# Characterization of Granular IMX-104

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U.S. Army Development and Engineering Command (ARDEC) has been evaluating 2,4-dinitroanisole (DNAN) based melt pour explosives such as IMX-101 and IMX-104 in PEO-AMMO legacy munitions and they have been garnering attention due to their low sensitivity to unplanned stimuli. These materials, designed to replace TNT and Composition B, have been qualified for use in the 155mm M795, 155mm M1122, and 105mm M1 HE Artillery Shells as well as M720A2 60mm and M821A3 81mm HE Mortar cartridges. Evaluation of ground flake IMX-104 pressed into standard warheads suggests that satisfactory performance can be achieved as the pressed density of the melt pours explosive increases, while maintaining the insensitive nature of the material. Previous laboratory work was successful in developing a suitable manufacturing method to produce granular IMX-104 suitable for high speed rotary pressing application. A fluorinated hydrocarbon based slurry coating system was selected for the initial slurry coating trials as 3-nitro-1, 2, 4-triazole-5-one (NTO), a key component of IMX-104 explosive formulation is highly soluble in water. Initial trials indicated the final material was similar to standard IMX-104 for both physical and thermal properties. Expanding on this laboratory work, both intermediate scale and large scale production trials were scheduled and attempted at Holston Army Ammunition Plant (HSAAP). BAE Systems was tasked with manufacturing granulated IMX-104 on production scale utilizing the newly developed laboratory manufacturing process as well as the existing manufacturing infrastructure at HSAAP.

This paper will detail the technical efforts to manufacture on the large scale granular version of melt pour IMX-104 formulation. Particular detail will be spent on comparison of the resulting granular version of IMX-104 to the standard melt cast material currently manufactured at HSAAP.

## Introduction

Pilot Scale (125 lbs) production of granular IMX-104 with different levels of wax was undertaken at BAE HSAAP facility utilizing fluorinated hydrocarbon based slurry coating system. Four batches with chlorez wax and indramic wax were produced at BAE to evaluate the material suitability for the pressed applications. The batch with indramic was deemed most suitable for press applications and was eventually down selected to produce the 600 lbs of material utilizing the same pilot scale equipment and the process

to ensure process feasibility and repeatability. The material produced from this effort is currently being utilized for energetic qualification and the material qualification in 155mm M795 IM projectiles to replace the existing melt-pour IMX-104 transfer charge and the PBXN-9 supplemental charge.

## Slurry Coating Process

The process involved charging all the IMX-104 ingredients (DNAN, NTO, and RDX), Indramic wax and the fluorinated hydrocarbon fluid in a 200 gallon Pfaudler glass lined reactor (figure 1). The wax was added to the formulation to improve the pressability of the formulation due to the brittle nature of DNAN. Temperature is raised and agitated for 30 minutes at max. Speed (125 rpm). Heating is then removed and the still is quenched to produce IMX-104 granules suitable for high speed press applications. Figure 2 below shows the granular IMX-104 with Indramic wax. Mixture from the still was discharged into the drying nutsches where performance fluid is recovered and pumped back into the drums for re-use.

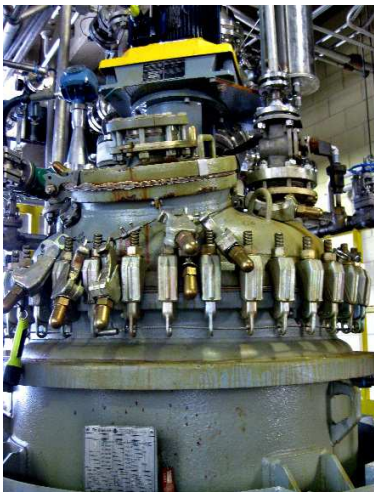

**Figure 1: Pfaudler reactor**

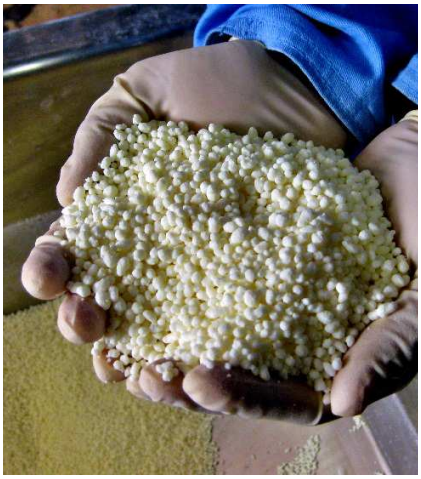

**Figure 2: IMX-104w/Wax**

## Differential Scanning Calorimetry (DSC)

The DSC was performed according to AOP-7, US 202.01.020 (MIL-STD-1751A (1072)) or STANAG 4515 where 20 mg of granulated IMX-104 with Indramic wax was subjected to a heating rate of 10°C/min until decomposition of the sample occurred. The sample endotherm(s), exotherm(s), onset temperature(s), and peak temperature(s) are recorded. Granulated IMX-104 with indramic wax exhibited an exotherm onset at 217°C, and an exotherm peak temperature at 257°C as shown in Figure 3. By comparison, regular flake IMX-104 exhibited an exotherm onset at 210°C, and an exotherm peak temperature at 224°C.

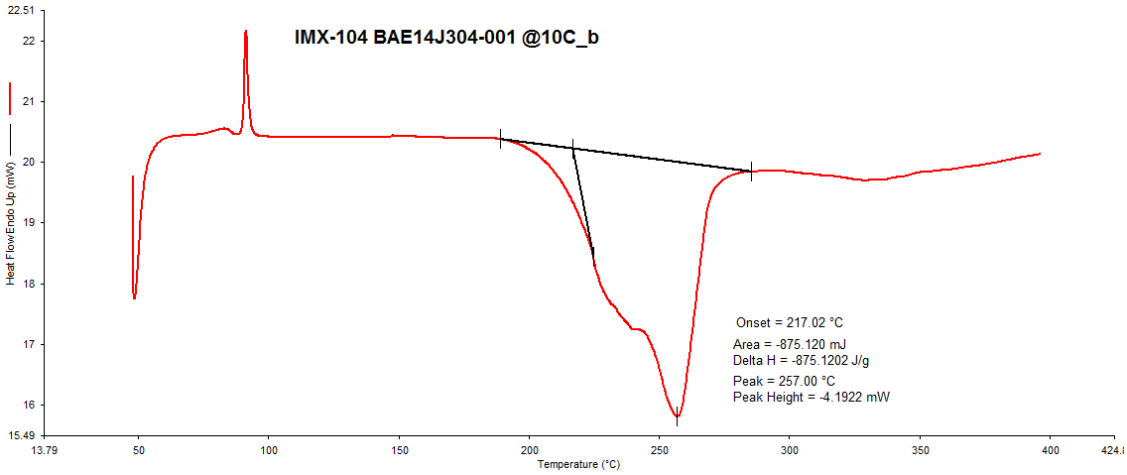


**Figure 3: Granulated IMX-104 DSC**

## Vacuum Thermal Stability (VTS) Testing

Vacuum Thermal Stability testing was performed in accordance to STANAG 4556 ED.1 (Explosives: Vacuum Stability Test). This standard testing procedure measures the stability of an explosive at an elevated temperature (100°C) under vacuum. The stability of a candidate explosive is determined by the amount of gas evolved. To qualify, the candidate explosive shall not exceed more than 2ml of gas evolved/gram for a 5 gram sample of explosive. The granular IMX-104 evolved 0.72ml of gas. 0.144ml/gram. For comparison, regular flake IMX-104 evolved 0.571ml of gas.

## Critical Temperature

Critical temperature (Tc) analysis was performed IAW AOP-7 202.01.021. Critical temperature is a measure of self-heating and is the temperature at which an energetic will self-initiate. Critical temperature is defined according to AOP-7, as the lowest constant surface temperature above which a given material of specific size and shape will catastrophically self-heat. The temperature is a factor of DSC measurements, material thermal conductivity, and material thickness. Passing criteria are:

• Self-heating should not cause deflagration

- Self-heating should not be detectable; i.e., less than 0.55°C from ambient temperature to 71 °C in the normal size and geometry
- The calculated critical temperature for a given mass and geometry should not be less than 82.2°C for 500 days at this temperature

The critical temperature determination was calculated using dimensions obtained from the 1-L cook-off test and the shape factor (2) for a cylinder. The granular IMX-104 critical temperature is 137.8°C. For comparison, the regular flake IMX-104 critical temperature is 164.33°C and therefore passed the criteria.

## Density

The density of granulated IMX-104 with Indramic wax was measured using a gas pycnometer IAW AOP-7, 102.01.071. The density was found to be 1.686 g/cc. For comparison, regular flake IMX-104 density was found to be 1.728 g/cc

## Coefficient of thermal expansion (TMA)

Thermo-Mechanical Analysis, the instrument performs measurements of thermal expansion coefficient of material as function of temperature. Heating rate was 5C/min. To define the thermal expansion coefficient an average was taken over the whole range. The thermal expansion coefficient for the granulated IMX-104 was determined to be 92.28e-6/C as shown in figure 4. For comparison, the regular flake IMX-104 was determined to be 138e-6/C.

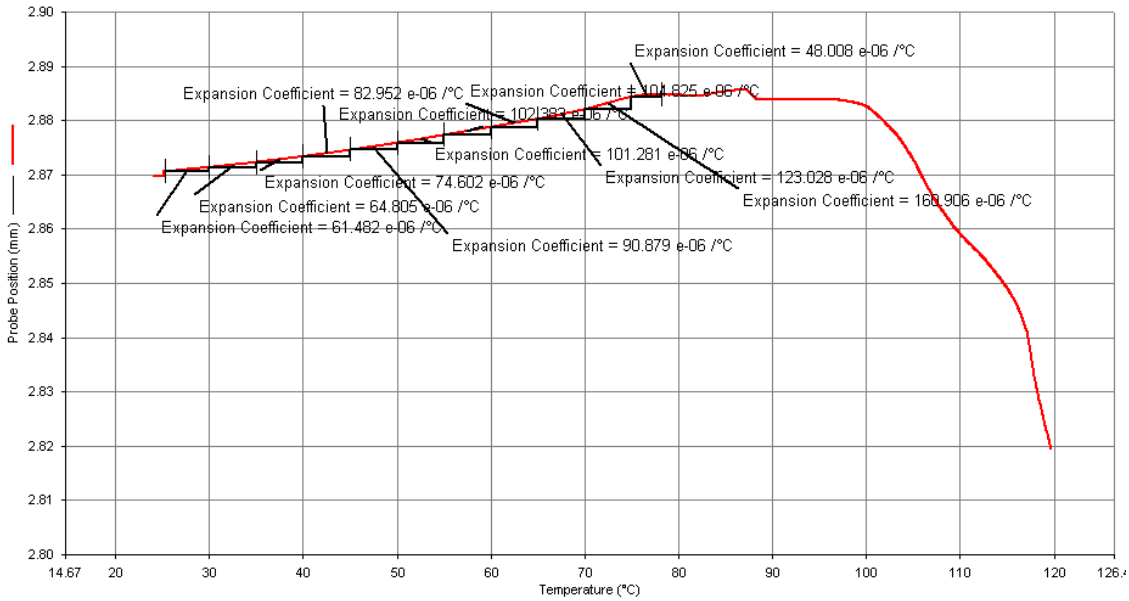


**Figure 4: TMA of Granulated IMX-104**

# SENSITIVITY TESTS

## Electrostatic Sensitivity (ESD)

The ABL ESD Test was used to determine the sensitivity of a material to electrostatic discharge. The approaching electrode electrostatic apparatus consists of a charging circuit and an approaching electrode assembly. The approaching electrode assembly is a pneumatic-operated device in which the upper electrode (needle) is lowered to a preset distance (0.015-inch) above the lower electrode (stainless steel sample holder) and immediately raised again to its initial position. Granulated IMX-104 with Indramic wax reacted at 0.031 J and did not react at 0.025 J in 20 trials. For comparison, regular flake IMX-104 also did not reacted at 0.25J

## Impact Sensitivity Test

The ERL, Type 12 Impact Tester, utilizing a 2 ½ kg drop weight, was used to determine the impact sensitivity of the sample. The drop height corresponding to the 50% probability of initiation is used to measure impact sensitivity. Granulated IMX-104 with Indramic wax did not reacted in 10 out of 100 trial at 100cm drop. This test result does indicate that granulated IMX-104 is insensitive to impact stimulus. For, comparison regular flake IMX-104 also doesn't reacts at 100 cm impact height.

The BOE Impact apparatus consists of an eight pound weight dropped onto a plunger-and-plug assembly which is in contact with the substance sample. The substance sample is placed on a die-and-anvil assembly confined in a cylindrical casing whose inside diameter is sufficient to permit free movement of the plunger and plug. Ten milligrams of the solid substance is loaded onto the die. The anvil and die are placed in the sample housing and the casing screwed down over them. The plug and plunger are then inserted on top of the sample. The drop weight is raised to the desired height and released. Ten trials are conducted at 4-inch drop height. The criteria used in the interpretation of this test are that a trial is considered positive if either an audible report or flame is observed. A sample is considered impact sensitive at a specific drop height if a flame or report is observed in at least 50% of the test trials. A sample which shows impact sensitiveness at a drop height of 4.0 inches or less is considered too sensitive for transport. Granulated IMX-104 with Indramic wax did not reacted in 10 out of 100 trial at a drop height of 4.0 inches. For, comparison regular flake IMX-104 also doesn't reacts at a drop height of 4 inches.

## Friction Sensitivity Test

The BAM Friction tester was used to determine the friction sensitivity of material. Approximately 30mg of sample was placed on the porcelain plate. The porcelain pin was

lowered onto the sample and a weight was placed on the arm to produce the desired load. The tester was activated and the porcelain plate was reciprocated once to and fro. The results are observed as either a reaction (i.e. flash, smoke, and/or audible report) or no reaction. An iterative procedure is used to determine the highest load at which no positive results are obtained in 10 trials. This value is considered the Threshold of Initiation Level (TIL). If a reaction occurs in ten trials, the load is reduced until no reactions are observed in ten trials.
Granulated IMX-104 with indramic wax did not reacted at 360 Newtons (N) in 10 trials. For, comparison regular flake IMX-104 reacted at 168.2 N and no reactions at 160 N

ABL friction test: The sample is subjected to vertical compression force under a non-rotating wheel, while the sample is moved in a horizontal direction on a sliding anvil. A sample is placed on the anvil, under the wheel, in a thin uniform layer (equivalent to one particle thickness for solids based on largest particle size in distribution). The wheel is lowered onto the sample and force is applied to the wheel. An initial force of 1,000 lb. (4450 N) is typical. Values of 1,800 lb. (8000 N) maximum to 10 lb. (44 N) minimum are used. A pendulum impacting on the edge of the anvil propels the anvil at a known velocity, perpendicular to the compressive force that is applied to the sample. An iterative procedure is used to determine the highest compressive force at which no positive results are obtained in 20 trials. This value is considered the Threshold of Initiation (TIL). A trial is considered positive if any of the following results is obtained:
a. Visible sparks
b. Visible flames
c. Audible explosion
d. Loud crackling noise
e. Detection of reaction products by a gas analyzer
Granulated IMX-104 with indramic wax did not reacted at 1800 (8000 N) in 20 trials. This is the maximum load available on this apparatus. For, comparison regular flake IMX-104 reacted at 128 N and no reactions at 120 N

## Shock Sensitivity Test

Large scale gap testing (LSGT) was performed IAW AOP-7, 201.04.001. The granulated IMX-104 with indramic wax LSGT pellets were pressed and loaded into the 1.5-in. diameter by 5.0-in. long steel tubing that was supported by a 0.375-in. thick witness plate. A detonator sat on top of penolite booster pellets that were separated from the test sample by a series of card gaps. The clear cut hole on the witness plate determined whether the test was a go or no go. The 50% point between go and no go for granulated IMX-104 baseline (p=1.66) is 155 cards (36.1 kbars). For Comparison, LSGT card gap value for regular flake IMX-104 melt casted into the tubes mentioned above is120 cards (49.6 Kbars)

## The 5-Second Explosion Temperature Test

No. 8 blasting caps are loaded with 30 mg of sample material pressed at 230 lb. then 30 mg of talcum powder pressed at 230 lb. The woods metal bath is heated to a desired temperature. The blasting cap is lowered into the molten woods metal bath and timer is started. When the blasting cap explodes, the timer is stopped. Successive tests are conducted in the same manner to obtain time-to-explosion temperature values over a time range of 1 to 7 seconds. The test data are plotted as the natural logarithm of the time-to-explosion vs. the reciprocal of the absolute temperature. This results in a straight line plot and a linear regression curve fit program is used to determine the temperature value in degrees Centigrade for the 5-second time-to-explosion. The 5-second explosion temperature for granulated IMX-104 with indramic wax is 253°C. For comparison 5-second explosion temperature for regular flake IMX-104 is 245°C

## The Minimum Concentration of a Dust Cloud Test

The standard Bureau of Mines Hartmann apparatus was used to determine the explosive dust flammability/explosibility of the lean concentration limits. A pre-weighed explosive dust sample is placed in the sample holder, dispersed via compressed air and flowed through an established electric arc. The energy of the arc is about 10 Joules. An iterative procedure is used to determine the lowest concentration at which no positive results are obtained in 10 trials. This value is considered the Threshold of Initiation Level (TIL). Testing is begun at 1.00 grams of explosive powder or lower if experience warrants it. If a reaction occurs in ten trials, the amount is reduced until no reactions are observed in ten trials. The minimum concentration of a dust cloud for granulated IMX-104 with Indramic wax is 100 to 200 grams per cubic meter. For comparison, the minimum concentration of a dust cloud for regular flake IMX-104 is 600 to 700 grams per cubic meter

## Thermal Stability Test

The granulated IMX-104 with Indramic sample was placed in a constant temperature explosive-proof oven IAW TB-700, paragraph 5-4.c. The temperature of the oven was kept at a constant 75°C for a period of 48 hrs. No discoloration, deformity, weight loss, or evidence of instability was observed. The weight of the sample started at 50.1051 g and ended at 50.0732g, a weight loss of 0.0319 g or 0.06% was observed. Passing criteria for this test is no visual evidences of ignition, explosion, and significant color change indicating some reaction or weight loss. This test is considered a pass. For comparison, regular flake IMX-104 observed a weight loss of 0.07%.

## Small Scale Burn Test

A small scale burn test (un-confined) was performed IAW TB 700-2, paragraph 5.4a, to determine the level of reaction of IMX-104 material in a fire. The 125-g samples of granulated IMX-104 with indramic wax were placed into plastic beakers. The plastic beakers were placed on a kerosene-soaked sawdust bed and ignited. Three tests were performed on individual samples. The result was a slow burn in all three tests. Passing criteria for this test was no occurrence of explosion or detonation; the granulated IMX-104 with indramic wax passed the test. For comparison, regular flake IMX-104 also passed this test.

## Cap Sensitivity

According to TB 700-2, paragraph 5-6.a, this test is designed to determine the sensitivity Of a substance to the shock from a standard detonator or blasting cap. The test yields quantitative and unambiguous results for very insensitive explosive material. For IMX-104, a no. 8 blasting cap was used. The material is deemed "cap sensitive," if in at least one trial a) the lead cylinder is compressed from its initial length by an amount of 3.18 mm or greater and b) the witness plate shows no penetration. Granulated IMX-104 with Indramic wax is not cap sensitive and passed this test. For comparison, regular flake IMX-104 also passed this test.

## Conclusions

The initial qualification test results indicate that Granular IMX-104 meets and exceeds the requirements for Material Release Qualification Program and has very similar properties to flake IMX-104. It is insensitive for a booster explosive. The ingredients used in this composition are currently available in National Technology and Industrial Base (NTIB). BAE Holston has produced over 1,000 lbs of Granular IMX-104 to date. This effort directly supports the PM-CAS MCP of Granular IMX-104 into the 155mm M795 Artillery Projectiles as both the transfer charge and supplemental charge.

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