PROCESS DEVELOPMENT OF A NITRAMINE PROPELLANT FOR THE GAU-8 30 mm GUN SYSTEM*

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Summary

The Air Force is actively pursuing an alternative to the double-base propellant used in the 30 mm GAU-8 system on the A-10 aircraft. To suppress the secondary gun gas ignition of that formulation, a 2% level of potassium nitrate is required. This has generated problems with combustion residue buildup in the engines from the hot gun gas flow. A nitramine propellant having the advantage of a higher delivered impetus, along with a lower flame temperature, has demonstrated increased delivered velocity with a reduction in the potassium salt concentration to 0.7%.

The Naval Ordnance Station has just completed the manufacture of a 2268-kg (5000-lb) sample lot for the Air Force to be used for actual flight test and evaluation. The various aspects of adapting conventional solvent processing techniques to process a gun propellant with a high solids loading of RDX and TAGN will be discussed. The development of a novel spray coating process to add the inhibitor to the seven-perforated grains has improved the ballistic uniformity of this propellant. The results of safety, thermal stability, and combustion analyses will be presented for this new nitramine formulation.

Introduction

The Naval Ordnance Station, Indian Head (NAVORDSTA/IH) has been involved in the nitramine propellant development work for the Air Force since 1979. The initial focus was on adapting a nitramine formulation and associated processing techniques developed by Rocketdyne Division, Rockwell Int., Canoga Park, CA, and Hercules, Inc. at the Allegany Ballistic Laboratory (ABL), Cumberland, MD, to our own solvent process equipment [1,2,3]. The propellant composition, designated RGP-150, selected for evaluation consisted of the following ingredients:

Triaminoguanidine nitrate (TAGN), ground	45.0%
RDX or HMX, ground	29.5%
Nitrocellulose (12.6% N)	20.0%
Isodecyl perlargonate	4.8%
Potassium sulfate	0.5%
Resorcinol	0.2%

Initial efforts involved a series of process variables studies to determine the optimum solvent level and ratios and solvent and nitramine addition sequence during mixing for the manufacture of quality propellant strands. Both RDX and HMX were used as the secondary nitramine filler during the small-scale processing phase of the development cycle. Work culminated in the production of a 454-kg (1000-lb) lot of propellant using TAGN and HMX as the two nitramine fillers [4].

Follow-up work involved the RGP-150 formulation with RDX as the secondary energetic ingredient. Several web dimension iterations and coating techniques were investigated prior to the production of a 2268-kg (5000-lb) lot of propellant for automatic gun firings. Two problems surfaced during the initial ballistic test phase of this lot: (1) gun chamber pressures at the upper operating temperature limit of 71°C (160°F) were above the ballistic performance requirements for the round, and (2) sustained secondary gun gas ignition was evident during automatic gun firings. To correct these problems, a plasticizer change was made, the flash suppressant level increased and new process controls instituted. The new composition, designated EGP-152-R, is as follows:

TAGN, ground	45.0%
RDX, ground	29.3%
Nitrocellulose (12.6% N)	20.0%
Dioctyl phthalate	4.8%
Potassium sulfate	0.7%
Resorcinol	0.2%

A 2268-kg sample lot of this propellant was manufactured in May 1984 and shipped to Aerojet Ordnance Company for loading in target practice (TP) rounds for automatic ground and air gun firings. Single shot Mann barrel firings conducted at Eglin AFB and Aerojet Ordnance Company have substantiated the ballistic acceptability of this propellant lot.

Processing

Adaptation of the solvent process technique and process controls for conventional double- and triple-base propellants was necessary in order to manufacture a gun propellant with such a high solids loading. A reliable surface inhibiting technique had to be developed to provide a propellant that was ballistically uniform. A discussion of the various phases of the batch solvent process and the changes that were necessitated will follow. Figure 1 shows the overall manufacturing process flow description.

Nitramine grinding

The preparation of the nitramine ingredients prior to mixing requires that the weight mean particle diameter be reduced to 4 to 6 micron in size
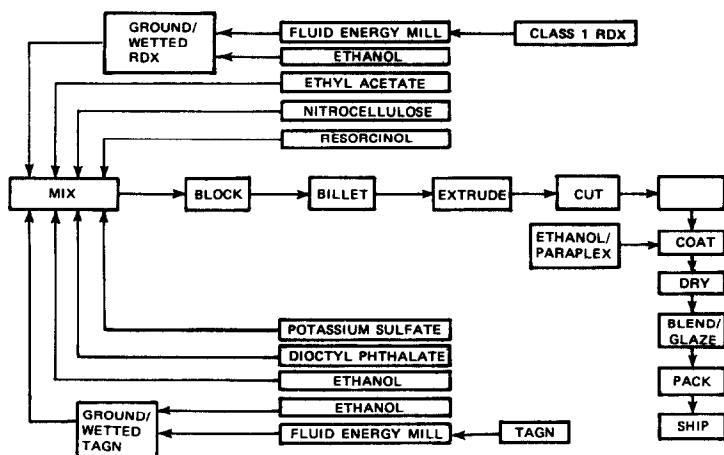
Fig. 1. Process schematic.

so as to obtain the proper combustion characteristics when incorporated into a propellant [5]. ABL determined that jet milling was more practical than either ball milling or vibro-energy milling [3]. This method of particle size reduction was continued at NAVORDSTA/IH with the installation of a 5-cm (2-in.) Jet-O-Mizer mill. The grinding facility used is equipped with a feed hopper, screener, screw feeder, fluid energy mill, and baghouse collection system. Figure 2 provides a process flow sheet for the operation.

The energetic material is received alcohol wet (TAGN) or water/alcohol wet (HMX and RDX). The wet material is placed on metal trays and forced air oven dried at 60°C (140°F) for approximately 5 days. Once dried, the material is added to a feed hopper in approximately 45-kg (100-lb) increments and then gravity fed to a Sweco screener to remove any foreign material and large nitramine agglomerates. A loss-in-weight screw feeder moves the material into the inlet air stream line into the jet mill. The material enters the mill from the bottom where it is impacted by two streams of high-velocity, humidity-controlled air. The particles are reduced through the abrading action of particle-to-particle contact. The ground material exits the center of the jet mill through a size classifier. The particles enter a collector where they accumulate in bags which are occasionally pulsed to release this material to a primary receiver. The conveying, grinding, and pulsating air vents to atmosphere. A dry sample for particle size analysis is taken from each run. The RDX is stored dry in preweighed batches, whereas the batches of TAGN are alcohol wetted prior to storage.

Ingredient preparation

The fine nitramine solids encompass approximately 74% by weight of the entire formulation. To ensure that these particles are evenly dispersed throughout the mix, the TAGN and RDX are both wetted with
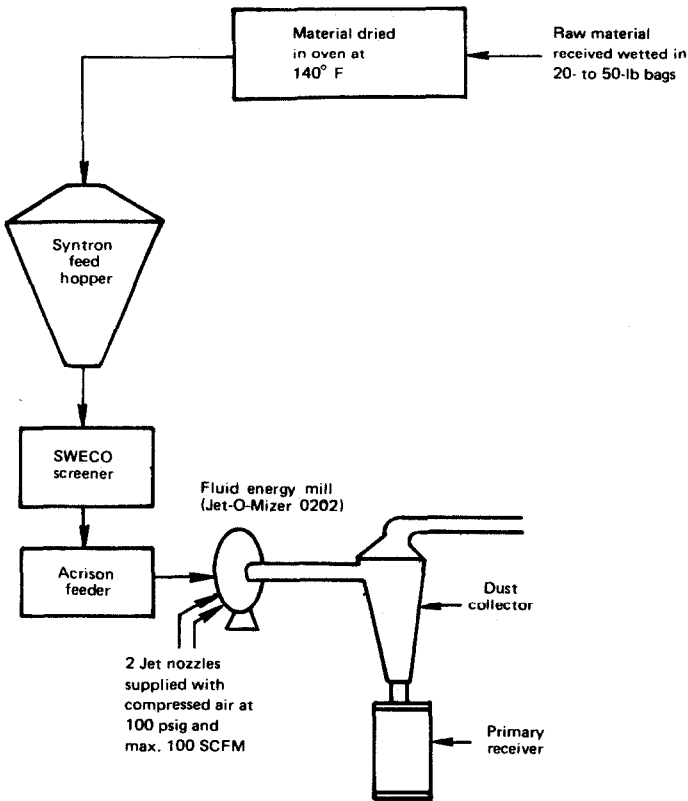
Fig. 2. Nitramine grinding system.

ethanol prior to addition to the mixer. Since TAGN is hygroscopic, it is stored in ethanol to reduce the tendency for the TAGN crystals to bridge and agglomerate. The RDX is stored dry and must be slurried with the wetting medium 24 h prior to addition to the mixer. The slurry process is a carryover from the composite-based casting powder days. The selection of ethanol over ethyl acetate is critical in the prevention of nitramine agglomeration during the mixing operation.

The nitrocellulose is received 20% to 30% alcohol wet and added to the mixer in this state. A total volatiles measurement is conducted prior to mixing to determine the proper amount of wetted material to be added.

Mixing

The mixing consists of three phases: (1) homogenization of ingredients, (2) solvent removal by pressurized air, and (3) cooling of the propellant mix dough.

The ingredients for the propellant are mixed to homogeneity in a dual-solvent medium using a horizontal, sigma-bladed mixer. The active solvent

is ethyl acetate while ethanol acts as the carrier liquid to ensure adequate solid ingredient dispersion during the mixing. The total solvent level using a 1:1 ratio of ethyl acetate to ethanol is 50% by weight of the dry weight of the mix. The raw ingredients are incrementally added with the alcohol-wet nitrocellulose, potassium sulfate, resorcinol, dioctyl phthalate, ethanol, and over half of the ethyl acetate added first to start the gelatinization of the binder. The alcohol-wet TAGN and additional ethyl acetate is then incorporated into the mix followed by the slurried RDX and the final increment of ethyl acetate. The mixer water jacket temperature throughout this phase is maintained at 60°C (140°F) in order to increase the solubility of the nitrocellulose in the ethyl acetate and maintain a mix viscosity within the limits of mix equipment.

The solvent removal, or blowdown, phase involves the attachment of an air spray nozzle to the mixer lid. The lid is left partially open as the mixer rotates allowing the air convection to increase the solvent evaporation rate. The direction and flow rate of the air are controlled with the intent to avoid the introduction of porosity into the mix dough through air entrapment. Removal of solvent continues until the mix attains a dough consistency, qualitatively determined by operator experience, that can be extruded in a pressure range known to yield an acceptable product.

The cooldown phase consists of reducing the water jacket temperature to 7°C (44°F) and mixing for 10 min prior to pulling the mix. This allows for the working of the propellant through the remainder of the batch process steps without excessive additional solvent loss.

Blocking and billeting

The mix dough is compressed into a solid cylinder under 7-MPa (1000-psi) hydraulic ram pressure and for a 2-min dwell time using a 30-cm (12-in.) vertical blocking press. This step is used to remove any occluded air trapped during the mix phase.

To date, all propellant processing has been conducted using pilot-scale equipment. A billeting step has been required during this scale-up operation to produce billets (charges) for the 10-cm (4-in.) finishing presses. A full-scale production line would have finishing presses capable of accepting the blocks as charges directly.

The billeting press is a basic 30-cm (12-in.) diameter horizontal solvent extrusion press equipped with a breech that holds a single 9-cm (3½-in.) diameter die. A strainer plate is placed at the entrance of the die, along with a 16-mesh stainless steel screen. The strainer plate and screen assembly simulates the macaroni press operation normally used in single-, double-, and triple-base production. The purpose of that step is to provide additional shear forces to work the propellant and filter out incompletely colloided nitrocellulose. With the use of cotton linters rather than wood pulp as the source of 12.6% nitrated nitrocellulose, there may not be a need for this step in the processing of nitramine gun propellants. Preliminary results,

based upon density measurements and closed bomb testing, showed no difference between strained and unstrained nitramine propellant samples. The screen removed agglomerated nitramine particles during the early process development work. Adjustment of the ingredient preparation solvent and nitramine addition cycle have alleviated the agglomeration problem.

Extruding and cutting

The extrusion process employs four vertical solvent presses each containing four dies. A 16-mesh stainless steel screen is used to retain any hard propellant pieces that may have formed in the billets and could potentially damage the die pins. The extrudate is a seven-perforated, circular strand which is collected in a coil for the cutting operation.

Modifications to the strand collection system and holding time prior to cutting were necessitated after automatic gun firings of the isodecyl perlargonate-plasticized propellant lot produced sustained secondary gun gas ignition. Visual inspection of the individual propellant grains showed that there were approximately 4% deformed grains with closed perforations. The closed perforations caused incomplete propellant burnout during the interior ballistic cycle, resulting in sliver ejection from the muzzle which was believed to be the ignition source for the secondary flash problem. Analysis of the individual processing steps pinpointed the problem to the strand collection trays. Aluminum ridges on these trays were used to separate the four polyethylene cones which collected the extrudate from each press. As the strands coiled, there was a tendency to overlap the ridges and deform the propellant. The ridges were eliminated and the strands were visually inspected during the cutting phase to maintain geometric integrity of the propellant. To ensure that the propellant did not smear over the perforations during the cutting operation, the extrudate was held for 15 min instead of cutting immediately after extrusion.

The strands are cut to length on a cannon powder cutter using a fine water mist as a coolant. Nine kilograms (20 lb) of the green propellant are put on each drying tray and then stacked on a cart.

Drying

The propellant is held at ambient temperature for a minimum of one day to prevent a hard crust from forming, and then dried in a forced air oven at 60°C for five days. Total volatiles content measured after the drying cycle is approximately 0.2%.

Coating

To meet the projectile velocity requirements, the EGP-152-R propellant is coated with a polyester surface inhibitor of Paraplex G-54 (Trademark of C.P. Hall Co.). This inhibitor coating delays the ignition of the propellant outer surfaces until the Paraplex is completely burned. Initially, the propellant grain's uncoated perforation surfaces are ignited. This causes the burn

to be progressive, and the high-velocity requirement can be attained at the lower charge weights.

The surface coating should cover all exterior surfaces evenly, and the perforations should remain open. Uneven application of the inhibitor coating and partial or complete closure of the propellant grain perforations can cause a high degree of ballistic variability. Three process methods were developed to coat the propellant: alcohol evaporation, water extraction, and spray coating.

Alcohol evaporation process

The alcohol evaporation process, developed by ABL for coating Paraplex G-54 on the propellant, was adapted on a laboratory scale using a 500-ml (0.1-gal) fluted flask, water bath, and rotary vacuum dryer. A mixture of alcohol and Paraplex was added to a 180-g (0.4-lb) propellant sample. The vacuumed flask was rotated in a 60°C water bath until the alcohol was evaporated and removed from the flask. The propellant was coated on the surfaces as the Paraplex precipitated out. The process was then scaled to a 400-l (106-gal) sweetie barrel. This barrel was not equipped with a vacuum, and the procedure was changed to air blowdown along with elevated temperature to evaporate the alcohol. This technique was abandoned because of several problems: (1) coating over of the perforation openings, (2) nonuniform coating of the propellant grains, and (3) loss of inhibitor to the barrel surfaces during the evaporation phase.

Water extraction process

Hercules, Incorporated, at Kenvil, New Jersey uses a water extraction technique for deterrent coating their double-base propellant. This process was adapted as our next attempt to coat the propellant at a small-scale level. The propellant bed was initially soaked in alcohol at 60°C in an attempt to fill the perforations with alcohol and prevent coating of the perforation surfaces during the extraction phase. A Paraplex and alcohol mixture was charged into the 400-l barrel which was rotated to ensure a complete saturation of the Paraplex—alcohol mixture into the propellant bed. Excess water was added, and the Paraplex was precipitated from the alcohol solution. After further rotation, the alcohol—water solution was discarded, and the coated propellant was then dried in a forced air oven for 24 h at 60°C.

This coating procedure has several inherent processing problems. From microscopic analysis and ballistic results, the Paraplex was applied unevenly to the propellant grain surface and in severe cases the perforations were coated closed. The procedure is labor intensive and requires the disposal of a relatively large amount of waste in the form of the alcohol—water solution. Mann barrel firings of propellant coated using this technique resulted in ignition problems that will be discussed in the next section.

Spray coating process

The spray coating procedure was developed by NAVORDSTA/IH engineers in order to reduce the coating variability of the water extraction process. A Paraplex and alcohol solution is air sprayed directly onto the propellant bed in the 400-l barrel. The pressurized air is externally mixed with the alcohol and Paraplex. This atomization effect separates the Paraplex and the alcohol. The viscous Paraplex droplets are fan sprayed directly onto the propellant bed, and the alcohol forms a fine mist which is blown out of the barrel. As a process engineering problem, the spray characteristics can be broken down into spray pattern, capacity, spray angle, spray atomization, impact, and velocity.

Spray pattern: A fan or flat spray pattern causes the liquid to form a flat sheet or flattened ellipse which is specifically used for coating surfaces [6].

Capacity: The flow rate dictates the type of nozzle; i.e., air or airless. This system has flow rates of 75 to 500 g/min (0.2 to 1.1 lb/min). At the lower ranges, an airless system cannot handle the flow rates without effecting its atomization properties. The air nozzle system can handle this range of flow rates and have good atomization [7].

Spray angle: Tabulated spray angles indicate approximate spray coverages based on water. Liquids more viscous than water form relatively smaller spray angles. For an effective spray angle, the distance should be set empirically [7].

Spray atomization: Fine atomization is most easily obtained with air atomization. For the special case of highly viscous liquid, an external air mix nozzle, where the compressed air is injected into the liquid stream external to the liquid nozzle head, achieves the fine liquid atomization needed for coating. For the external air mix nozzle, atomization is controlled by varying the air pressure and not by changing the liquid flow rates. This property allows the nozzle to handle highly viscous material at low flow rates [7].

Impact and velocity: Impact and velocity are not major variables for this unit operation. These factors are important in solid stream applications.

The spray coating procedure is neither time nor labor intensive. The 400-l barrel is normally charged with 23 kg (50 lb) of propellant, and the barrel is rotated at ambient temperature to ensure maximum exposure of the propellant to the spray. The spray system is turned on for one minute. The liquid flow rate depends upon the level of Paraplex G-54 coating required for the propellant's ballistic performance. After the coating cycle is finished, the barrel is heated by the water jacket to 60°C and glazed with 0.25% by weight of graphite. The coated propellant is dried in a forced air oven for 24 h at 60°C. The graphite addition reduces the conglomeration of the coated propellant bed during the drying cycle as well as aids in obtaining the required loading density of the seven-perforated propellant charge.

The spray coating method proved through microscopic analysis and ballistic results that the application of the Paraplex gave an even coating with no grain perforation closures. Table 1 shows that the water extraction method uses 125% more material than the spray coating method. The spray coating process eliminates the alcohol—water waste and reduces the labor requirements.

TABLE 1

Deterrent coating process ingredients

Material	Water extraction level (%)	Spray coating level (%)
Propellant	100.0	100.0
Alcohol	25.2	1.0
Paraplex G-54	1.0	1.0
Water	100.0	0.0

Ballistics

Propellant coated using the water extraction method developed ignition problems during 30 mm Mann barrel firings at NAVORDSTA/IH. To measure chamber pressure, the Mann barrel was instrumented with a piezoelectric gage, which was located 16.5 cm (6.5 in) from the breech end of the barrel. The projectile must travel 2.5 cm (1 in) prior to uncovering of the gage port. Propellant samples from the same mix were coated using both the water extraction and spray technique. Figures 3 and 4 show the difference in muzzle velocity and action time (the time from striking of the percussion primer to muzzle exit of the projectile) for the two techniques at identical charge weights over the operating temperature range of the propelling charge. The spray-coated propellant exhibited an increase in muzzle velocity and a decrease in action time. Investigation of typical pressure—time curves obtained with ambient temperature firings show that this fact can be attributed to an ingnition delay evidenced by an ignition saddle for the water extraction-coated propellant (Fig. 5). Table 2 shows that the spray-coated propellant is more ballistically uniform based on the velocity standard deviation of five shots. Both propellant samples were fired at a charge weight of 145 g (0.3 lb).

Combustion analysis

Relative quickness (RQ) and relative force (RF) measurements obtained in closed bomb testing were used as an aid in determining the amount of inhibitor coating to apply to the nitramine propellant to match the ballistic performance requirements for the TP round. The double-base propellant, designated HC-25, presently manufactured at Hercules, Inc., Kenvil, NJ
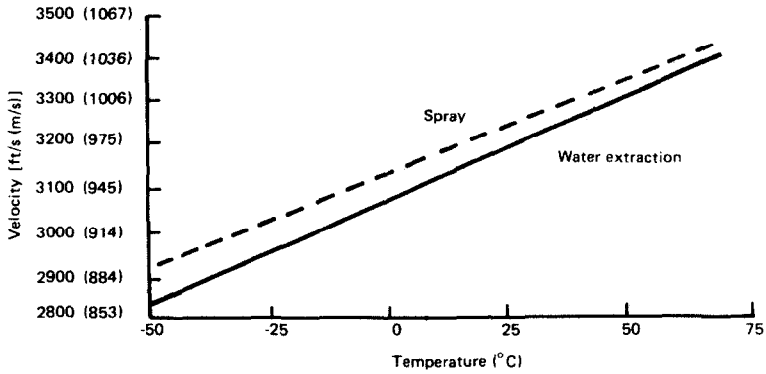
Fig. 3. Velocity versus temperature for water extraction and spray coating.
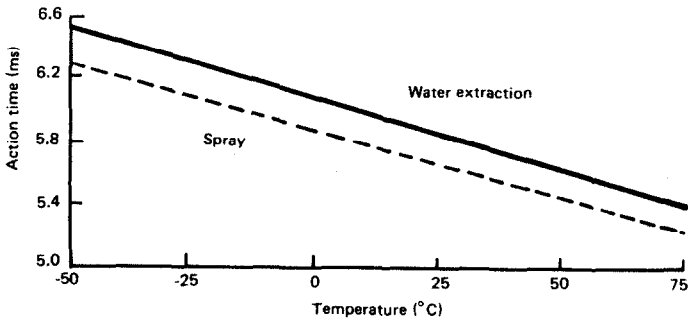

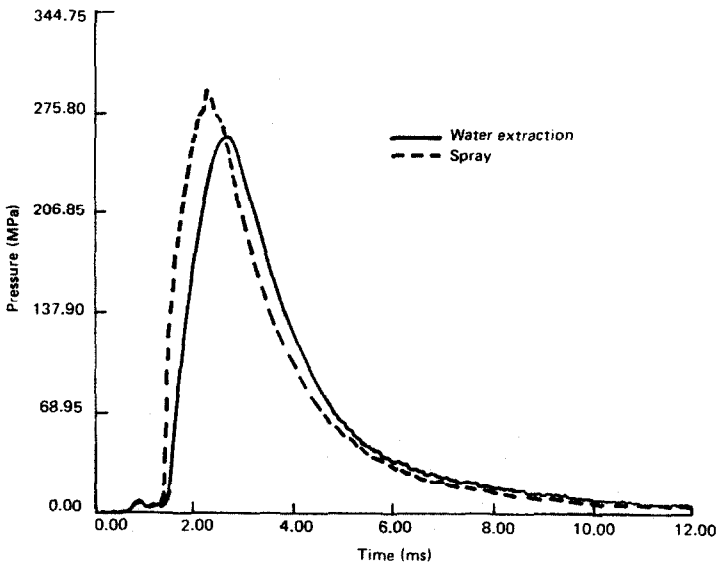Fig. 4. Action time versus temperature for water extraction and spray coating.

Fig. 5. Pressure versus time for water extraction and spray coating.

TABLE 2

Velocity uniformity results of alternative coating techniques

Temperature (°C)	Average maximum pressure (MPa)	Average muzzle velocity (m/s)	Muzzle velocity standard deviation (m/s)
Water extraction-coated propellant			
−50	223.6	867.2	9.4
21	268.3	957.1	6.1
71	325.5	1031.4	5.5
Spray-coated propellant			
−50	251.2	892.1	5.5
21	287.2	971.7	2.1
71	351.9	1045.8	2.1

and loaded in the TP rounds at Aerojet Ordnance Co., Downey, CA was used as the reference propellant. The results in Table 3 show that the typical uncoated nitramine propellant is 14% more energetic than Hercules' present propellant and requires inhibiting to match chamber pressures and muzzle velocities at comparable charge weights.

Safety

A series of safety and stability tests were conducted on the EGP-152-R propellant to determine the hazard classification. Based on the results presented below, the propellant was assigned a DoD Hazard Class/Division 1.3, the same classification as the present propellant being replaced.

Impact (5 kg weight)	175 mm
Friction (ABL sliding)	>980 lbf (4360 N)
Electrostatic discharge	>12.5 J
Card gap	<25 cards
Unconfined burn	Negative, sample burned in 11 s
Cap test (No. 8 cap)	Negative, no detonation, no burn
Thermal stability (DSC)	170°C onset
	210°C peak

Conclusions

As a result of the process development work conducted on the nitramine propellant formulation, the following conclusions can be made:
(1) A ballistically acceptable nitramine propellant can be produced using conventional solvent process equipment and techniques.

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TABLE 3

RQ and RF results of uncoated nitramine propellant

Sample	ΔP/ΔT (GPa/s)				Maximum pressure (MPa)	RQ (%)	RF (%)
	at 34.5 MPa	at 69.0 MPa	at 103.4 MPa	at 137.9 MPa			
HC-25	13.1	32.8	54.7	68.3	221.1	—	—
EGP-152-R	18.1	33.0	57.7	79.1	251.5	115.0	113.7

(2) Nitramines are wetted with ethanol prior to addition to the mix to ensure adequate dispersion and prevent agglomeration.
(3) Holding the extrudate for 15 minutes prior to cutting prevented smearing over of the grain perforations.
(4) The spray coating technique provided a propellant that was ballistically more uniform than either obtained using the alcohol blowdown or water extraction technique.
(5) The nitramine propellant, EGP-152-R, had sensitivity characteristics comparable to that of the standard double-base propellant HC-25.

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