

**BAE SYSTEMS**

FURTHER DEVELOPMENT AND  
OPTIMIZATION OF IM INGREDIENTS AT  
HOLSTON ARMY AMMUNITION PLANT

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## 1. ABSTRACT

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Historically, TNT has been the primary, melt cast binder for melt pour explosive formulations, and all of the melt cast LAP plants are configured for its use. However, due to sensitivity, stability and exudation issues, replacements for TNT and Comp B explosives have long been of interest. Recently, 2,4-dinitroanisole (DNAN) has found widespread use as a TNT replacement and NTO as a RDX substitute.

A number of non-traditional energetic ingredients can be incorporated into DNAN formulations that contribute significantly to the energy output and Insensitive Munitions characteristics of the resulting explosive. These ingredients include nitrotriazolone (NTO), nitroguanidine (NQ), dinitroglucoluril (DNGU), triaminotrinitrobenzene (TATB) and others, to yield products that match the performance of TNT or Comp B. The formulated melt-cast materials are far less sensitive than TNT, providing ordnance that is much safer and exhibits an enhanced level of survivability when subjected to unplanned stimuli.

Processes for the synthesis and optimization of melt-pour ingredients have been developed on the large production utilizing the explosive manufacturing infrastructure at Holston Army Ammunition Plant. These ingredients are used to manufacture and supply cost competitive, IM explosives to meet all projected requirements of artillery, mortar, tank ammo, and submunition systems in the U.S. arsenal which currently use sensitive, legacy explosives such as TNT or Comp B.

This paper describes the synthesis, scale-up, and large-scale manufacture of the stated IM ingredients as well as data from optimization and material studies. Included are discussions of an improved process for the manufacture of high bulk density NQ (HBNQ), improvements in the crystal quality of NTO produced at HSAAP, and a material study focusing on interesting thermal characteristics of DNAN.

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## 2. BACKGROUND AND OBJECTIVES

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Energetic materials utilized in explosive and propellant applications are an integral component and have significant impact on the performance and safety of weapon systems. Current Department of Defense (DOD) initiatives, focused on weapon and munitions systems that are safer for the U. S. Warfighter while simultaneously maintaining or improving performance, offer challenges for the development and scale-up of high performance, insensitive energetic materials. For an energetic material or other critical ingredient to be used as a credible component in a weapon system, the material should ideally meet the following criteria:

- Available from a robust synthesis process that is safe and not harmful to the environment.
- Available from a stable manufacturing operation that has sufficient capacity to meet baseline and surge requirements for the material.
- Available from a CONUS (continental U. S.) source.
- Available at an affordable cost.

- Improved IM performance over legacy explosives while maintaining similar explosive performance

Holston Army Ammunition Plant (HSAAP) in Kingsport, Tennessee has historically been the major supplier of explosive materials (especially RDX and HMX based products) to the U. S. Department of Defense (DOD). BAE Systems Ordnance System Inc, the Operating Contractor at HSAAP, specializes in the development, scale-up, and manufacture of explosive ingredients and formulations. As the U. S. supplier of RDX and HMX based explosives, the HSAAP facility has a very substantial, active manufacturing capability for nitration chemistry, acid handling and recovery, and other chemical processing capabilities that are unmatched in the Defense Industry.

In addition to the production capabilities at HSAAP, BAE Systems has established a world-class research facility focused on organic synthesis research and development of credible manufacturing processes for energetic materials and other products considered critical to the performance and safety of DOD weapon systems. BAE Systems has a “production mentality” in its synthesis research and specializes in the development of synthesis methodologies that are readily scalable to true manufacturing operations. By combining the stated research capability with the comprehensive manufacturing infrastructure comprising HSAAP, BAE Systems can credibly supply energetic and other critical materials in quantities ranging from grams to millions of pounds. This includes melt-pour ingredients, where the processes for the synthesis have been developed and optimized on the lab-scale then scaled-up to utilize the current explosive manufacturing infrastructure at HSAAP.

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### **3. HSAAP PRODUCT PORTFOLIO**

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Historically, explosive research and development activities at HSAAP have been performed in conjunction with DOD government laboratories and commercial defense contractors. Established manufacturing processes currently exist for the majority of the explosive materials currently fielded in legacy DOD weapon systems. Examples of such products include but are not limited to RDX, HMX, TNT, and ammonium perchlorate (AP). In isolation, these energetic materials do not exhibit the characteristics required to meet the IM initiatives adopted for DOD munitions. Robust synthesis and manufacturing processes are needed for other energetic materials which can make a positive contribution toward achievement of targeted performance and safety parameters in weapon systems. Recent research conducted by BAE Systems on explosive formulations has focused on such areas as low cost synthesis and manufacture of insensitive explosive ingredients, recrystallization of explosive ingredients to offer improved crystal size and shape, and Insensitive Munitions (IM) melt-cast formulation development. Table 1 list some of the traditional explosive ingredients and formulations made at HSAAP as well as some of the new IM ingredients.

**Table 1**

<b>TRADITIONAL EXPLOSIVE INGREDIENTS AND FORMULATIONS</b>
• RDX (MIL-DTL-398D, ALL CLASSES) AND FEM GRADES
• HMX (MIL-DTL-45444D, ALL CLASSES) AND FEM GRADES
• PRESSABLE EXPLOSIVES:
• PBXN-5, N-7, N-10, N-11; LX-14, PAX-2A, PBXW-14, ETC.
• CAST-CURED PRECURSOR EXPLOSIVES:
• CXM-3, -7, -9; CXM-AF-5, -7, ETC.
• TRADITIONAL MELT-CAST EXPLOSIVES:
• COMPOSITION B, OCTOL, CYCLOTOL, ETC.
• PLASTIC EXPLOSIVES (COMPOSITION C-4)
• DMDNB (TAGGANT FOR COMPOSITION C-4)
• SUPERFINE PETN
<b>IM INGREDIENTS AND FORMULATIONS</b>
• INSENSITIVE MELT-CAST EXPLOSIVES:
• IMX-101, IMX-104, PAX-48, OSX-12
• DNAN (TNT REPLACEMENT)
• NTO (COARSE AND FINE GRADES)
• TATB
• HIGH BULK DENSITY NQ

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#### **4. IM MELT-POUR FORMULATIONS DEVELOPMENT**

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Traditional melt-cast explosive fills, such as TNT and Comp. B, have good explosive performance and a low cost associated with high-volume manufacture. However TNT-based traditional melt-cast fills have poor IM performance. When targeting new melt-cast fills, the new material needs to have improved IM performance while still having similar or better explosive performance and a low cost to manufacture. In addition, the material should use existing LAP facilities to reduce to product transition cost.

Table 2 lists some of the new IM melt-pour explosive formulations manufactured at HSAAP. All of these formulations seek to maximize IM and explosive performance, while minimizing cost and the need to replace existing formulation and LAP equipment.

**TABLE 2**

<b>IMX-101</b>	DNAN, NTO, and NQ formulation. Selected as common TNT replacement. Applications include 105mm, 120mm, & 155 mm munitions. Qualified by the U.S. ARMY as main fill explosive in the 155mm M795 Artillery Projectile.
<b>IMX-104</b>	Contains DNAN, NTO, and RDX in various grades. Selected by the U.S. ARMY as the common Comp B replacement in IM Mortar systems (60mm, 81mm, & 120mm) and various submunitions.
<b>PAX-48</b>	Contains DNAN, NTO, and HMX in various grades and provides excellent IM and energetic performance properties. Being evaluated in 60mm Mortar (Europe) and 120mm HE-T Tank Ammunitions (FMS).
<b>OSX-12</b>	An aluminized version of IMX-104 which offers excellent IM properties combined with high blast energetic output.
<b>PAX-21</b>	DNAN based melt-cast explosive which is currently qualified and fielded in the U.S. ARMY 60mm Mortar system.
<b>PAX-41</b>	DNAN based melt-cast explosive which is currently qualified and fielded in the U.S. ARMY SPIDER Area Denial System.

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## **5. NON-TRADITIONAL INGREDIENTS DEVELOPMENT**

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### **5.1 Dinitroanisole (DNAN)**

Dinitroanisole (DNAN) is a key IM melt-phase ingredient that is currently featured in all IM melt-pour formulations developed by the U.S. Army as a TNT replacement. Historically, DNAN has been sole-sourced from China. Additionally, quality issues (product purity) have existed in the past with the Chinese sourced DNAN. BAE SYSTEMS developed a robust, cost-competitive synthesis process for DNAN which met all purity and specification requirements. This process was facilitated and scaled to production operations at HSAAP in 2004 using a batch nitration process. DNAN is now a standard production item at HSAAP with over 200,000 lb produced to date (3,300 lb batch sizes). The synthesis and initial scale-up development of DNAN was reported at IMEMTS 2004.

Since DNAN is processed essentially the same as TNT, it allows for the material to be processed essentially the same as for TNT. In addition, it can be demilitarized the same as for TNT (using the same recover / re-use hardware). Currently formulations are tailored to have TNT / Comp. B performance, while having decreased sensitivity. DNAN is currently fully qualified and fielded in the PAX-21 explosive, which is used in the U. S. Army 60mm Mortar. It is also used in the U.S. Army Spider system. The material is currently undergoing qualification in several other artillery, mortar, and submunition systems including the M795 155mm Artillery munition (fully qualified in 2010).

**FIGURE 1 – DNAN**



## 5.2 Nitrotriazole (NTO)

3-Nitro-1,2,4-triazol-5-one (NTO) is an established explosive that has energetic performance similar to RDX but exhibits improved sensitivity properties. No CONUS source existed for NTO, which certainly limited its applications in DOD munitions. BAE SYSTEMS has developed a robust manufacture process for NTO that is compatible with existing HSAAP manufacturing infrastructure. A product scale-up and facilitization program for NTO was completed at HSAAP in 2004. Substantial quantities of NTO have been generated in the production operations. Over 100,000 lb have been produced to date in full production scale (3,500 lb batch sizes). The HSAAP NTO product is currently undergoing qualification is several DOD artillery, mortar, and submunition systems including the M795 155mm Artillery munition. The formulation of NTO with DNAN produces an explosive melt-pour with Comp. B performance, but a dramatic decrease in sensitivity. The physical properties of NTO is listed below in Table 3.

**FIGURE 2 – NTO**



**Table 3 – Physical Properties of NTO**

Purity: >99%	Vacuum Thermal Stability:(100°C, 48h) 0.2 ml/g
Holston impact: 52cm, (RDX:30cm)	Acidity (pKa): 3.76, 2.35 in a 0.1 M solution
DSC (peak exotherm): >270°C	Energy of Formation: -164.69 kcal/kg=-689.10 kJ/kg
DSC (exotherm onset): >262°C	Oxygen balance: -24.6%
Density: 1.91 g/cm <sup>3</sup>	Impact sensitivity: ≥120 kp m= ≥1200 N m
Melting Point: 268-271°C	Friction sensitivity: at 36 kp= 353 N pistil load no reaction
Detonation Velocity: Unconfined 7860 m/s at ρ= 1.80 g/cm <sup>3</sup> Confined 7940 m/s at ρ= 1.77 g/cm <sup>3</sup>	Solubility: soluble in water and polar solvents

### 5.3 FEM NTO and RDX

HSAAP is the center of excellence in energetic milling. The facility currently houses a 24-inch, 15-inch, and 4-inch Fluid Energy Mill (FEM) systems. Products can be designed or processed at the gram to pound scale by employing the pilot 4-inch FEM system. The processing parameters can be immediately transitioned to the production 24 inch or 15 inch milling systems where up to millions of pounds of energetic materials can be processed to meet industry requirements. Process development using the FEM systems begins with feed rate, venturi airflow and grind airflow parameter evaluation all conducted on the laboratory FEM equipment. Transition to the production facility for the design process is directly scalable. RDX, HMX, and NTO are traditionally milled via FEM for new IM requirements. These processes reduce the sensitivity of the energetic product and maintain processability in formulation design.

**Figure 3 – Fluid Energy Mill**



Fluid-energy milled (FEM) NTO was developed using existing infrastructure at HSAAP to produce a new class of NTO with smaller particle size (<20 μm mean size). FEM NTO is currently produced on the production scale with over 20,000 lb of material produced to date.



**Figure 4 – FEM NTO**



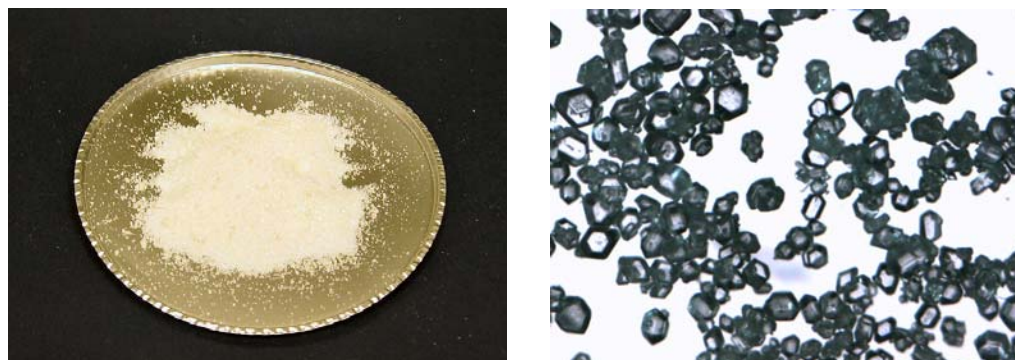
Fluid-energy milled (FEM) RDX was developed at HSAAP to produce a new class of RDX (~4  $\mu\text{m}$  mean size). FEM RDX has been shown to provide improved IM properties over regular grade RDX.

#### **5.4 High Bulk Density Nitroguanidine (HBD NQ)**

High bulk density nitroguanidine (HBD NQ) is currently produced at HSAAP on the production scale from low-bulk density nitroguanidine (LBD NQ) using a recrystallization procedure developed at Holston. HBD NQ is currently used as a major insensitive energetic ingredient in the IMX-101 formulation, with over 50,000 lb of material produced to date (3,000+ lb batch sizes). Although HBD NQ has slightly less energy output than RDX and NTO, it dramatically decreases the sensitivity of the formulated materials.

Current supplies of LBD NQ are provided from US army stockpiles and foreign sources in needle form, which is unsuitable for use in formulation activity. HSAAP developed an inexpensive and efficient proprietary recrystallization process to produce cubical HBD NQ with an average particle size of ~300  $\mu\text{m}$ . There is over a 100% increase in the bulk density from the starting material to the recrystallized HBD NQ. Key processing parameters studied for the recrystallization included NQ concentration, reaction temperature, agitation level, choice of crystal habit modifiers, and amount of foaming. The HBD NQ was evaluated based upon particle shape, size, and bulk density.

**FIGURE 5 – HBD NQ**



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## 6. CONCLUSIONS

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A new generation of IM melt-pour explosives is now available from BAE SYSTEMS at the Holston Army Ammunition Plant. For example, IMX-101 and IMX-104 have demonstrated excellent IM properties over TNT and Comp B, respectively, while having similar performance. Both explosives utilize a wide range of insensitive melt-pour ingredients. The synthesis and optimization of these ingredients have also successfully been developed and transitioned using existing manufacturing infrastructure at HSAAP. Some of the ingredients that readily available and manufactured at Holston include DNAN, NTO, HBD NQ, TATB, and FEM RDX. There are also plans to scale-up manufacturing of other novel ingredients including DNGU, and multiple types of TATB. During this effort, there remains a continuous R&D effort on the synthesis and optimization of novel insensitive ingredients and formulations.

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