

ESTCP Cost and Performance Report

(CP-9708)



Recovery and Reuse of HMX/RDX from Propellants and Explosives (150 lbs/day Pilot Plant)

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ENVIRONMENTAL SECURITY
TECHNOLOGY CERTIFICATION PROGRAM

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LIST OF ACRONYMS

ANFO	Ammonium Nitrate/Fuel Oil
ANPF	Ammonium Nitrate/Polymeric Fuel
Comp A-3	Composition A-3, 91% RDX and 9% Wax
DoD	Department of Defense
DOE	Department of Energy
EPCRA	Emergency Planning and Community Right to Know Act
HMX	Octogen, Homocyclonite (cyclotetramethylenetetranitramine)
HPLC	High Performance Liquid Chromatography
HW	Hazardous Waste
IM	Insensitive Munitions
IRR	Internal Rate of Return
LANL	Los Alamos National Laboratory
LX-14	High explosive, 95% HMX and 5% Estane™ Binder
NO _x	Oxides of Nitrogen
NPV	Net Present Value
NSWC Crane	Naval Surface Warfare Center, Crane Division
OB/OD	Open Burning/Open Detonation
PBX	Plastic Bonded Explosive
PLC	Process Loop Control
RDX	Hexogen, Cyclonite (cyclotrimethylenetrinitramine)
RRDA	Resource Recovery and Disposition Account
SBIR	Small Business Innovation Research
SOP	Standard Operating Procedure
TRI	Toxic Release Inventory
VOCs	Volatile Organic Compounds

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Technical material contained in this report has been approved for public release.

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1.0 EXECUTIVE SUMMARY

- **Background.** DoD has over 21,352 tons of HMX-containing and 14,545 tons of RDX-containing materials in the Resource Recovery and Disposition Account (RRDA) and expects to generate several thousand more tons over the next five years [1]. These materials are currently destroyed by open burning and open detonation (OB/OD). These destructive means of disposal cause environmental problems, due to the release of toxic by-products into the environment. Also, a valuable resource is lost when these explosives are destroyed. TPL, Inc., at Ft. Wingate Army Depot, Gallup, New Mexico, under contract to Naval Surface Warfare Center, Crane Division (NSWC Crane), demonstrated a technology for the recovery of HMX and RDX from LX-14 and Composition A-3 for reuse applications. TPL's process involved solubilizing or melting the explosives' binders with either acid or hot water/ surfactant solution, then separating the explosive from the binder solution by centrifugation. A single facility capable of processing 150 lbs per day of both types of explosives was designed and successfully demonstrated.
- **Objectives of the Demonstration.** This technology was developed to provide DoD with a means of recovering high-value energetic materials. A facility capable of recovering different types of energetics was of interest. The recovered materials were expected to be of high purity at a high yield and meet Military Specifications. In order to evaluate the versatility of the system for recovering two different explosives, TPL performed a two-day demonstration of the recovery of HMX from LX-14 and also a two-day demonstration of the recovery of RDX from Composition A-3. The demonstrations were performed between March 21-25, 1999 [2]. The recovered RDX and HMX were of high purity at a high yield and had melting points comparable to those of pure RDX and HMX.
- **Regulatory Drivers.** The DoD has been disposing of excess, obsolete, and unserviceable munitions via OB/OD for decades. Public awareness has prompted the DoD to look for alternative methods of disposal. Open burning on soil has been banned, therefore burning in steel pans has been the alternative. Even this alternative has been under scrutiny. There have been strict regulations on the quantity of energetics treated and on weather conditions before and after the OB/OD operations. As required by the Hazardous and Solid Waste Amendments, the Environmental Protection Agency required that all installations desiring to continue OB/OD operations submit Subpart X permit applications or close their OB/OD sites. These permits have been known to be costly, ranging to well over \$1M. Executive Order 12856 required the DoD to comply with all provisions of the Emergency Planning and Community Right to Know Act (EPCRA), including Toxic Release Inventory (TRI) reporting, since 1994. The Deputy Under Secretary of Defense (Environmental Security) issued guidance requiring the TRI reporting for demilitarization of munitions beginning with the calendar year 1999 reporting cycle, July 2000.
- **Demonstration Results.** The HMX and RDX recovered during the demonstration were analyzed for purity, melting point, physical appearance, and batch yield. HMX recovery resulted in greater than 98% in purity and batch yields, while RDX recovery was 95% or better with an average purity of 98%. The average processing times for HMX and RDX recovery were 24 and 6 hours, respectively. The data collected provided a clear indication of the product quality based on comparison to values given in applicable Military

Specifications and on past experience with analyzing materials from both the pilot plant and the demonstration plant. Past experience has indicated that materials which meet purity levels and melting point criteria were also consistently and repeatably within specifications for acidity levels and insolubles. Based on this acquired experience, the samples measured from the demonstration were considered acceptable for evaluation by potential customers for alternate applications. Two batches of LX-14 were processed to recover HMX. Three batches of Composition A-3 were processed to recover RDX.

The cost benefit study for HMX recovery showed a favorable payback period of 3.6 years. The net present value (NPV) was \$3.2 million after fifteen years. This yielded an internal rate of return (IRR) of 30.1% over fifteen years, when considering the dual benefits of material sales and the avoidance of the cost of OB/OD.

Overall, the recovery of RDX does not represent an economic benefit over the status quo of open burning or detonation of energetic materials. The original capital investment is never recovered over the fifteen year time period, as the estimated revenues from product sales of recovered RDX are not sufficient to cover the cost per pound for the recovery process.

The subscale pilot plant provides a cost-effective alternative to the open burning or detonation of energetic materials, wherein the market price or production cost of virgin materials is relatively high, as is the case for HMX. The cost avoidance associated with OB/OD is currently a factor in the overall financial results of both materials, and could have a materials impact in the future should cost increase significantly.

- **Stakeholder/End-User Issues.** The end-users of this technology will be DoD and DOE, who are seeking a new source of HMX at a reduced cost. The reduced cost of recovered HMX would make it feasible to use HMX in many munitions that currently do not meet Insensitive Munitions (IM) requirements. For example, the reduced cost of HMX should make it more feasible to replace Comp B with PBXN-110. Also, the recovered HMX would provide the DOE an alternative domestic source of HMX for use in PBX-9501, which is currently in its initial stage of formulation. If this technology can be transitioned to a large-scale production, a new means of disposing of HMX-containing munitions will be available. The cost of OB/OD will be eliminated, and eventually DoD will reap the benefits through the sale of recovered HMX.

2.0 TECHNOLOGY DESCRIPTION

2.1 TECHNOLOGY DEVELOPMENT AND APPLICATION

This technology was developed to provide DoD with a means of recovering high-value energetic materials. It eliminates the need to dispose of these items by open burning or open detonation (OB/OD), which is detrimental to the environment and a waste of recoverable resources. The technology has been demonstrated for two different recovery processes. HMX was recovered from LX-14 and RDX was recovered from Composition A-3.

The processes are quite simple. The explosive materials consist of a high explosive and a binder. The materials are loaded into a reactor where the binder may be separated from the explosive by solubilization or melting. The binder and explosive phases are then separated by centrifugation. The by-product of the HMX recovery process is Ammonium Nitrate/Polymeric Fuel (ANPF), which has properties similar to those of Ammonium Nitrate/Fuel Oil (ANFO), and may be used in similar applications. The RDX recovery process by-product is explosives-contaminated wax, which must be disposed of as hazardous waste. The calcium chloride/surfactant solution used as a solvent in the RDX recovery process may be recycled.

2.2 PROCESS DESCRIPTION

The HMX/RDX recovery plant was set up at Ft. Wingate Army Depot, Gallup, New Mexico. The processing was done in Building 542, and a remote control room unit was constructed near Building 516. See Figure 2.1 for a map of the area.

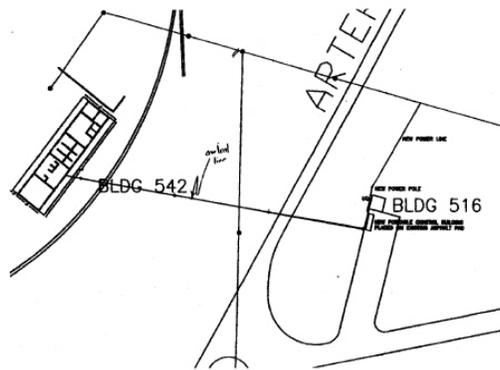


Figure 2.1. Map of Site.

Three operators were required to run the demonstration plant, under the direction of a process engineer. Two operators were directly involved with the recovery plant at Building 542 while the third operator split time between the operations at Building 542 and the ANPF recovery drum dryer operations housed on the chemical pad north of Building 528. Operators required the ability to interpret process data and communicate process results, and demonstrated the mechanical aptitude to perform routine maintenance and up-keep tasks. They required no specialized training to operate the plant.

Figure 2.2 shows the general process flow diagram for the sub-scale HMX/RDX recovery process.

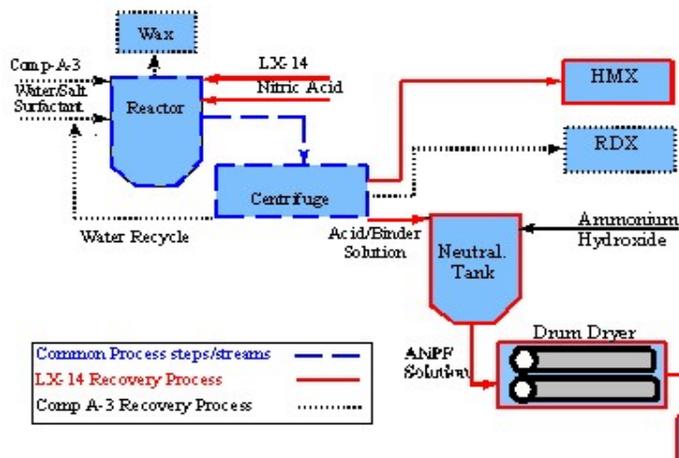


Figure 2.2. Sub-Scale HMX/RDX Recovery Process Flow.

The plant was designed to process 150 lbs per day of LX-14 or Comp A-3. For the processing of LX-14, nitric acid emissions were required to be within regulatory limits, and were monitored accordingly by nitric acid sensors installed in the NO_x monitoring system. The system is only monitored by operators during times when work or heat energy is input to the process. Overnight acid digestions do not require around the clock monitoring, which reduces labor requirements and makes the system easy to use.

Operational parameters for the systems demonstrated were developed through the careful scale-up from the bench-top, to the pilot plant, to the demonstration level plant. These parameters are given in Tables 2.1 and 2.2. Several iterations of the critical parameters were made in order to achieve a safe, productive, and efficient system that yielded a high quality product.

2.2.1 Recovery of HMX from LX-14

LX-14 is a high explosive composed of 95% HMX and 5% Estane™ binder. 150 lbs of LX-14 were manually loaded into the 80-gallon stainless steel reactor, which had been charged with concentrated nitric acid (1 kg LX-14: 1 liter of nitric acid). The resultant mixture was allowed to react overnight to allow the Estane™ binder to be solubilized in the nitric acid. The reactor temperature was elevated to 70 °C after the overnight digestion and allowed to react at the elevated temperature for one hour.

The slurry was then introduced to a basket centrifuge, where the acid/binder slurry was removed from the HMX using a polypropylene filter bag. The HMX was retained in the filter bag while the binder acid slurry was pumped to a neutralization tank.

The effluent was neutralized with ammonium hydroxide to a pH ranging from 6-7. When the spent nitric acid/degraded polymer is neutralized with ammonium hydroxide, an aqueous fuel/oxidizer mixture results. This aqueous solution was transferred in tanks from Building 542 to Building 528 for processing. The solution was then introduced to a steam-heated double drum dryer with the purpose of yielding, through water evaporation, a compound salt identified as Ammonium Nitrate/Polymeric Fuel (ANPF).

Table 2.1. Targeted Process Parameters for HMX Recovery from LX-14.

Parameter	Range
Batch weight	150 +/- 5 lbs
Digestion acid	18 +/- 0.5 gal
Digestion time (passive)	12 to 24 hrs
Digestion temperature	158 +/- 5 °F
Digestion time (at elevated temp.)	1 hr +/- 5 min
Agitation speed	400 +/- 50 rpm
Centrifuge speed	825 +/- 75 rpm
Rinse acid	2 +/- 1 gal
Rinse water, reactor	50 +/- 5 gal
Rinse water, centrifuge	200 +/- 5 gal
Centrifuge duration	30 +/- 10 min
ANPF solution pH	6.5 +/- 0.5
Neutralization agitation speed	500 +/- 100 rpm
Neutralization tank temperature	75 +/- 25 °F
Drum dryer throughput rate	70 +/- 10 lb/hr

Table 2.2. Targeted Process Parameters for RDX Recovery from Comp A-3.

Parameter	Range
Batch weight	75 +/- 5 lbs
Process water	45 +/- 1 gal
Salt (CaCl ₂)	150 +/- 5 lbs
Surfactant (Tween 20)	18.8 lbs +/- 0.5 lbs
Process temperature	176 +/- 2 °F
Separation time (at elevated temp.)	0.5 +/- .25 hrs
Agitation speed	300 +/- 50 rpm
Quench water	10 +/- 2 gal
Centrifuge speed	825 +/- 75 rpm
Rinse water	50 +/- 20 gal
Centrifuge duration	30 +/- 10 min

The recovered HMX retained in the centrifuge filter bag was rinsed with water to remove any acid residue present from the digestion process. The rinse water was introduced into the neutralization tank as well. The HMX was manually removed from the centrifuge, loaded into plastic packaging bags, and 15 wt-% water was added in order to desensitize the nitramine. The reactor and centrifuge were rinsed with acid. The acid used for this rinse was recycled for use in the subsequent batches of LX-14.

2.2.2 RDX Recovery from Comp A-3

Composition A-3 is a high explosive mixture consisting of 91% RDX and 9% wax binder. For the demonstration of RDX recovery from Comp A-3, the system was configured differently than for the HMX recovery process. A pre-mixed solution of water, calcium chloride salt, and Tween 20 surfactant was introduced into the 80-gallon stainless steel reactor. The solution ratio of ingredients was 1:0.4:0.05 by weight of water: salt: surfactant.

75 lbs of Comp A-3 were loaded, with a 5:1 by weight ratio of water solution to Comp A-3. The reactor contents were stirred and the reactor temperature was raised to 80 °C. After the target temperature was reached, the reactor was held at temperature for 30 minutes. At this time, due to the density differences between the RDX (1.6 g/cc), the salt solution (1.18 g/cc), and the wax (0.9-1.0 g/cc), a 3-layer stratification resulted. The higher density RDX settled to the bottom of the reactor while the lower density wax floated to the top of the solution. Mechanical means were employed to skim off the cooled layer of wax as it reached the top of the solution. Quench water was added to the reactor to further cool the solution, in order to extract additional wax floating on top of the solution. After the majority of the wax was removed by mechanical means, the smaller wax particles were removed via aspiration methods using a vacuum pump.

When all of the wax was removed from the reactor, the remaining solution was discharged into the basket centrifuge where the RDX was retained and the water/salt/surfactant solution was pumped to a recycle tank for use in subsequent process batches. The recycled water solution was checked for density reduction caused by the attrition of salt and replenished as necessary for the next processing batch. The RDX was rinsed with fresh water to remove any water solution residue present from the processing. The RDX was removed from the centrifuge manually and packaged.

2.3 PREVIOUS TESTING OF THE TECHNOLOGY

Initial laboratory and bench scale testing of this technology began under the Small Business Innovation Research (SBIR) program. Under a NSWC Crane Phase I SBIR contract, TPL, Inc. showed the feasibility of using common mineral acids to separate the binders from the explosives in various plastic bonded explosives [3]. Under a Phase II SBIR contract, TPL, Inc. developed a pilot plant that demonstrated the ability to solubilize Estane™ binder and remove the HMX from LX-14 [4].

2.4 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

Currently, there are no proven capabilities within DoD to recover HMX and RDX. This technology has been demonstrated for two different energetic materials, but may be adapted to a wide variety of other materials.

2.4.1 HMX Recovery Process

The major advantage of this technology for recovering HMX is that the cost per pound for reclaimed HMX is lower than the cost of the virgin material. In some cases, the cost difference is low enough to feasibly substitute HMX for TNT in non-precision weapons. See Section 5.0 Cost Assessment for factors influencing costs. There are weapons in the inventory that do not pass insensitive munitions requirements. These usually employ a TNT-based explosive, which does not meet cook-off requirements. If these weapons utilized an HMX-based explosive formulation, meeting

insensitive munitions requirements would be less of an obstacle, as HMX has better cook-off characteristics than TNT-based explosives. Another advantage of this process is that it would provide the DoD with a domestic source of HMX. Currently, DoD purchases HMX from foreign vendors.

One of the weaknesses of the HMX recovery process is the use of concentrated nitric acid to dissolve the binder. There are many hazards associated with the handling of this material, as it is highly corrosive, highly toxic by inhalation, and a strong oxidizing agent. However, the resulting waste stream may be further processed to obtain a valuable by-product. The principal by-product of the HMX recovery process is spent nitric acid, which contains a degraded Estane™ polymer. This organic material is a fuel, albeit relatively simple. After the water is removed, via an evaporation process, the resulting material is analogous to ammonium nitrate/fuel oil (ANFO), a commonly used blasting agent. Consequently, TPL has named this byproduct ANPF, an abbreviation for ammonium nitrate/polymeric fuel. ANPF has detonation properties similar to ANFO. ANPF has been formulated with a granular plastic bonded explosive to make a unique metal bonding explosive BondEx A™ [5]. The military application for this technology is the lining of gun tubes with refractory metals in order to eliminate corrosive wear. TPL in a Phase I SBIR with the Army Research Organization demonstrated that 120 mm diameter tubes could be clad. A subsequent Phase II program has been awarded to TPL and medium caliber liners are currently being investigated [6,7]. TPL will be teaming with General Dynamics Armament Systems and/or others to fabricate gun tubes for service life testing.

2.4.2 RDX Recovery Process

A major advantage to this technology for the recovery of RDX is that there are no safety handling issues associated with using the water/surfactant/salt solution. The solvent is non-toxic, non-corrosive, and 100% recyclable. Also, the process would provide DoD a domestic source of RDX, which is currently procured from foreign vendors. One of the weaknesses of this process involves the disposition of the wax binder. Because the wax will contain residual RDX, steps will have to be taken to ensure its safe disposal.

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3.0 DEMONSTRATION DESIGN

3.1 PERFORMANCE OBJECTIVES

The process will be evaluated using the following criteria (Table 3.1), as delineated in the technology demonstration plan for the project [8]:

Table 3.1. Performance Objectives.

Type of Performance Objective	Primary Performance Criteria	Expected Performance Metric	Actual Performance Objective Met?
Quantitative	1. Process Waste - HMX recovery - RDX recovery	No waste	Yes No
	2. Cost - Recovered HMX - Recovered RDX	Less than cost of virgin material	Yes No
	3. Throughput	150 lbs / day	Yes
	4. Purity	>97%	Yes
Qualitative	1. Safety	Remote operation	Yes

The objective of the demonstration was to recover 150 lbs/day of RDX from Composition A-3 and HMX from LX-14 at 97% purity or greater to satisfy the requirements of potential buyers. The HMX should meet the requirements of MIL-H-45444B, and the RDX should meet the requirements of MIL-R-398C. The costs for accomplishing these objectives with a maximum of safety should be optimized. The by-products of the process should be recycled or recovered for another use, if possible. These performance criteria were met for the recovery of HMX, but the cost for the recovery of RDX was too high.

3.2 SELECTION OF TEST SITE/FACILITY

The site selected was Building 542 at Ft. Wingate Army Depot, Gallup, New Mexico. This site was selected because it was an available existing structure and it had adequate available floor space. There were adjacent locations available for performing remote operations, and it had the appropriate siting for energetic material handling.

The site had an existing infrastructure of utilities such as steam, air, water, and power, which were necessary for process operation. The building's reinforced concrete wall construction and its location from the main access road made it an excellent facility for the safe processing of the materials. In addition, utilizing Building 542 has provided a permanent processing facility that will easily transition into production and serve as the main facility for the recovery of HMX from surplus energetic materials.

3.3 TEST FACILITY HISTORY/CHARACTERISTICS

The site selected for the processing of these energetic materials was previously designed and used for the handling of munitions and other energetic material devices. The distance of the building from the road allows for handling up to 500 lbs of Class 1.1 materials, based on interline distances. A control room for remote operation was installed adjacent to Building 516. The existing power poles between Building 542 and Building 516 allowed for a reduced number of control line poles to be placed between the two buildings to support the process control lines and video equipment lines.

Reinforced concrete separating walls also allowed for isolating distinctly different process operations, such as the acid and base process for the recovery of HMX from LX-14, as well as creating natural divisions for in-process raw material, final product, and process materials storage.

A portable unit was set up adjacent to Building 516 to serve as a remote control room. This unit was outside the explosives quantity-distance arc for the operations in Building 542. Power and control lines were installed to support this remote operating facility. Remote cameras within Room 8 of Building 542 allowed operators to monitor all activities from a safe distance in the control room.

3.4 PHYSICAL SET-UP AND ORGANIZATION

The equipment layout is shown in Figure 3.1. Power was added to Building 542, Room 8 to support the processes associated with the demonstration plant. The electrical power service installed to operate the demonstration plant is as follows:

480 VAC/3 phase/40 amps
208 VAC/single phase/20 amps
110 VAC/single phase/130 amp

Other utility usage for the plant includes 15-psi steam for reactor heat-up and building heating. Air is supplied at 90 psi to operate the pneumatically actuated diverter valves, flow control valves, and diaphragm pumps. Plant-supplied water is used for processing, as well as for cleanup of the building and equipment.

Maintenance requirements for the system include a routine preventive maintenance schedule for all of the major equipment such as the air compressor, reactor agitator motors, centrifuge, flow control valves, and process instrumentation. Standard maintenance practices for all equipment are needed to ensure the reliable performance of the equipment on a daily basis. Replacement parts must be kept on hand for high-use items. Minimizing downtime due to equipment wear or malfunction will support the operation of process and maintain the anticipated throughput rates over extended times. Parts required as spare parts inventory include back-up instrumentation, pumps, diverter valves, solenoids, centrifuge bags, and process-wetted gaskets and seals.

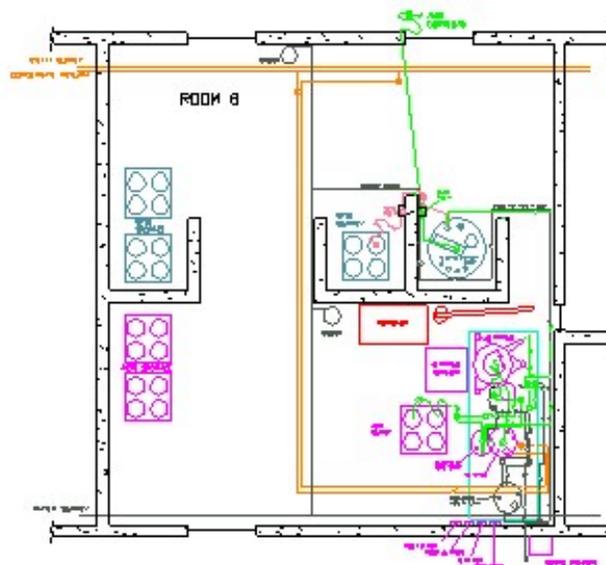


Figure 3.1. Process Layout for Building 542.

Operation of the plant for the demonstration was conducted according to the schedule in Table 3.2. Times listed were typical for the plant operation with little deviation experienced in the schedule. The total batch times for HMX and RDX recovery were 24 hours and 6 hours, respectively.

The only waste stream for the process is the wax removed from the recovery of RDX from Comp A-3. The final disposition of the wax is currently under investigation. For this reason, the RCRA classification is unknown at this time, and consequently the cost of future reuse or disposal is unknown.

3.5 SAMPLING/MONITORING PROCEDURES

The sampling plan involved a direct measurement of the product quality using several laboratory techniques. A sample was collected from the centrifuge cake for each batch of material produced. A total of three samples were collected from the RDX process, while two samples were collected for the HMX process. Analytical data were collected for the following tests: purity, melting point, physical appearance, and batch yield. The sampling procedure for the demonstration batches was performed as follows:

Table 3.2. HMX/RDX Recovery Sub-Scale Plant Demonstration Schedule.

Date	Time	Event
Sunday, 21 March	1:00 pm	Weigh-up/load LX-14 into reactor
Monday, 22 March	7:00 am	Check-out/heat-up
	11:00 am	Download HMX
	11:30 am	Download ANPF
	12:00 pm	Bay cleanup
	3:00 pm	Weigh-up/load LX-14 into reactor
Tuesday, 23 March	7:00 am	Check-out/heat-up
	11:00 am	Download HMX
	11:30 am	Download ANPF
	12:00 pm	Bay cleanup
	1:00 pm	Transition process for Comp A-3
	5:00 pm	Shutdown
Wednesday, Thursday*, 24 & 25 March	7:00 am	Check-out/weigh-up/load Comp A-3
	8:00 am	Heat-up
	9:30 am	Quench/Skim
	11:00 am	Centrifuge
	12:00 pm	Download RDX
	1:00 pm	Check-out/weigh-up/load Comp A-3
	2:00 pm	Heat-up
	3:30 pm	Quench/Skim
	5:00 pm	Centrifuge
	6:00 pm	Download RDX
7:00 pm	Shutdown	

*The fourth batch of Composition A-3 was not processed due to mechanical problems relating to the boiler used to heat the process. It was determined that the previous three batches were sufficient to demonstrate the technology effectively.

1. Obtain one 20-gram sample from each centrifuge cake. Place sample in a glass vial, sealed with a screw-on lid. Label each vial with lot number, date, material type, and sample location.
2. Evaluate visual appearance of the centrifuged material and record in "Observations" section of the process form.
3. Test each sample per mil specs for the properties listed above.

3.6 ANALYTICAL PROCEDURES

The following analytical methods were used and performed by TPL, Inc.:

Purity - High performance liquid chromatography (HPLC) was used to measure the HMX content and RDX content in the samples provided.

Melting Point - Samples from each batch were evaluated in a capillary melting point indicator per the specifications given in Section 3.1.

Physical Appearance - Physical appearance was noted by the operators at the time of manufacture as well as verified during the analytical testing phase. This was a qualitative measurement of the product's physical appearance.

Batch Yield - This was a calculated value based on the values recorded for the initial batch weight, recovered product weight, and moisture content of the final product.

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4.0 PERFORMANCE ASSESSMENT

4.1 PERFORMANCE DATA

Material properties were measured for both the HMX and RDX from the demonstration batches. The analytical laboratory of TPL, Inc. performed the material properties analyses. The data is given in Table 4.1. Due to cost and schedule constraints, analyses were performed on only one sample from each batch. These samples were assumed to be representative of their respective batches.

Table 4.1. Material Properties for Demonstration Batches.

Lot No.	Melting Pt. (°C)	Purity* (%)	Yield (%)	Physical Appearance
HMX ^a	277			White
5	278	98.13	99.5	White
6	278	99.60	98.5	White
RDX ^b	203.5			White
8	199	98.01	97.7	Gray/white
9	198	98.97	95.5	Gray/white
10	197	97.74	95.5	Gray/white

^a Pure HMX

^b Pure RDX

* values represent total of RDX or HMX present in product

4.2 PERFORMANCE CRITERIA

The following criteria were established for the evaluation of the demonstration plant. These criteria were established to accurately assess the efficiency, economics, and safety of the plant.

- a. **Process Waste**-Process waste should not be present in the HMX or RDX recovery processes. All effluent streams should be recycled into reusable feedstocks for the process, or reprocessed into alternate commercial products.
- b. **Cost**-The HMX and RDX recovery process should not exceed the cost of virgin material which is readily available. This recovery process will provide the DoD with another source of HMX and RDX, while providing an environmentally sound method of disposal of excess energetics.
- c. **Throughput**-The anticipated recovery load should be 150 lbs for both HMX and RDX.
- d. **Purity**-In order for the reuse of HMX and RDX to be possible, the recovered products must meet purity levels greater than 97%, as well as meet other military specifications.
- e. **Safety**-The main hazards involved in this process are the handling of energetics and the large volumes of acid used in HMX recovery. The process will be operated remotely to reduce the exposure of energetics and chemicals to operating personnel, and personal protective equipment will be utilized for handling acids and other materials.

- f. **Factors Affecting Technology Performance**-Several processing parameters have an effect on the resultant product yield, quality, and performance. Evaluation of the process parameters have shown the temperature of the digestion, mass of the solution, quantity of rinse water, centrifuge cycle time, digestion time, and centrifuge load have effects on the resultant product.
- f. **Reliability**-The proposed process is considered robust in that it is not affected by extreme changes in environmental conditions. Potential or anticipated equipment breakdowns will be planned for by maintaining a reserve of spare parts. In particular, long lead-time parts will be reserved in stock to minimize downtime. All equipment will be subjected to daily operator check-out prior to the start of operations and scheduled periodic maintenance to insure the equipment is evaluated thoroughly to minimize the chance of major equipment failure during the processing.
- h. **Ease of Use**-Two operators will run recovery plant, with an additional operator for by-product recovery operations. Crew will be cross-trained to allow for interchangeability of staff, and trained back-up will be available on site in the event of operator absence.
- i. **Versatility**-The technology can be used on other explosives with additional pilot plant evaluation.
- j. **Off-the-Shelf Procurement**-All equipment will be commercially available.
- k. **Maintenance**-Periodic maintenance schedules will be utilized to service equipment. Journeyman tradesman or an individual with basic understanding of the pipefitting and electrical maintenance trades will be suitable for this operation.
- l. **Scale-up Issues**-Scale-up issues associated with the process involve sizing the equipment properly to obtain similar heat transfer and mass transfer characteristics. Proper scaling also involves designing manageable operator interfaces to the process. Material handling vessels, feedstocks, and plant lay-out all must be considered when increasing the plant capacity. Handling increased quantities of explosives is also a concern during process scale-up. Minimizing the quantities of explosives in one location is vital to the protection of the personnel, equipment, and facilities. In addition, propagation of an event surrounding explosive materials must be reduced or eliminated when dealing with larger quantities of materials.

Table 4.2 summarizes how these criteria were met by the technology as demonstrated.

4.3 DATA ASSESSMENT

As shown in Table 4.1, the performance criteria for achieving > 97% purity was met for HMX and RDX. The batch yields for both processes were > 95%. The data indicates that the recovered HMX meets the melting point, batch yield, and physical appearance criteria needed for potential resale. While the material property criteria were nearly met for RDX, the RDX recovery process did not meet the reutilization criteria, because of its wax by-product.

Table 4.2. Performance Assessment and Performance Confirmation Methods.

Performance Criteria	Expected Performance (pre demonstration)	Performance Confirmation Method	Actual Performance (post demonstration)
PRIMARY CRITERIA (Performance Objectives) (Quantitative)			
Process Waste - HMX Recovery - RDX Recovery	No waste	Observation	- None - Wax
Cost - HMS Recovery - RDX Recovery	Less than cost of virgin material	Cost analysis	- Less than cost of virgin material - More than cost of virgin material
Throughput	Process 150 lbs of LX-14 and Comp A-3	Weight observation	Process 150 lbs LX-14 and Comp A-3
PRIMARY CRITERIA (Performance Objectives) (Qualitative)			
Purity	>97%	MIL-H-45444B MIL-R-398C	>97%
Safety - remote operation	Remote operation	Observation	Remote operation
SECONDARY PERFORMANCE CRITERIA (Qualitative)			
Factors Affecting Technology Performance - temperature of digestion - mass of solution - quantity of rinse water - centrifuge cycle time - digestion time - centrifuge load	See purity above	Operation at various parameters	See purity above
Reliability	Few process upsets/ No breakdowns	Experience from demonstration operation	Few process upsets/ No breakdowns
Ease of Use	Only three operators required and no special education is required	Experience from demonstration operation	Only three operators required and no special education is required
Versatility	Use with other explosives	Experience from demonstration operation	LX-14 Comp A-3
Off-the-Shelf Procurement	Utilize commercially available equipment	Experience from demonstration operation	Utilize commercially available equipment
Maintenance	Periodic maintenance schedules	Experience from demonstration operation	Periodic maintenance schedules
Scale-up Issues - engineering - material handling	Increase heat and mass transfer Increase quantities of explosives	Experience from demonstration operation	HMX demonstration scale is acceptable. RDX scale will need modification.

The HMX recovery process successfully met all performance criteria. There is no waste generated in this process since the by-products are reutilized as a blasting agent. The RDX process generates a wax by-product that must be considered a hazardous waste. The salt solution settling unit allows the salt solution to sit undisturbed for a period of time so that any RDX fines may settle to the bottom of the unit. After the time has elapsed, the majority of the salt solution is sent to the recycled salt solution holding tank. The settled RDX is recycled through the centrifuge for separation. It is unknown whether the minute amounts of RDX left in the waste stream must be considered hazardous waste. If so, then hazardous waste disposal is an issue. The demonstration plant achieved purity levels greater than 97% and batch yields greater than 95% for both the HMX and RDX processes.

Safety issues were resolved through the use of remote operations wherever possible. As evidenced from the demonstration plant, the reliability, ease of use, versatility, and maintenance criteria were met. This demonstration showed that the plant could be used for at least two different kinds of energetics. All parts needed for the plant construction were procured from commercial sources and therefore, the plant is amenable to quick replacements and repairs. No scale-up issues were directly addressed for the HMX process, because it will be operated commercially at the demonstration plant scale. The scale-up of the RDX plant was addressed and it was found that some modifications were required. The prototype plant design identifies an improved separation technique, which includes a vortexial separator, dual-stage centrifuge, and an oil skimmer. The upgraded design will include the addition of two more reactors, for a total of three reactors. The prototype plant will be located at a government installation to facilitate the direct transfer of Composition A-3 from a waterjet washout facility to the RDX recovery plant.

4.4 TECHNOLOGY COMPARISON

Current methods of disposal for HMX- and RDX-containing munitions are OB/OD and incineration. Both methods involve the destruction of the HMX and RDX, and thus the loss of a valuable resource. In addition, both methods are considered to be environmentally unsound due to the release of toxic gases into the atmosphere.

Other technologies for the repeated extraction of HMX or RDX from the feedstocks provided have been investigated. In 1998, research on the use of supercritical fluids for the removal of RDX from Composition B was completed [9]. This research focused on the separation of TNT and wax from RDX using supercritical carbon dioxide. Supercritical CO₂ is considered to be an ideal solvent due to its non-toxic, non-corrosive properties. This method proved to be successful but required high temperature and high pressure conditions. The RDX that was recovered was discolored, and further chemical analysis would be required to determine its suitability for reuse.

Another comparable technology involves the use of liquid ammonia to extract, separate, and recover rocket propellant and warhead ingredients within a closed-loop operation [10]. This technology has been used to recover HMX from Class 1.1 motors. This process requires several aliquots of liquid ammonia to solubilize the HMX, because the solubility of HMX in liquid ammonia is about 10-30% by weight. In addition, a plastic by-product remains from the liquid ammonia process, which must be treated to assure no residual HMX remains. It is also noted that the recovered HMX requires recrystallization prior to reuse.

The technology demonstrated by TPL, Inc. is unique in producing HMX or RDX without significantly changing the particle size or shape in the process. The process' performance is superior

to other competing technologies based on its simplicity, effectiveness, and the use of common, readily available process equipment. The technology is inherently safe to personnel due to the remote operation of the process, the high solvent to feed ratio, and the relatively low processing temperatures required to affect a proper extraction of the product from the feedstock. Because off-the-shelf processing equipment is employed in the process, replacement parts are not difficult to obtain, and repair costs are not exceedingly high. The simplicity of the process allows for operator interaction and understanding of each process step. Any problems that occur are less complex to identify and repair than with other more sophisticated systems. This process has proven to be scalable from the bench-top to the demonstration scale with minimal problems encountered in reaching the program objectives.

System reliability is dependent solely on the function of the reactor heating mechanism, agitation, and centrifuge performance for the HMX recovery process. The process is PLC controlled, so the reactor temperature is maintained within 5 degrees of the set-point temperature. Agitation speeds are controlled as well, with less than 10% variance in any agitation speed. Centrifuge speed is fixed based on the drive system and fluctuates only when load weights change during the continuous centrifugation process cycle. For the RDX recovery process, wax removal techniques represent the largest degree of variation due to its manual nature. Skimming of the large pieces of wax from the top of the reactor works sufficiently well as a coarse removal technique. Aspirating the finer wax remnants from the solution represents a variable that removes, along with the wax, a small amount of RDX fines from the system. This variable in the system was not optimized for the demonstration due to processing equipment constraints associated with handling two distinctly different feedstocks. Improvements to the wax removal operations are expected and planned for future dedicated Composition A-3 plant designs. It is anticipated that a Voraxial separator will be utilized for the removal of wax. This separator works by rotating the solution rapidly in a pipe. The heavier materials (the salt solution) are pushed to the outer walls and the lighter materials (the wax) stay in the center of the pipe. At the end of the pipe, the inner and outer layers are physically separated. The water will be sent to the salt solution settling tank and the wax will be collected in a container.

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5.0 COST ASSESSMENT

5.1 COST REPORTING

An Environmental Cost Analysis Methodology (ECAM) report was prepared by Concurrent Technologies Corporation [11]. The analysis was conducted at Fort Wingate by using data collected on site and at TPL, Inc.'s offices in Albuquerque, New Mexico, during a site visit February 5-7, 2001 [12].

The HMX/RDX recovery and reuse technology is compared to the current practice of open burning/open detonation (OB/OD), historically used to dispose of excess munitions. Energetic materials are either burned in an open pit or incinerator and the ash is disposed of as hazardous waste in a designated landfill or materials are detonated and buried in-situ. The analysis assumed an OB/OD cost per pound of \$8.50 including labor and disposal costs. This value is based on the actual disposal costs at TPL, Inc. Based on a yearly production of 33,000 lbs of recovered HMX, the cost avoidance achieved by not burning the energetic materials is \$280,500. RDX, with a lower production capacity of 15,400 lbs per year would result in a cost avoidance of \$130,900. The sale of ANPF blasting agent by-product was not included in the ECAM cost analysis. The following tables summarize the annual costs of the HMX process (Table 5.1), RDX process (Table 5.2), and the capital costs for both processes (Table 5.3).

Unit costs were estimated using the bottom-up approach for each of the resources consumed by the direct inputs and outputs identified by TPL, Inc. In the bottom-up approach, unit costs are obtained directly from records or files already available, or are estimated by personnel familiar with the process.

For this analysis, a study period of 15 years was chosen, and a discount rate of 4.0 percent was used. Because a 15-year life was chosen, it was necessary to calculate a rate between the 10- and 30-year maturity rates. Interpolating between the 10-year rate of 4.0 percent, and the 30-year rate of 4.2 percent, the 15-year rate is calculated to be 4.0 percent. Table 5.4 summarizes the cost comparison of the innovative technology at 150 lbs sub-scale and OB/OD.

Table 5.1. Direct Resources Annual HMX 150 lb. Sub-Scale Plant Costs.

Direct Resource	Total Quantity Consumed	Unit Cost	Annual Cost
Non Environmental			
Material recovery (Throughput)	33,000 lbs	---	---
Labor (Operators)	3,520 hrs	\$24.27/hr	\$85,430
Supervision	350 hrs	\$44.13/hr	\$15,534
Material Handling	212 hrs	\$18.72/hr	\$3,960
Maintenance	350 hrs	\$35.30/hr	\$12,426
PreProcessing/Packaging Labor	212 hrs	\$18.72/hr	\$3,960
Utilities	---	---	\$8,250
Repair Parts	---	---	\$15,000
Nitric Acid	4,382 gals	\$2.56/gal	\$11,220
Drums and Liners	220 drums	\$8.65/drum	\$1,903
Water	59,400 gals	NA	\$0
Ammonium Hydroxide	2,185 gals	\$7.25/gal	\$15,840
Sub-Total			\$173,523
Environmental			
Training	93 hrs	\$44.13/hr	\$4,118
Lab Analysis	220 tests	\$82.50/test	\$18,150
Hazardous Waste ^a	373 lbs	\$8.85/lb	\$3,300
Sub-Total			\$25,568
TOTAL			\$199,091

^a A cost is associated with the waste such as cardboard packaging, plastics, etc.

Table 5.2. Direct Resources Annual RDX 150 lb. Sub-Scale Plant Costs.

Direct Resource	Total Quantity Consumed	Unit Cost	Annual Cost
Non Environmental			
Material recovery (Throughput)	15,400 lbs	---	---
Labor (Operators)	3,520 hrs	\$24.27/hr	\$85,430
Supervision	350 hrs	\$44.13/hr	\$15,534
Material Handling	212 hrs	\$18.72/hr	\$3,960
Maintenance	350 hrs	\$35.30/hr	\$12,426
PreProcessing/Packaging Labor	212 hrs	\$18.72/hr	\$3,960
Utilities	---	---	\$3,850
Repair Parts	---	---	\$15,000
Reagents	31,570 lbs	\$0.40/lb	\$12,628
Drums and Liners	102 drums	\$8.65/drum	\$888
Water	59,400 gals	NA	\$0
Sub-Total			\$153,676
Environmental			
Training	93 hrs	\$44.13/hr	\$4,118
Lab Analysis	220 tests	\$82.50/test	\$18,150
Hazardous Waste ^a	348 lbs	\$8.85/lb	\$3,080
Sub-Total			\$25,348
TOTAL			\$179,024

^a A cost is associated with the waste such as cardboard packaging, plastics, etc.

Table 5.3. Equipment Capital Costs.

Direct Resource	Total Cost
Non-Environmental	
Equipment	\$545,689
Installation	\$352,455
Engineering	\$444,239
TOTAL	\$1,324,383

Table 5.4. Cost Comparison.

Cost Type	OB/OD	HMX Recovery	RDX Recovery
Capital Costs		\$1,342,383	\$1,342,383
Annual Operating Costs:			
Amortization		\$89,492	\$89,492
Labor	\$148,607	\$121,310	\$121,310
Materials		\$28,963	\$13,516
Utilities		\$8,250	\$3,850
Lab Analysis		\$18,150	\$18,150
Repairs		\$15,000	\$15,000
Other	\$132,000	\$7,418	\$7,198
Annual Sub-Total	\$280,607	\$288,583	\$268,516
Cost per pound	\$8.50/lb	\$8.74/lb	\$17.44/lb
Revenue		\$10.00/lb HMX	\$3.00/lb RDX
Cost Avoidance:		\$8.50/lb OB/OD	\$850/lb OB/OD
Sales	\$0	\$330,000	\$46,200
OB/OD	\$0	\$280,607	\$129,640
Sub-Total	\$0	\$610,607	\$175,840
Total (1st Year) Net Savings/(Costs)		\$322,024	\$(-92,676)

Based on the results, the recovery of HMX from LX-14 feedstock is economical. The net present value (NPV), payback period, and internal rate of return (IRR) are positive indicators of the financial benefits of the recovery of HMX energetic materials. For the HMX 150-lb sub-scale plant, a life-cycle cost savings of \$3.2M over a 15-year period is calculated, with a payback period of 3.6 years with OB/OD benefits and of 13.5 years without OB/OD benefits. The internal rate of return is 30.1% over the life of the project. The life-cycle cost of OB/OD would be approximately \$4.2M. The uncertainty for the capital equipment and OB/OD cost estimate and HMX revenue sales is calculated to be $\pm 30\%$. The other costs associated with this process were too small to impact the NPV in most cases, less than 1% of the total costs. The recovery of RDX did not produce a positive return on investment and the original capital investment cost is not recovered.

5.2 COST ANALYSIS

The cost analysis was performed by Concurrent Technologies Corporation, using P2/FINANCE software. For the HMX recovery process the NPV was \$3.2M and the annual cost savings was calculated to be \$130,909. The calculated payback period and IRR were calculated to be 3.6 years and 30.1%, respectively.

5.2.1 Sensitivity Analysis

There are several cost drivers for this process. Since the cost drivers with the greatest variability and potential impact were the assumed selling price of the HMX, the labor costs, and disposal costs associated with the baseline OB/OD process, a sensitivity analysis on the HMX recovery process was performed based on these parameters (Figure 5.1). The relatively high capital costs of the

stainless steel process plant and the explosion-proof facility are also cost drivers, but are not expected to vary significantly.

Changing the market price of the HMX and/or cost avoidance benefits associated with the OB/OD will impact the forecasted NPV. A total of 2,000 trials were conducted using the Monte Carlo statistical analysis technique, randomly changing the estimated price for sales of HMX from \$7.54 to \$12.87 per pound. For the labor and disposal costs associated with OB/OD, the range assumed values from \$10.79 to \$46.26 per pound. The analysis shows that the change in NPV is correlated about equally with changes in the market price for the material and the cost of OB/OD.

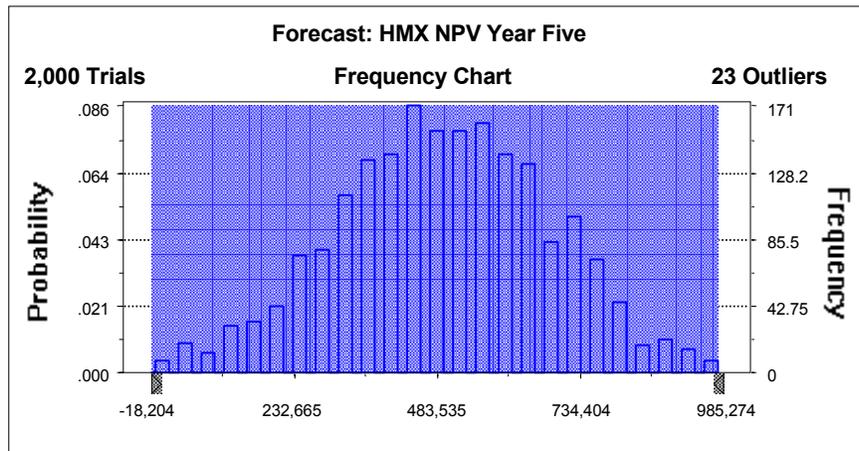


Figure 5.1. Sensitivity Chart for HMX Process.

5.2.2 DoD Cost Savings

It is forecast that between FY 2000 and FY 2009, over 96,000 pounds of LX-14 will be placed in the Tactical Missile Resource Recovery and Disposition Account [1]. This calculates to approximately \$960,000 worth of material resale as well as a cost avoidance of \$816,000. These calculations are based on a HMX resale value of \$10/lb and the disposal cost of \$8.50/lb.

5.3 COST COMPARISON

The cost for OB/OD is \$8.50/lb. The operating costs for the HMX and RDX recovery processes for the first year are \$8.74/lb and \$17.44/lb respectively, see Table 5.4. The operating cost per pound for the recovery of RDX is much higher than for HMX due to the limited processing capacity for RDX recovery. For this reason, the cost to recover RDX is significantly more than for OB/OD. Revenue from the sale of the recovered materials decreases the net cost for the recovery of both materials, but since the market price of RDX is low, it is still more expensive to recover and sell RDX than to dispose of it by OB/OD. A net cost savings is realized when the recovered HMX is sold.

Because virtually zero waste was generated, there are no permit and wastewater treatment/disposal costs associated with this innovative technology. Also, HCl emissions associated with OB/OD are eliminated when the recovery process is used instead of OB/OD, yielding an environmental benefit.

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6.0 IMPLEMENTATION ISSUES

The major factor affecting project cost is labor. Three operators are required to operate the plant at all times. One operator is required to transport the ammonium nitrate by-product to a different building for further processing. The other two operators run the plant. If more automation was utilized, the number of operators could be reduced.

Currently, only LX-14 has been processed cost effectively using this innovative process. A design has been developed for a 500 lb RDX recovery plant. This design incorporates up to four reactors for the separation of RDX, for a maximum process of 2000 lbs per day. This increased capacity vastly improves the process economics, bringing the projected cost of RDX recovery down to \$3.33 per lb. This scale-up should not alter the number of operators. It is estimated that one shift of two operators and one supervisor will be sufficient to process 1200 lbs of RDX per day. At this point, there are no plans to implement the RDX recovery process.

6.1 COST OBSERVATIONS

For the HMX recovery process, the primary performance objectives were met. The recovered HMX met the cost criteria, purity, and throughput rates. Most of the secondary performance criteria were also met, except for the versatility and scale-up issues. While the acid digestion appears to be feasible for most HMX-containing munitions, more studies will need to be performed to accurately understand the process for munitions that differ from the LX-14 composition. Digestion times will vary for different munition types. Whether they will be reduced or lengthened has yet to be determined. If they can be reduced significantly, the plant capacity could be increased, which would be of an economic benefit.

6.2 PERFORMANCE OBSERVATIONS

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6.3 SCALE-UP

Scale-up issues were not extensively studied due to the fact that the demonstration scale plant will be utilized for the commercial-scale operation. The most significant factor affecting scale-up is the increase in net mass and heat transfer associated with increased quantities of explosives. Studies will need to be done to obtain optimal feed throughput for larger explosive quantities.

6.4 OTHER SIGNIFICANT OBSERVATIONS

The implementation of this technology should be straightforward. The contracting firm that developed this technology will also be the operators when commercially implemented.

6.5 LESSONS LEARNED

The demonstration was an important milestone for the successful operation and understanding of the technology. It was a useful tool for properly evaluating the functionality of the processing plant. Several comments and suggestions offered by those attending the demonstration allowed for adjustments and improvements to be made to the process that were not previously identified. These improvements included changes to the manner by which the pH adjustments were made to the neutralization tank; the cooling process of the RDX solution to decrease the time needed to solidify the wax; the addition of monitoring sensors for nitric acid and NO_x in the production area; and the aspiration process for wax removal. The demonstration for the recovery of RDX also allowed us to determine the need and importance for a heated lid and vortex separator for a more efficient separation of wax from the RDX. For the demonstration, it was important to have all necessary documentation available for review by those attending the demonstration. The availability of process documentation allowed questions to be addressed thoroughly, effectively, and promptly.

Operator training and familiarity with the technology was an extremely useful tool to allow for the candid and complete exchange of information between the demonstrating company (TPL, Inc.) and the representatives attending the demonstration. With the active participation of the process operators in the demonstration and in related discussions, it was evident that the process was easily understood and operated by those who have been properly trained.

6.6 END-USER ISSUES

Various kinds of munitions will have to be explored to fully utilize this process. The major processing difference for different munitions is changes in heat and mass transfer. For example, rocket motors have been explored for the recovery of HMX. The problem with rocket motors is that they contain aluminum, which has been known to exothermically react in this process. Caution must be used to prevent thermal runaway. More studies will have to be performed to determine optimal feed rates so that heat transfer is controlled. Other munitions items such as PBX-filled items (PBXN-110, PBXN-3, PBXN-104) have also been considered. Again, initial studies will have to be performed to understand the reaction of acid with different polymers.

The end-users of this technology will be DoD and DOE, who are seeking a new source of HMX at a reduced cost. Currently, HMX is procured from BAE, Royal Ordnance North America (previously the Holston Army Ammunition Plant) or from Pantex. The foreign vendors' cost of virgin HMX is considered to be too high for use in many munitions. The reduced cost of recovered HMX would make it feasible to use HMX in many munitions that currently do not meet Insensitive Munitions (IM) requirements. Naval Surface Warfare Center, Indian Head Division and Los Alamos National Laboratory (LANL) are very interested in recovered HMX. NSWC Indian Head is currently looking into the replacement of Comp B with PBXN-110. The significant decrease in the cost of HMX would make this program more feasible. LANL is depleting the last of its strategic reserve of HMX while initial efforts are underway to formulate PBX-9501, an HMX-containing PBX.

6.7 APPROACH TO REGULATORY COMPLIANCE AND ACCEPTANCE

TPL, Inc. has provided information to the State of New Mexico to ensure compliance with environmental regulations. A revised Notice of Intent was filed with the State Air Quality Bureau and approved prior to the start-up of the demonstration plant. The Notice of Intent accounted for the increase in nitric acid emissions from the plant, as well as any potential increase in NOx emissions. The Air Quality Bureau informed TPL, by letter on 1 October 1997, that no operating permit was required based on the fact that the sum of all emissions was below the permit threshold. A revision to the Notice of Intent was filed in December 1998 and accepted in January 1999. This revision included all the necessary facility additions to allow for the operation of the demonstration plant below the permit levels.

6.8 DISPOSITION OF DEMONSTRATION EQUIPMENT

The demonstration equipment will be utilized as a production-scale facility for the recovery of HMX. Efforts are currently underway to assess the qualification of recovered HMX for reuse in munitions. A 500 lb reactor design plant has been developed for a prototype-scale RDX recovery facility. The RDX recovery plant has been proposed to be constructed at Crane Army Ammunition Activity. The prototype plant would be built to facilitate the direct transfer of washed-out Composition A-3 in to the RDX recovery plant. Before this technology can be implemented, the facility design must be completed and further analyses and demonstrations must be conducted. Regulatory approval and additional funding would also be required. It has not been determined how, by whom, or in what time frame these issues will be addressed. Currently, no further plans have been made to implement this recovery process.

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APPENDIX A

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