

**Recovery and Reuse  
of HMX/RDX  
from Propellants and Explosives**

**TPL, Inc.**

**12 July 1999**

| Report Documentation Page                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                    |                                     |                                                                  | Form Approved<br>OMB No. 0704-0188                  |                                    |
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# **Recovery and Reuse of HMX/RDX from Propellants and Explosives**

**TPL, Inc.**

**12 July 1999**

## **1. Introduction**

### **1.1 Background Information**

Currently DoD has no method of recovering high value energetics, such as HMX/RDX, from Class 1.1 propellants and explosives for reuse applications. DoD has over 53,000 tons of HMX/RDX material in the Resource Recovery and Disposition Account (RRDA) and expect to generate several thousand more tons over the next five years. Without a reclamation and reuse process, the value of these products are lost as they are destroyed by open burning and open detonation.

### **1.2 Official DoD requirement Statement(s)**

Navy 3.I.13.a Reuse/Recycle of Hazardous/Polluting materials  
Navy 2.I.1.h Control Emissions from Ordnance Manufacture and Demilitarization Operations  
Navy 3.I.6.c Energetics Production Pollution Prevention  
Air Force 96-1704 Reclamation/Recycling/Disposal of Munitions  
Navy 2.III.1.t Alternative Ordnance Disposal  
Army 3.3.c Reduce VOCs in Ordnance Manufacture  
Air Force T2700.02 New Technologies for Clean Air Act Compliance  
Navy 3.I.2.a Reduction of TRI Emissions and HW Disposal

### **1.3 Objectives of the Demonstration**

This technology will provide DoD with a means to recover high value energetics. This will also eliminate the need to burn or detonate these items as a means of disposal. The demonstration will be conducted at the TPL, Inc. facility at Ft. Wingate, New Mexico. In order to properly evaluate the agility of the system, TPL will perform a two day demonstration of the HMX

recovery from LX-14 and also a two day demonstration of the RDX recovery from Composition A-3.

#### 1.4 Regulatory Issues

Open burn open detonation is not a viable alternative for these energetic material due to the value of the product and the environmental unacceptability of disposing of these materials in that manner.

#### 1.5 Previous Testing of the Technology

Not applicable

## 2. Technology Description

### 2.1 Description

#### PROCESS DESCRIPTION-HMX RECOVERY FROM LX-14

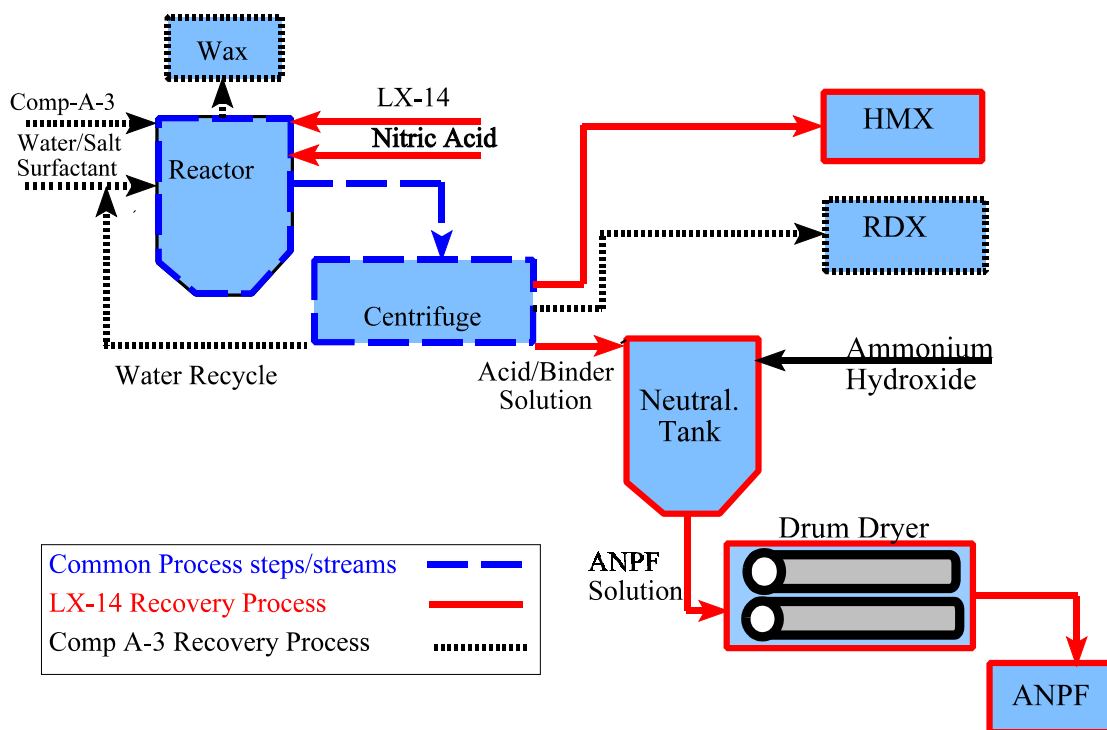


Figure 1. Sub-scale HMX/RDX Recovery Process Flow

### 2.1.1 HMX Recovery from LX-14

Refer to Figure 1 for graphical representation of process steps described below.

For the demonstration of the processing plant, 150 lbs of LX-14 were manually loaded into the stainless steel, 80 gallon reactor, Figure 2. The reactor prior to the introduction of LX-14 was charged with 19 gallons of concentrated nitric acid (1 kg LX-14: 1 liter of nitric acid). The resultant mixture was allowed to passively 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 1 hour.



Figure 2

The slurry was then introduced into a 40" x 18" basket centrifuge,



Figure 3

Figure 3, where the acid/binder slurry was removed from the HMX through a polypropylene filter bag. The HMX was retained in the filter bag while the binder acid slurry was pumped to a neutralization tank, Figure 4. The effluent was neutralized with ammonium hydroxide to a pH ranging from 6-7.



Figure 4

The solution was then introduced to a steam-heated double drum dryer (Figure 5) with the purpose of yielding, through water evaporation, a compound salt identified as Ammonium Nitrate Polymeric Fuel (ANPF).



Figure 5

The recovered HMX retained in the centrifuge filter bag (Figure 6) 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.



Figure 6

The HMX was manually removed from the centrifuge, loaded into plastic packaging bags (Figure 7) and 15 wt-% water was added in order to desensitize the nitroamine.

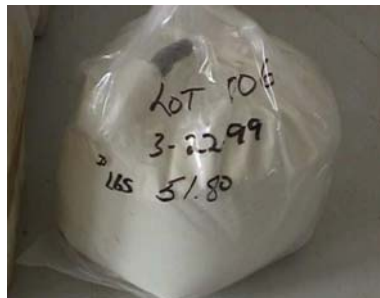


Figure 7

Any acid used in the rinsing process through the reactor and centrifuge was recycled for use in the subsequent batches of LX-14.

### 2.1.2 RDX Recovery from Comp A-3

Refer to Figure 1 for graphical representation of process steps.

For the demonstration of RDX recovery from Comp A-3, the system was configured differently than 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 of water: salt: surfactant by weight.

After the salt solution density was loaded into the reactor, the Comp A-3 was charged into the reactor as well. The reactor capacity allowed 75 lbs of Comp A-3 to be loaded at one time. The water solution to Comp A-3 was processed at a 5:1 by weight ratio. The Comp A-3 water solution was stirred and the reactor temperature was raised to 80°C.

After the target temperature was reached the process was allowed to remain at temperature for 30 minutes. At this time the melted wax that floated to the top of the solution 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 (Figure 8).

Quench water was added to the reactor to further cool the solution in order to extract additional wax floating on top of the reactor. After the majority of the wax was removed by mechanical means, the smaller wax particles were removed via aspiration methods using a vacuum pump.



**Figure 8**



**Figure 9**

When all of the wax was removed from the reactor, the remaining solution was introduced into the basket centrifuge where the RDX was retained and the water/salt/surfactant solution was pumped to a recycle tank (Figure 9) for use in subsequent process batches. The solution density of the recycled water was checked for density reduction caused by attrition of salt and replenished as necessary for the next processing batch.

The RDX is rinsed with fresh water to remove any water solution residue present from the processing. Again, the RDX is removed from the centrifuge manually and packaged.

A common P&ID for both recovery processes is depicted in Figure 10.

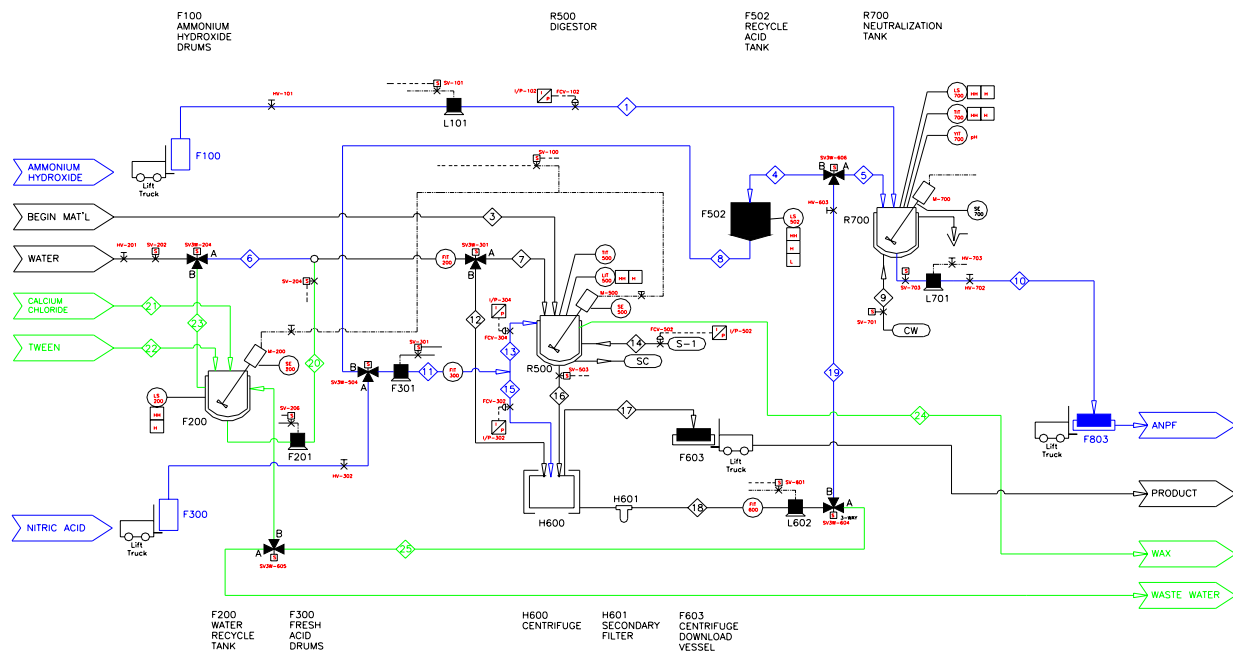


Figure 10. Process Flow Diagram for Both HMX and RDX recovery Processes

## 2.2 Strengths, Advantages, and Weaknesses

Currently there are no capabilities within DoD to recover these high value energetics. The technology can be adapted to a wide variety of energetic materials. The only limitations are the time and funding required to develop the process for the individual energetic material systems and the subsequent modifications to the system.

## 2.3 Factors Influencing Cost and Performance

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. Several iterations of the parameters were realized in order to achieve a safe, productive and efficient system that resulted in a high quality product.

Targeted process parameters for each process are listed below. These parameters were refined and based on the development runs that were made, as well as the demonstration runs performed.

### LX-14

|                           |                 |
|---------------------------|-----------------|
| Batch weight:             | 150 +/-5 lbs    |
| Digestion acid:           | 18 +/- 0.5 gal  |
| Digestion time (passive): | 16 +8, -4 hrs   |
| Digestion temperature:    | 158 +/- 5 deg F |

|                                    |                    |
|------------------------------------|--------------------|
| Digestion time (at elevated temp): | 1 hr +/- 5 minutes |
| 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 deg F    |
| Drum dryer throughput rate:        | 70 +/- 10 lb/hr    |

### Comp A-3

|                                      |                      |
|--------------------------------------|----------------------|
| Batch weight:                        | 75 +/- 5 lbs         |
| Process water:                       | 45 +/- 1 gal         |
| Salt (CaCl <sub>2</sub> ):           | 150 +/- 5 lbs        |
| Surfactant (Tween 20):               | 18.8 lbs +/- 0.5 lbs |
| Process temperature:                 | 176 +/- 2 deg 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 minutes    |

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 insure the reliable performance of the equipment on a daily basis.

Replacement parts must be kept on hand for high use items. Minimizing down-time due to equipment wear or malfunction will support the process and maintain the anticipated throughput rates over extended time. Parts required as spare parts inventory include back-up instrumentation, pumps, diverter valves, solenoids, centrifuge bags, and process-wetted gaskets and seals.



### 3. Site/Facility Description

#### 3.1 Background

The site selected is building 542 (Figure 11) at Ft. Wingate Army Depot, Ft. Wingate, New Mexico. The criteria that led to the selection of this site, were its availability as an existing structure, the available floor space, adjacent locations available for remote operations, and existing siting for energetic material handling.

The site selected had an existing infrastructure of utilities such as steam, air, water, and power necessary for plant operation. The building construction of reinforced concrete walls and its location from the main access road provided an excellent means 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 to recover HMX from surplus energetic materials.



Figure 11

#### 3.2 Site/Facility 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 building distance from the road allows for the handling of up to 500 lbs of class 1.1 materials based on interline distances. The existence of power poles between building 542 and



Figure 14

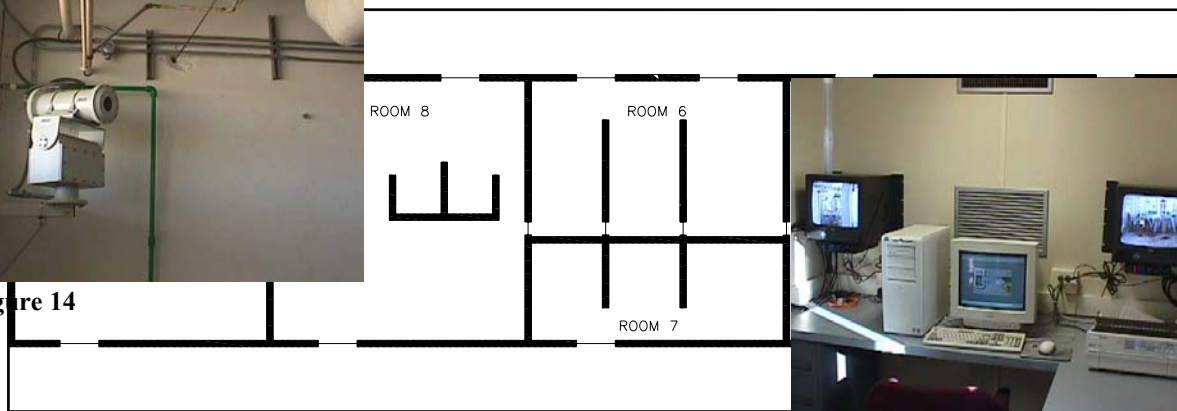


Figure 15

BUILDING 542 – FLOOR PLAN



Figure 12. Building 542 bay lay-out

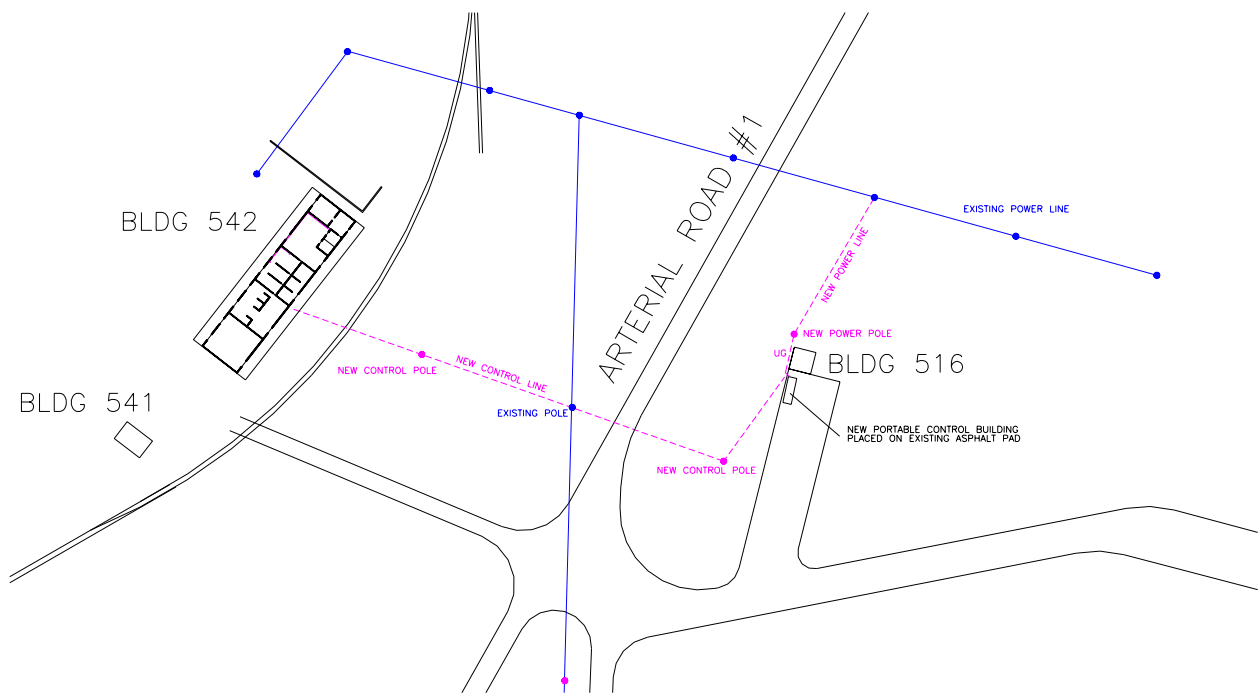


Figure 13. Control Room and Building 542 Pole Placement

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. See (Figure 12) for an overview of building 542 and the associated processing room 8. (Figure 13) includes the location of the remote control room and pole placement locations required for the operation of the processes in building 542. Reinforced concrete separating walls also allowed for isolating distinctly different process operations such as the acid and base process in the recovery of HMX from LX-14, as well as creating natural divisions for in process raw material, final product and process materials storage. Remote cameras (Figure 14) within room 8 of building 542 allows the operators to monitor all activities from a safe distance in the control room (Figure 15).

## 4. Demonstration Approach

### 4.1 Performance Objectives

The process will be evaluated using the following criteria:

- a. Cost
  1. Labor
  2. Materials
- b. Purity of recovered products
- c. Safety
- d. Disposition of by-products

### 4.2 Physical Setup and Operation

The increase in quantity of explosives being processed in the demonstration plant necessitated the installation of a remote control room (Figure 16). A portable unit was set up adjacent to building 516, which is outside the quantity-distance arc from the operations in building 542. Power and control lines were installed to support this remote operating facility. 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:



Figure 16

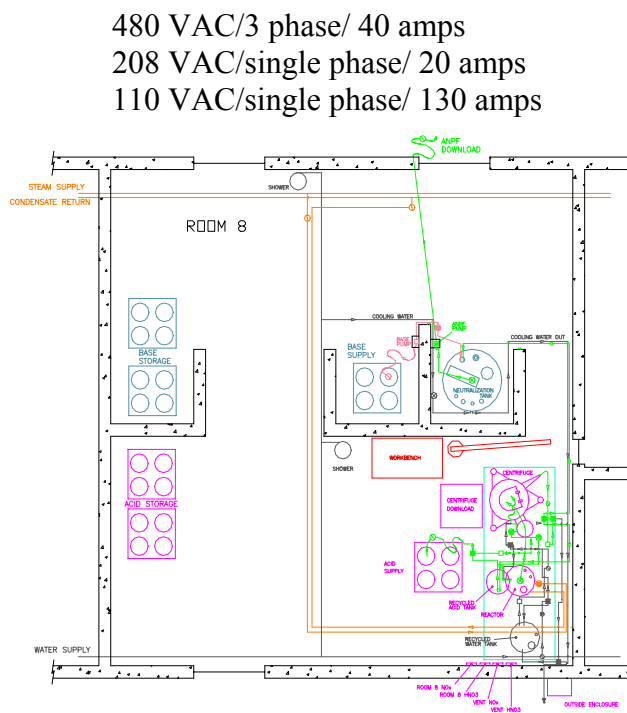


Figure 17. Process Lay-out for Building 542

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 clean-up of the building and equipment. Equipment lay-out is included in (Figure 18).

Operation of the plant for the demonstration was held to the schedule listed below. Times listed were typical for the plant operation with little deviation experienced in the schedule.

**HMX/RDX Recovery Sub-scale Plant Demonstration Schedule**

| Day                             | Time    | Event                            |
|---------------------------------|---------|----------------------------------|
| Sunday, 21 MAR                  | 1:00pm  | Weigh-up/Load LX-14 into Reactor |
| Monday, 22 MAR                  | 7:00am  | Check-out/Heat up                |
|                                 | 11:00am | Download HMX                     |
|                                 | 11:30am | Download ANPF                    |
|                                 | 12:00pm | Bay Clean-up                     |
|                                 | 3:00pm  | Weigh-up/Load LX-14 into Reactor |
|                                 | 4:00pm  | Shutdown                         |
| Tuesday, 23 MAR                 | 7:00am  | Check-out/Heat up                |
|                                 | 11:00am | Download HMX                     |
|                                 | 11:30am | Download ANPF                    |
|                                 | 12:00pm | Bay Clean-up                     |
|                                 | 1:00pm  | Transition process for Comp A-3  |
|                                 | 5:00pm  | Shutdown                         |
| Wednesday, Thurs.*<br>24&25 MAR | 7:00am  | Check-out/Weigh-up/Load Comp A-3 |
|                                 | 8:00am  | Heat-up                          |
|                                 | 9:30am  | Quench/Skim                      |
|                                 | 11:00am | Centrifuge                       |
|                                 | 12:00pm | Download RDX                     |
|                                 | 1:00pm  | Check-out/Weigh-up/Load Comp A-3 |
|                                 | 2:00pm  | Heat-up                          |
|                                 | 3:30pm  | Quench/Skim                      |
|                                 | 5:00pm  | Centrifuge                       |
|                                 | 6:00pm  | Download RDX                     |
|                                 | 7:00pm  | 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.

The demonstration plant was operated by 3 operators 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.

Operating Procedures are attached in Appendix C for the HMX recovery process and in Appendix D for the RDX recovery process.

Materials processed in the demonstration plant included LX-14 and Composition A-3. Batch sizes for the HMX recovery process were developed at 150 lbs. RDX recovery batch sizes were limited to 75 lbs based on reactor vessel size constraints with 2 batches processed per day. Both materials yielded 150 lbs per day. The LX-14 processing was performed first and on consecutive days. After a short turn-around cycle for the processing of Composition A-3, an additional 2 consecutive days of processing were completed.

#### 4.3 Sampling Procedures

The sampling plan involved a direct measurement of the product quality through laboratory methods defined in MIL-H-45444B for HMX and MIL-R-398C for RDX. Samples were collected from the centrifuge cake (Figure 19) for each batch of material produced. Analytical data was collected for the following tests:

Purity  
Melting point  
Physical appearance  
Batch yield (calculated)



Figure 19

The sampling procedures for the demonstration were performed as follows:

1. Obtain 1 each 20 g sample from the centrifuge cake. Place samples in a glass vial, (Figure 20) 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.



Figure 20

#### 4.4 Analytical Procedures

Analytical methods used are as follows:

Purity - High performance liquid chromatography (HPLC) was used to measure the HMX content and RDX content in the samples provide. Sample size, preparation, and measurement techniques were performed per MIL-H-45444B for the HMX sample and MIL-R-398C for the RDX samples.

Melting Point - Samples from each batch were evaluated in a capillary melting point indicator per the mil specs listed above.

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

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

### 5. Performance Assessment

#### 5.1 Performance Data

Material properties were measured for both the HMX and RDX from the demonstration batches. The data from those batches are included below.

##### HMX

| <u>Lot No.</u> | <u>Melting Pt.</u><br>°C | <u>Purity</u><br>% | <u>Yield</u><br>% | <u>Physical Appearance</u> |
|----------------|--------------------------|--------------------|-------------------|----------------------------|
| 5              | 278                      | 98.13              | 99.5              | White                      |
| 6              | 278                      | 99.60              | 98.5              | White                      |

##### RDX

|    |     |             |      |            |
|----|-----|-------------|------|------------|
| 8  | 199 | 98.01* 97.7 |      | Gray/white |
| 9  | 198 | 98.97       | 95.5 | Gray/white |
| 10 | 197 | 97.74       | 95.0 | Gray/white |

\* values represent total of RDX and HMX present in product

Data collected from each batch included one sample based on cost and schedule constraints. Values listed above are not reduced, averaged, manipulated from the measured data for each batch of material recovered.

Sample mass balances for the process are contained in Appendix E for the HMX recovery process and Appendix F for the RDX recovery process. These balances are based on typical data gathered from the demonstration plant and contain calculated values for those streams which were not measured during the process

## **5.2 Data Assessment**

The data collected provides a clear indication of the product quality and performance objectives based on comparison to mil spec values and past experience with analyzing materials from both the pilot plant and demonstration plant. Past experience has indicated that materials which meet purity levels and melting point were consistently and repeatably within specifications for acidity levels and insolubles. Based on this acquired experience, the samples measured from the demonstration are considered acceptable for evaluation by potential customers for alternate applications.

## **5.3 Technology Comparison**

No current technologies exist for the repeated extraction of HMX or RDX from the feedstocks provided. This demonstrated technology is unique in producing HMX or RDX without significantly changing the particle size or shape in the process.

The performance of the process is superior to other competing technologies based on its simplicity, effectiveness, and 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 effect a proper extraction of product from the feedstock. Due to the off-the-shelf processing equipment employed in the process, replacement parts are not difficult to obtain and repair costs are not exceedingly high. The process simplicity allows for operator interaction and understanding of each process step. Any problems that occur are less complex to identify and repair than 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. Due to a PLC controlled process, the reactor temperature is maintained within 5° 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 distinct feedstocks. Improvements to the wax removal operations are expected and planned for future dedicated Composition A-3 plant designs.



### 5.3.1 By-product Disposition

The principal by-product of the HMX recovery process is spent nitric acid. The nitric acid contains a degraded Estane™ polymer. The exact nature of the polymer is not known, but the fragments of the polymer are basically C-H-N-O combinations. This organic material is a fuel, albeit relatively simple. When the spent nitric acid/degraded polymer is neutralized with



**Figure 21**

ammonium hydroxide, an aqueous fuel/oxidizer mixture results. This aqueous solution is transferred by tanks (Figure 21) from Building 542 to the drum dryers for processing. After the water is removed, via an evaporation process, the resulting material is analogous to ammonium nitrate/fuel oil (ANFO) which is a commonly used blasting agent. Consequently, TPL has named the HMX recovery by-product ANPF; an abbreviation for ammonium nitrate/polymeric fuel.

The ANPF is similar in detonation properties to ANFO. ANPF has been found to be detonable when boosted with 1 ½ lb pentolite boosters. Very finely divided ANPF may

be detonable. Approximately 724 g of material was boosted with two blasting caps and a dent in aluminum plate was observed; a similar result was observed with a single cap. No dent was observed if the ANPF was coarse.

TPL has been developing explosives for explosively cladding dissimilar metals. ANPF has been formulated with a granular plastic bonded explosive to make a unique metal bonding explosive. This explosive, BondEx A™ and its derivatives, detonates as slow as 1.6 mm/μsec in annular thickness as thin as 13 mm. This is precisely the need for explosively cladding refractory metals to the interior of tubes. 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. (Figure 22) shows the clad of tantalum to the 120 mm tube and an inset photograph of the bonded region. A subsequent Phase II program has been awarded to TPL and medium caliber liners are currently being investigated. TPL will be teaming with General Dynamics Armament Systems and/or others to fabricate gun tubes for service life testing.

BondEx A™ and its derivatives have applications beyond explosive metal cladding. A. Cain of TPL demonstrated that the explosive



**Figure 22.** Tantalum alloy bonded to carbon steel with the inset showing the interfacial region.

can be made to detonate at rates as low as 1.0 mm/ $\mu$ sec in diameters as small as 19 mm. This explosive can be used for dimensional stone mining where these characteristics are necessary.

## **6. Cost Assessment**

### **6.1 Cost Performance**

Expected operating costs were analyzed for the fully operational HMX recovery system. Based on the data known at the time of the analysis regarding raw material costs, labor costs, equipment operating costs, maintenance costs, quality assurance costs, program management costs, and shipping costs the average price per pound of recovered HMX is \$6-8.

RDX costs when steady-state conditions are realized are expected to settle near \$3-5/lb based on the factors listed above and the anticipated ease of dealing with a water-based process.

### **6.2 Cost Comparisons to Conventional and Other Technologies**

Costs associated with the conventional manufacture of HMX are known to range from \$18-25 per lb. Costs associated with the conventional manufacture of RDX are approximately \$3-4 per lb.

## **7. Regulatory Issues**

### **7.1 Approach to Regulatory Compliance and Acceptance**

TPL, Inc. has provided information to the State of New Mexico to insure 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 NO<sub>x</sub> 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.

## **8. Technology Implementation**

### **8.1 DoD Need**

The DoD has significant quantities of explosives and propellants containing HMX and RDX. The large rocket motors in the DoD inventory provide a tremendous disposal problem which could be alleviated with this type of technology. The recovery of these significant quantities of

HMX would provide a means of off-setting some of the disposal costs. The Comp A-3 loaded projectiles within the inventory number in the hundreds of thousands and the washout system installed at Crane Army Ammunition Activity has just recently begun operating and generating washed out explosives for reclamation.

## **8.2 Transition**

The plan for transition from the demonstration will involve operating the plant in a commercial manufacturing mode for the demilitarization of LX-14 and for the recovery of HMX. The plant will be staffed by two full-time operators and operated on a 4-5 day/week schedule to obtain 30,000 lbs/year of HMX. It is estimated to take three years to deplete the current 100,000 lbs in inventory currently at Ft. Wingate. The RDX recovery plant will transition in a different manner. The need for a higher capacity plant dictates a design incorporating lessons learned from the demonstration. The larger scale prototype plant will be located at a government installation to facilitate the direct transfer of washed-out Composition A-3 into the RDX recovery plant. Additional demonstrations are not required for the HMX recovery plant. The RDX recovery prototype plant will involve additional developmental work to verify alternate wax removal techniques. Additional regulatory approval is not required for the operation of the demonstration plant.

Additional design work is required for the prototype processing plant that will be capable of producing 500 lb/day. Alternate wax removal techniques will be identified and addressed to allow for the efficient removal of wax from the high capacity recovery plant.

TPL is currently moving the HMX product into the commercial realm through industry contact, product development and analysis, and production and sampling of the HMX to potential customers for performance evaluation.

The proper implementation pathway for this technology rests in the ultimate performance of the product, acceptance of the product by the customer, and a competitive price for the product. The current implementation plan calls for a market survey for the demand for the product. Upon completion of this survey the appropriate customers will be identified, required quantities determined, and qualification samples delivered to various customers to allow for the review of the product performance and quality. Upon completion of the analysis and acceptance by the customer, orders will be accepted and fulfilled for the product of this technology.

The prime contractor TPL, Inc., is the industrial component involved during the demonstration and the industry partner for implementation of the production. Additional industry involvement is represented by three entities, which are the three primary potential customers for the product:

Accurate Arms Co. for the use in well perforating charges, detonation cords, and other associated applications

Ensign Bickford Co. for the use in shock tubes, detonation cords in the mining and oil industries

Schlumberger for use in well perforating charges and other associated applications

Contacts have been established at all of these companies with sampling plans confirmed with the end user to facilitate the product's acceptance into production.

In addition to the recovery and commercial sale of HMX, plans are being prepared to develop and design a prototype recovery plant for the processing of washed out Composition A-3. The following represents a plan for the planned future work and a description of the issues that will be addressed.

Many processing methods and equipment used in the sub-scale plant were more suited for recovery of explosive constituents from LX-14 than from Comp A-3. Equipment modifications have been implemented in the attempt to improve the efficiency of binder removal from Comp A-3. The design will be modular to maintain the flexibility to process various amounts of Comp A-3 and it will be remotely operated to allow for personnel safety. Since this report covers only preliminary steps, many processing parameters are still vague. Throughout this conceptual process narrative, these parameters will be expressed and two or more alternative processes will be offered.

As a prelude to the process narrative, a brief description of the equipment envisioned is appropriate. As illustrated in Figure 23 (PFD), each module of the preliminary design calls for a

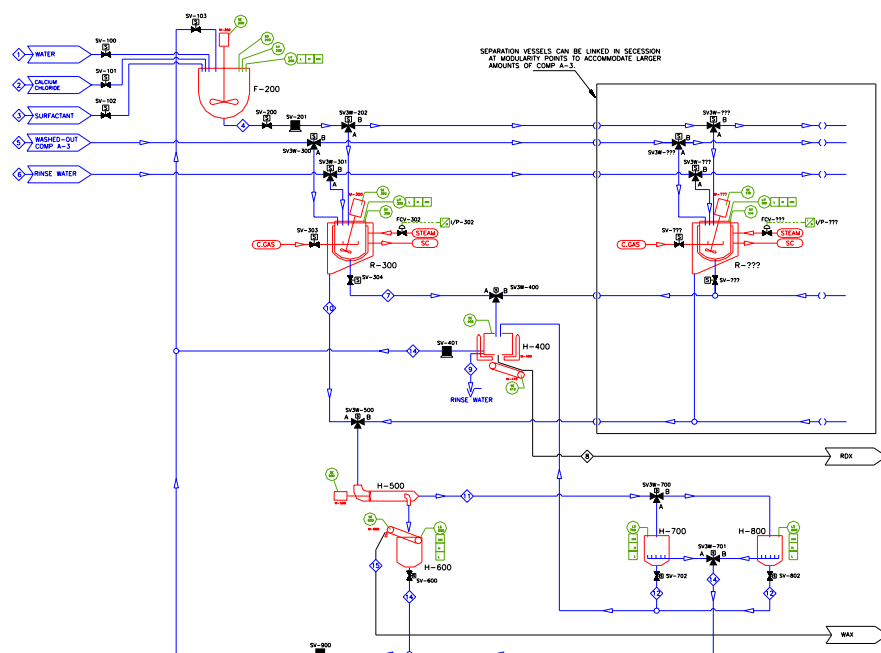


Figure 23. Prototype Plant Process Flow Diagram

salt/surfactant solution source vessels, reactor, centrifuge, centrifugal separator, conveyor fitted vessel, and two settling vessels. The solution vessel will be two, possibly three, stainless steel vessels fitted with a stirrer. The reactor will have an open top design to allow for binder overflow and faster cooling of the solution once the binder is separated from the RDX. It will be partially jacketed and also fitted with a stirrer. To contain the overflow, the reactor will be placed within another conical tank. Based on information known at this stage, the centrifuge will most likely be of the continuous flow design: specifically a pusher or peeler type. In order

to be able to recycle the solution within the system, all of the binder must be removed from the solution after each RDX/binder separation. Currently a centrifugal separator, specifically a voraxial separator, is being investigated to accomplish this important task. The advantage of the voraxial separator is discussed further in the body of the narrative. Again, in order to recycle the solution the residual RDX fines remaining in the overflow must also be removed. This will be accomplished using two settling tanks. Each module is based on a Comp A-3 throughput of 500 lbs. per day. Preliminary sizes are as follows:

| Equipment description  | Approximate size/volume |
|------------------------|-------------------------|
| Solution source vessel | 2 @ 1,500-gallon        |
| Reactor                | 500-gallon              |
| Centrifuge             | TBD                     |
| Centrifugal separator  | 2" dia., 25-100 G.P.M.  |
| Conveyor fitted vessel | 250-gallon              |
| Setting tanks          | 2 @ 500-gallon          |

At this time, the state of the feedstock is not known, therefore, two processes have been defined to deliver the washed out Comp A-3 to the separation vessel. The first and most probable delivery system will involve realtime introduction of the Comp A-3 slurry directly from the washout process into the reaction vessel. An alternative delivery method could be to use a conveyor system if the feedstock is too viscous to pump. Once approximately 500 lbs. of Comp A-3 has been added to the reaction vessel, saltwater/surfactant solution will increase the total volume to about 500 gallons. The volume will be agitated and heated to a temperature slightly above the melting point of the binder and held for a predetermined period of time. Both stirring and heating will then cease and the volume will be allowed to cool for a few hours. As conditions become quiescent and the temperature falls below the melting point of the binder, some separation will occur. In order to alleviate the inefficiencies encountered with the sub-scale aspiration technique, the separated binder will most probably be removed using a two-step aeration/overflow method. The first step will be to introduce a compressed gas between the separated layers. Finely divided bubbles will cleanly and efficiently float the small binder particles to the top of the vessel. The final step will be to slowly overflow the vessel using the salt/surfactant solution leaving behind only RDX.

RDX can now be sent from the reactor into a centrifuge capable of handling continuous flow. As discussed previously, both pusher and peeler type centrifuges have been considered so far, but further discussion of safety issues and investigation of other options is warranted.

The binder effluent from the reactor overflow will need to be separated. Preliminary design includes a centrifugal separator to accomplish this. What makes the centrifugal separator attractive is that it takes advantage of differing specific gravities to separate slurries. The heavier material, salt/surfactant solution in this case, will tend toward the outer walls of the separator and the lighter material, binder in this case will migrate toward the center of the vortex.

Design of the centrifugal separator provides means to recover the concentrated lighter material while allowing the heavier material to pass. Further considerations of issues such as impeller clearances and solids loadings must be addressed before this type of equipment can be specified. In the event a centrifugal separator is deemed inappropriate, other binder removal options exist, such as employing a settling tank or filtering equipment.

The salt/surfactant solution from the voraxial separator will be piped into settling tanks to allow removal of very fine RDX. After a predetermined residence time, the salt/surfactant solution will be returned to the system for use in the next RDX/binder separation. The binder stream from the voraxial separator will be discharged onto a perforated conveyor, which will act as a moving filter to remove any excess salt/surfactant solution. The conveyor will deposit the wax into a hopper.

## **9. Lessons Learned**

The demonstration was an important milestone for the successful operation and understanding of the technology. It was a useful tool to properly evaluate 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. It was important for the demonstration to have all necessary documentation available for review by those attending the demonstration. This practice 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) and the representatives attending the demonstration. With the active participation of the process operators in the demonstration and related discussions, it was evident that the process was easily understood and operated by those who have been properly trained.

## **10. References**

Not Applicable

## **Appendix A**

### **Points of Contact**

#### **Contractor (TPL, Inc.) Project Manager**

Tom Schilling  
TPL, Inc.  
3921 Academy Parkway N, NE  
Albuquerque, NM 87109-4416  
505-342-4436 Fax 505-343-1797

#### **Principal Investigator**

Mike Miks  
TPL, Inc.  
3921 Academy Parkway N, NE  
Albuquerque, NM 87109-4416  
mmiks@tplinc.com  
505-342-4440 Fax 505-343-1797

#### **Government Project Manager**

Dan Burch  
Naval Surface Warfare Center, Crane Division  
Code 4073  
300 Highway 361  
Crane, Indiana 47522  
burch\_dan@crane.navy.mil  
812-854-5651 Fax 812-854-2699

#### **Contracting Officer's Representative**

Keith Sims  
Naval Surface Warfare Center, Crane Division  
300 Highway 361  
Crane, Indiana 47522  
sims\_keith@crane.navy.mil  
812-854-5651 Fax 812-854-2699

#### **Government Sponsor**

Jim Wheeler  
Defense Ammunition Center  
1 C Tree Road  
McAlester, Oklahoma 74501  
918-420-8901 Fax 918-420-8717

## **Appendix B**

### **Data Archiving an Demonstration Plan**

Data collected from the demonstration is located at the processing site with the personnel operating the process. Batch files are kept for every lot produced which includes process observations, operating parameters, batch weights, and any process anomalies.

A copy of the approved Demonstration Plan is on file with the Contracting Officer's Representative and with the ESTCP Program Office.