# Evaluation of SNPE I-RDX as a Solid Rocket Propellant Ingredient

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ABSTRACT

A study was conducted to evaluate I-RDX that was synthesized by the French company, SNPE, as a rocket propellant ingredient. Because of the material's proposed insensitivity compared with standard RDX, the testing focused on chemical composition, particle morphology, compatibility with routine propellant ingredients, and sensitivity. A side-by-side comparison of the material with standard Holston RDX was conducted. Chemical analyses of the material included liquid chromatography (chemical purity), ICP (metals impurity), and X-ray diffraction (polymorphs). Particle size analysis was conducted to determine the average diameter and modality of the material. An initial screening of the thermal stability of the raw material was conducted using differential scanning calorimetry (DSC). The same method was applied to determine compatibility of the material with a combination of routine propellant ingredients. Routine testing for lab hazards was carried out to address handling concerns. These tests included friction and impact sensitivity. Once the initial screening had been completed, several one-pint mixes were made to determine the processability of the material. Ballistic data was collected via strand burning rate analysis. From this data, a comparison of pressure exponent was also determined.

## INTRODUCTION

In an ongoing effort to evaluate new materials for the desensitization of solid rocket propellant, the U. S. Army AMCOM Propulsion and Structures Directorate obtained a quantity of SNPE's RDX for a side-by-side comparison with standard RDX of like particle size. SNPE's RDX is reputed to be less sensitive than standard RDX. To distinguish the two materials, the SNPE material is referred to as I-RDX in the body of this paper.

A typical minimum signature propellant formulation was selected as the test basis for the evaluation. The basic formulation is shown below in Table 1:

| Ingredient | % |
|---|---|
| Pre-polymer / Curing Agent (PGA / N100) | 7.48 |
| Nitrate Ester Plasticizers (BTTN / TMETN) | 27.00 |
| RDX | 62.00 |
| NC | 0.46 |
| MNA | 0.50 |
| Lead Citrate | 1.50 |
| ZrC | 1.00 |
| TPB / MA | 0.06 |

Table 1 - Basic Minimum Signature Propellant Formulation

For chemical analyses, standard 17µ RDX was evaluated along with the as-received SNPE material (~200µ) and the 200µ standard RDX. Testing of the material centered around four major areas: chemical analysis, compatibility, sensitivity, and ballistics.

## EVALUATION

### Chemical Analysis:

Samples of the SNPE I-RDX were submitted for chemical analysis to verify composition and purity. Both the I-RDX and standard RDX materials were analyzed by x-ray diffraction spectroscopy (XRD), Fourier transform infrared spectroscopy (FTIR), density, inductively coupled plasma atomic emission spectroscopy (ICP / AES), and high pressure ionic chromatography (HPIC).

### Density Determination

A gas pycnometer method was used to determine density values of the three RDX samples. The results of these tests are shown in Table 2.

| TEST | RESULTS | ANALYSIS |
|---|---|---|
| Density | I-RDX (200μ) - 1.739 g / cc | No differences noted in density between samples of like particle size. |
| | RDX (200μ) - 1.735 g / cc | |
| | RDX (17μ) - 1.684 g / cc | |

Table 2 - Density Determination.

ICP Analysis

No metals were found in the samples. The metals analyzed for and not found were: Mo, Cr, W, Zn, Pb, Co, Cd, Ni, Fe, B, Si, Mn, Mg, Sn, Bi, V, Cu, Ti, Zr, Ca, Al, Sr, Cs, Ba, Na, Li, and K.

X-Ray Diffraction

X-ray diffraction analysis indicated the crystalline structure of the I-RDX sample was closer to the 17μ RDX sample than to the 200μ RDX sample, but neither matched very well with the I-RDX sample. Both the 17μ and 200μ RDX samples were more similar in crystal structure to each other than either was to the I-RDX sample.

| **Peak #** | **I-RDX** | **RDX, 17 μ** | **RDX, 200 μ** |
|---|---|---|---|
| 1 | 4.15 | 3.03 | 6.71 |
| 2 | 5.72 | 4.93 | 4.32 |
| 3 | 2.43 | 4.33 | 2.56 |
| 4 | 4.92 | 6.71 | 4.95 |
| 5 | 2.76 | 5.06 | 5.07 |
| 6 | 4.96 | 3.29 | 6.50 |
| 7 | 4.37 | 4.02 | 4.01 |
| 8 | 3.75 | 3.25 | 3.05 |
| 9 | 3.03 | 2.75 | 5.12 |
| 10 | 5.91 | 3.49 | 2.80 |

Table 3 - Comparison of 10 Strongest Peaks from X-Ray Diffraction Patterns of RDX Samples.

The values in the first column refer to the relative peak intensities of the diffraction patterns. The values in columns 2-4 are the d-values, in Angstroms, of each sample derived from their respective diffraction patterns. D-values are the distances between reflective planes of atoms in a crystalline structure which produce constructive interference. They are indicative of the crystal structure of a material and can be matched by absolute value as well as order of match.

The better the match between the d-values of diffraction patterns of materials, the more similar the crystal structure of the materials.

| I-RDX | RDX, 17 µ | RDX, 200 µ |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | 4.93 (2) | |
| 5 | 2.75 (9) | |
| 6 | | 4.95 (4) |
| 7 | | |
| 8 | | |
| 9 | 3.03 (1) | 3.05 (8) |
| 10 | | |

Table 4 - Matches Between Peaks of I-RDX and RDX, 17µ and RDX, 200µ Samples

This table indicates the matches between the d-values of I-RDX and the 10 strongest peaks from the other 2 samples. A match is considered as being valid if the d values agree within $\pm$ 0.02 Angstroms. The value in parenthesis indicates the peak strength associated with that d value.

| Peak # | RDX, 17 µ | RDX, 200 µ |
|---|---|---|
| 1 | 3.03 | 3.05 (8) |
| 2 | 4.93 | 4.95 (4) |
| 3 | 4.33 | 4.32 (2) |
| 4 | 6.71 | 6.71 (1) |
| 5 | 5.06 | 5.07 (5) |
| 6 | 3.29 | |
| 7 | 4.02 | 4.01 (7) |
| 8 | 3.25 | |
| 9 | 2.75 | |
| 10 | 3.49 | |

Table 5 - Peak Matches Between RDX, 17µ and RDX, 200µ Samples.

This table indicates there is a better match between the crystalline structures of the RDX, 17µ and RDX, 200µ samples than either sample matches with the I-RDX. However, the structures of the two RDX samples do not match perfectly, either in d-value or relative intensity. This can be due to a number of factors, some of which include; differences in processing of the 2 samples, such as a difference in cooling rates, which can favor one crystalline structure over another; differences in particle size, which affect the packing fraction of the material, which in turn directly impacts the d value of the diffraction pattern; differences in sample preparation for x-ray analysis, such as preferred orientation; differences in amount of contamination in the samples, and so on. So, the x-ray diffraction pattern of a material indicates only crystalline structure. Samples of materials of identical chemical composition can be expected to have similar diffraction patterns, but not necessarily identical ones.

FTIR Analysis

The FTIR indicated that the samples were chemically identical.

DSC Compatibility

A first step in evaluating a new material is to determine its thermal stability at propellant processing temperatures, both as a neat material and in combination with routine solid rocket propellant ingredients. The results of these studies are shown in Table 6.

<table>
<tr><th>TEST</th><th>RESULTS</th><th>ANALYSIS</th></tr>
<tr><td rowspan="2">Differential Scanning Calorimetry (DSC)</td><td>Standard (200μ) RDX<br>Endotherm - 180°C<br>- 190°C<br>- 215°C<br>Exotherm - 185°C<br>- 200°C<br>- 220°C</td><td rowspan="2">The number of thermal changes in the standard RDX could be attributed to impurities (e.g. HMX).</td></tr>
<tr><td>I-RDX (200μ)<br>Endotherm - 200°C<br>Exotherm - 210°C</td></tr>
<tr><td>DSC Compatibility of I-RDX in a Typical Minimum Signature Formulation</td><td>Exotherm at 160°C</td><td>Determined as safe to process under normal procedures.</td></tr>
</table>

Table 6 - DSC Compatibility

Particle Size Analysis

Using an Horiba laser light scattering particle size analyzer, the as received I-RDX was evaluated to determine average particle size and distribution. The results are shown in Table 7.

| TEST | RESULTS | ANALYSIS |
|---|---|---|
| Particle Size Analysis (PSA) | Mean Diameter - 207µ<br>Median Diameter - 201µ<br>Standard Deviation - 111.9 µ | Relatively tight distribution around 200µ, although a long shoulder of particle sizes as low as 8µ existed at a small frequency percent with respect to the major fraction. This accounts for the substantial standard deviation. |

Table 7 - Particle Size Analysis

Sensitivity Testing

To ensure safe handling of new materials, small-scale propellant mixes (35 grams) are made and tested for friction and impact sensitivity. A side-by-side comparison of both standard RDX and I-RDX was conducted. The results are tabulated in Table 8.

| TEST | RESULTS | ANALYSIS |
|---|---|---|
| Propellant Sensitivity (uncured propellant) | **Standard (200µ) RDX**<br>Impact<br>(negative @ 110 kg·cm)<br>Friction<br>(negative @ 500 psi)<br>**I-RDX (200µ)**<br>Impact<br>(negative @ 120 kg·cm)<br>Friction<br>(negative @ 500 psi) | Both the standard RDX and the I-RDX yielded essentially identical results. |

| | | |
|---|---|---|
| Propellant Sensitivity (cured propellant) | **Standard (200µ) RDX**<br>Impact<br>(negative @ 90 kg·cm)<br>Friction<br>(negative @ 500 psi) | Both the standard RDX and the I-RDX yielded essentially identical results. |
| | **I-RDX (200µ)**<br>Impact<br>(negative @ 80 kg·cm)<br>Friction<br>(negative @ 500 psi) | |

Table 8 - Friction and Impact Sensitivity

Ballistic Evaluation

Propellant formulations containing RDX and I-RDX were made in a one-pint mixer (300 grams), cured, and submitted for an initial ballistic evaluation using a strand burner. Each formulation was evaluated over a pressure range of 500 to 2000 psi.

| TEST | RESULTS | ANALYSIS |
|---|---|---|
| Ballistic Analysis (Strand Burning Rate) in a Typical Minimum Signature Propellant Formulation | **Standard (200µ) RDX**<br>$R_b$@1000 = 0.37 in / sec<br>Pressure Exponent (n) = 0.84 | Both the standard RDX and the I-RDX yielded essentially identical results. |
| | **I-RDX (200µ)**<br>$R_b$@1000 = 0.38 in / sec<br>Pressure Exponent (n) = 0.82 | |

Table 9 - Strand Ballistic Evaluation

Plots of the strand burning rate data for the I-RDX based formulation are shown below in Figure 1 and the comparative data for the standard RDX in Figure 2.

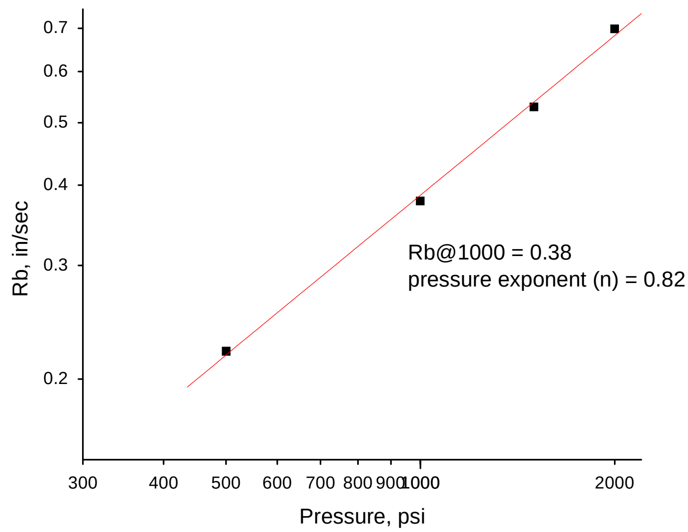


Figure 1 - Strand Burning Rate Data (I-RDX)

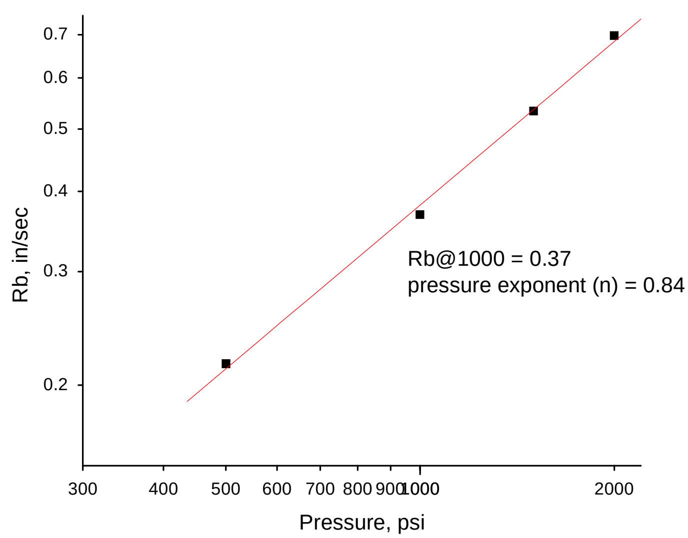


Figure 2 - Strand Burning Rate Data (Standard RDX)

# CONCLUSIONS

The preliminary evaluation of standard Holston RDX versus SNPE's I-RDX indicated that both materials are essentially identical in chemical composition, allowing for the amount of HMX in the standard RDX. Chemical compatibility of both materials was established in combination with routine minimum signature solid rocket propellant ingredients. Cured and uncured friction and impact sensitivity indicated no difference in the sensitivities of the materials. We anticipate having card gap comparisons of the formulations completed by the end of the calendar year. Ballistically, both RDX and I-RDX yielded the same results.