transportable, requires minimal controls, and is easy for a technician to operate remotely. Battelle has developed a design concept for use of the ADP for large containers or munitions, as well as a design concept for simultaneously destroying multiple munitions.

Scale up of the process hardware is not limited since the components are readily available from commercial sources. Throughput is limited by the reaction rate with the steel and not by explosive weight, fill weight, or size of the equipment. Recovery and reuse of the acid is also feasible with commercially available equipment.

With additional operational experience and detailed characterization of the waste resulting from smaller sites or developmental testing, Battelle expects to show that significant amounts of materials resulting from treatment can be recycled, recovered, or reused.