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Energetic Residues from the Detonation of IMX-104 Insensitive Munitions

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Abstract: The development of insensitive munitions by NATO countries is an ongoing effort. Less-sensitive ingredients in both explosives and propellants will ensure the protection of deployed troops against an unwanted reaction to an external stimulus on the munitions stockpile. In the US Army, current efforts are directed towards the development of melt cast insensitive explosive formulations. Various formulations, mainly based on DNAN and NTO, have been developed and are now being fielded. Our research goal is to measure the deposition rate of energetics compounds from various insensitive munitions detonation scenarios. Our hypothesis is that the relative insensitiveness of these formulations leads to slightly higher deposition rates

than conventional explosive formulations. This paper describes detonation residues research on mortar rounds containing IMX-104 explosive. Analyses indicate that high-order detonation residues are slightly greater for this formulation than for conventional munitions. However, blow-in-place detonations (BIPs) resulted in much higher residues deposition, indicating that a larger donor charge is required for efficient detonation. The highly soluble compound NTO was particularly problematic, with BIP deposition approaching 95% of the original load. Toxicological studies of NTO are not finalized, leaving considerable uncertainty regarding the feasibility of approving these rounds for distribution

Keywords: Insensitive munitions • Detonation residues • IMX-104 • NTO • Environment

1 Introduction

The storage and transportation of live munitions are hazardous operations. Since 1926, the US military has experienced 17 major munitions-related accidents resulting in over 600 deaths and more than US \$4B in equipment losses [1]. Although most modern munitions are relatively insensitive compared to those used in the past, they continue to detonate unintentionally. With the advent of large-scale asymmetrical warfare in Iraq and Afghanistan, US ground forces are experiencing significant losses in the field from unintentional detonation of transported munitions. For this reason, a new emphasis has been placed on the deployment on munitions containing insensitive high explosives.

The first artillery insensitive munition (IM) to be approved for use in training and combat in the US was the 60 mm mortar cartridge containing Picatinny Arsenal Explosive 21 (PAX-21). This insensitive high explosive formulation contains the traditional explosive compound cyclotrimethylenetrinitramine (RDX), the insensitive high explosive (IHE) 2,4-dinitroanisole (DNAN), and the oxidizing salt ammonium perchlorate (AP) that, in small crystal sizes ($<15~\mu m$), reacts explosively [2,3]. Environmental field-testing of this munition following deployment demonstrated that large quantities of perchlorate residues would result from highorder and blow-in-place (BIP) detonations of the rounds. For a high order detonation, an average of 14 g of perchlorate

rate remained following each detonation, enough to contaminate 7 million liters of groundwater above current US drinking water standards (2.0 ppb) [4]. For a BIP operation, the perchlorate residue mass averaged 35 g, enough to contaminate over 17 million liters of water above the drinking water limit [5]. Production of the rounds was cancelled and limitations were imposed on the use of the remaining stock of munitions, which was reclassified as "not for training." Costs associated with these actions were high, estimated at about US \$300M based on prior munitions acquisitions. However, the environmental liabilities avoided

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Table 1. Munitions used for IM detonation residues tests.

| ltem | DODIC ^{a)} | Designation | Quantity | Mass and type of energetics | Percent in formulation ^{b,c,d)} |
|------------------------------|---------------------|-------------|----------|-----------------------------|--|
| Mortar cartridge – 60 mm | - | M720 | 14 | 338.7 g IMX-104 | 32% DNAN, 15% RDX, 53% NTO |
| Mortar cartridge – 81 mm | CA16 | M821 | 14 | 807.3 g IMX-104 | 32% DNAN, 15% RDX, 53% NTO |
| Demolition charge | M023 | M112 | 60 | 567 g C4 | 91 % RDX |
| Blasting cap | M130 | M6 | 55 | 0.94 g RDX | RDX |
| Fuze simulator ^{e)} | - | _ | 20 | 12 g C4 | 91 % RDX |

a) DODIC: US Department of Defense Identification Code; b) Values shown are mid-range for the formulations. Range is $\pm 2\%$ except for RDX in C4 ($\pm 1\%$); c) DNAN: 2,4-Dinitoranisole, RDX: cyclotrimethylenetrinitramine, NTO: 3-nitro-1,2,4-triazole-5-one; d) HMX (Octogen) present in quantities of up to 9% w/w of RDX as a manufacturing byproduct; e) Supplied by CRREL. C4 Booster charge added at test site.

by this quick action could have totaled in the billions of dollars US [1].

The US Army is currently focused on insensitive mortar munitions containing the formulation Insensitive Munitions Explosive 104 (IMX-104). This formulation does not contain the problematic component AP. The three major explosive compounds are RDX, DNAN, and 3-nitro-1,2,4-triazole-5-one (NTO) [6]. A series of tests to determine the energetic residues from the field detonation of 60 mm and 81 mm mortar projectiles containing IMX-104 was executed under the US Department of Defense Strategic Environmental Research and Development Program Environmental Restoration - Insensitive Munitions Program with support from the US Army Ammunition Research Development and Engineering Center (ARDEC) [7]. The objective was to determine the consumption efficiencies of the three explosive compounds during various detonation scenarios prior to fielding the rounds for training and combat. This post-detonation deposition data compliments the air emissions data collected during life cycle environmental assessments of munitions conducted in the US. Our hypothesis, based on the insensitive nature of the IHE formulation, was that these rounds would not detonate as efficiently as conventional rounds containing Composition B (Comp-B), which contain RDX and trinitrotoluene (TNT), but that they would still perform well. This paper presents the results for two of these tests: High-order detonations and BIP detonations of both the 60 mm and 81 mm mortar projectiles.

2 Experimental Section

2.1 Field Tests

A series of four detonation tests were conducted on an ice-covered impact range located in Alaska. Participants in the tests included the US Army Cold Regions Research and Engineering Laboratory (CRREL); Envirostat, Inc.; ARDEC; Defence Research and Development Canada—Valcartier (DRDC); and the 716th Explosive Ordnance Disposal unit, US Army Alaska. Munitions used for the tests were provided by ARDEC and drawn from a manufacturing test run at the General Dynamics – Ordnance and Tactical Systems ammunition plant in Le Gardeur, QC, Canada, through DRDC. Tests utilized both 60-mm and 81-mm mortar bodies con-

taining IMX-104 IHE with PBXW-14 supplementary charges. A fuze simulator containing C4 explosive was used in place of the normally issued fuze (Table 1). Note that mass values for the formulations are mid-range values from the material safety data sheet for IMX-104.

Tests were grouped by caliber and operation. High-order detonation tests utilizing a fuze simulator to initiate detonation were conducted on different days with seven 60 mm and seven 81 mm mortar projectiles mounted vertically on 10×10 cm aluminum plates (Figure 1). The assemblies were located approximately 50 m from each other on the clean snow surface covering the ice. Hardened 30 cm thick snow blocks were placed between the base plates and the snow surface to elevate the rounds above the snow surface. The BIP tests were conducted in a similar area. The rounds were placed horizontally on 20 cm thick ice blocks located 50 m apart on the snow surface. All rounds contained fuze simulators with booster cup loads of 12 g of Composition 4 (C4) explosive (91% RDX). The booster loads were based on prior testing that demonstrated a 9-10 g load resulted in a low-order detonation. Climatic conditions were near ideal with mostly overcast skies, winds from the south at $0-3 \text{ m s}^{-1}$, and no precipitation. Daily high temperatures hovered around -2 °C. All rounds in each test were detonated simultaneous except in one



Figure 1. High-order detonation test set-up.

case, where a misfire of one round out of seven occurred. The blasting cap failed for that round. The initiator was replaced and that round was detonated within a half hour and before the other six plumes were sampled.

Following completion of the detonations, sampling of the residues occurred. Sampling followed the protocol established by Walsh et al. for energetics residues on a snow surface [8]. This method has been used successfully for characterizing live-fire munitions residues at firing points (propellants) and detonation points (explosives) [9,10]. Each detonation was divided into different functional areas for sampling that we call sampling units. Residues plumes, the primary sampling units, were visually demarcated while walking the edge of the dark surface residues. A technician with a geographical positioning system followed the path in the snow to record the plume perimeter. These data were used later to estimate the plume areas, which are used to calculate the mass of residues within the plume. Triplicate samples were taken independently from within each plume using the Multi-Incremental® (MI; Multi-Incremental[®] is a registered trademark of Envirostat, Inc.) systematic-random sampling method. Approximately 100 increments were collected using a 10×10×2.5 cm deep polytetraflouroethylene-lined flat-bottomed scoop. Samples were collected and stored in lab-grade clean 3-mil polyethylene (PE) bags that were labeled prior to being tagged and recorded in the field. The sample bags were then placed in overbags to prevent cross contamination. The samples were taken to a lab on post, where they were rebagged to prevent cross-contamination and melted for processing.

Field quality assurance was conducted during each test series. Prior to testing, a multi-increment background sample was taken from the test areas. This sample would show us if there was any pre-existing contamination from the compounds of concern (COCs): RDX, DNAN, and NTO. As stated above, all detonation plumes were sampled in triplicate. For each plume, an MI sample was taken from an annulus 3 m deep from the edge of the plume (outside the plume or OTP sample). On one plume for each series of tests, this 0–3 m OTP was sampled in triplicate. A second OTP, from 3 to 6 m from the edge of the plume, was taken from randomly chosen plumes. The objective of these samples was to determine if the plume was correctly demarcated.

For one plume of each series, a fourth multi-increment sample was obtained. First, snow from a $20\times20\times2.5$ cm deep area was removed from the plume surface. Then, a sample increment was taken at a depth of 2.5 to 5 cm deep from the cleared area with the $10\times10\times2.5$ cm scoop. The objective of this sample was to determine if the 2.5 cm sampling depth of the MI samples was sufficient to capture most (>98%) of the detonation residues. This data, along with the other field QA data, is used to determine the robustness of the sampling method as well as the data.

2.2 Sample Processing and Lab QA

In the field lab, each sample was processed to prepare it for shipment. After rebagging in the lab, the samples were placed in clean PE bins for melting. Melting generally occurred overnight, with some snow still in the melted samples. The samples were vacuum filtered to separate the solid fraction from the aqueous fraction. Filters were airdried away from light in clean 125 mL amber glass jars before storage in a refrigerator (T=4 °C). The aqueous portion was measured for volume, shaken, and two to four aliquots decanted into 500 mL amber glass bottles for subsequent analysis for RDX and DNAN. In addition, a 120 mL aliquot was decanted for analysis for NTO. Solid-phase extractions (SPEs) were then performed on one to three of the 500 mL aliquots for future analyses of RDX and DNAN. The 5 mL aliquot was concentrated at 100:1 in acetonitrile (AcN) during the extraction of the analytes from the Waters Porapak RDX SPE cartridge. Sample preparation followed EPA SW-846 Method 8330B [11]. One 500 mL bottle of the aqueous fraction was stored in the refrigerator as was 3.5 mL of the 5 mL SPE concentrate. The remaining items were shipped to the analytical laboratory at CRREL in Hanover, NH, for final processing and analysis.

Laboratory QA was conducted at the field lab. Three triplicate aqueous fractions were taken from randomly selected samples. These samples were processed and analyzed the same as for any aqueous sample. The objective of this procedure was to evaluate the processing and analysis of the aqueous portion of the samples for reproducibility. Four filtered water blanks (Barnstead D4641 E-Pure 80 M Ω), one for each series of tests, were run through the complete processing and analysis procedures to check for cross contamination in the field lab from the cleaning of the glassware. An SPE blank, 500 mL of E-Pure water run through the Waters Porapak RDX SPE cartridge, was processed and analyzed to determine if any analyte was contained in the filtered water used in the lab. A series of five laboratory control samples, consisting of a known concentration spiked solution added to the E-Pure water, was run to determine the recovery of RDX, HMX, and DNAN using SPE. Matrix spiked samples, at 0.0020 mg/L of RDX, HMX, and DNAN, were made with replicate 500-mL aqueous aliquots from one of the samples to determine if the compounds were being properly retained and recovered through the processing and analytical steps. Finally, a 3 mg piece of IMX-104 obtained from BAE Systems (Kingsport, TN) was dissolved in 20 mL of AcN to verify that the estimates of RDX, DNAN and NTO were within specifications based on the analytical methods described below.

2.3 Analytical Methods

Analyses for DNAN, HMX, and RDX in the residues were conducted in the analytical chemistry lab at CRREL. Filters containing the solids fraction were extracted with AcN on

a shaker Table t = 18 h). Aqueous and solids sample extracts were analyzed using high-pressure liquid chromatography (HPLC) following EPA SW846 Method 8330B [11]. The AcN extracts were mixed 1/3 v/v with reagent-grade water and filtered with a Millex-FH (Millipore, PTFE, 0.45 μm) filter unit prior to analysis. Determinations were made on a modular system from ThermoScientific composed of a Spectra-System Model P4000 pump, a Spectra-System UV2000 dual wavelength UV/VS absorbance detector (cell path 1 cm) set at 230 for HMX and RDX and 295 nm for DNAN, and a SpectraSYSTEM AS300 autosampler. Samples were introduced with a 100 µL sample loop. Separations were achieved on a 15 cm×3.9 mm (4 μm) NovaPak C8 column (Waters Chromatography Division, Milford, Massachusetts) at 28 °C and eluted with 1.4 mL min⁻¹ of 15:85 (v/v) 2-propanol/water. The detection limit in AcN extracts was 0.02 mg L⁻¹ for the three compounds, which is equivalent to 0.0002 mg L⁻¹ in the aqueous samples that were pre-concentrated by a factor of 100 using SPE.

NTO was analyzed following the Standard Operating Procedure DLS810, which was obtained from M. Hable at the U.S. Army Public Health Command and is based on the methods of LeCampion [12,13]. Samples were injected using a matrix of 1:1 water: AcN. For the aqueous (melted snow) samples, 1.00 mL of the aqueous sample was mixed with 1.00 mL AcN. For the soot extracts, 1.00 mL of the AcN extract was mixed with 1.00 mL of reagent grade water. Determinations were made using a modular system similar to above except that the column was a 15 cm \times 4.6 mm (5 μ m) Hypercarb from ThermoScientific eluted with 1.5 mL min $^{-1}$ of 25:75 (v/v) water/AcN with 0.1% trifluoroacetic acid (Fisher HB9813-4) at 28 °C. The UV detector was set at 315 nm. The detection limit for NTO was 0.005 mg L $^{-1}$ for both the aqueous samples and the AcN extracts.

Confirmation of NTO was done using an HPLC/MS with electrospray ionization in the negative ion mode, single ion monitoring, at m/z = 129. A 100 mm \times 4.6 mm (2.5 μ m) XBridgeTM BEH Amide (Waters 8 Chromatography Division, Milford, Massachusetts) was eluted with 0.3 mL min⁻¹ of 75/25 (v/v) AcN/water.

Calibration standards for RDX and HMX were prepared from commercial solutions obtained from Restek Corpora-

tion (Bellefonte, PA). Calibration standards for DNAN and NTO were from solid reference materials from BAE Systems (Kingsport, TN). Replicate stock solutions at $1000~\text{mg}\,\text{L}^{-1}$ were prepared in AcN. The calibrated range for the UV detector was from 0.02 to $10~\text{mg}\,\text{L}^{-1}$ for RDX, HMX, and DNAN and 0.005 to $10~\text{mg}\,\text{L}^{-1}$ for NTO. The pH of the NTO samples were also checked using Micro Essential Laboratory, Inc., Hydrion® litmus paper.

3 Results

Samples were analyzed for RDX, HMX, DNAN, and NTO. Pretest background samples did not contain detectable levels of any of the target analytes. The pH of the filtrate was between 3 and 4. Results will be presented according to the type of detonation.

3.1 High-Order Detonations

Results of the high-order detonation residues analyses extrapolated over their respective sampling units and totaled for each test are presented in Table 2. Where appropriate, the plume, OTP, and subsurface results are combined to derive the mass estimate for the plumes. The overall mean detonation efficiency (consumption) of the DNAN and RDX/HMX for the two types of rounds was > 99.994%, indicative of high-order detonations (99.99% consumption of compounds). For the NTO, however, the mean detonation efficiencies were 99.6% for the 81 mm rounds and 99.8% for the 60 mm rounds. The result is gram quantities (≈ 2 g) of NTO on average per detonation. The mean of the residue masses for the DNAN and RDX/HMX are much lower than for NTO.

The average residues plume sizes were $250 \, \text{m}^2$ and $350 \, \text{m}^2$ for the 60 mm and 81 mm detonations, respectively. The OTPs averaged $200 \, \text{m}^2$ for the 60 mm detonations, 80% of the plume areas. For the 81 mm detonation, the OTPs averaged $230 \, \text{m}^2$, 66% of the original plume areas. OTP residues averaged < 0.001% of the total residues mass for the 60 mm detonations. Difficulty demarcating the plumes for the 81-mm tests resulted in the OTPs containing

Table 2. Mean high-order detonation IM deposition results.

| Munition | Plume [m²] | OTP [m²] | Analyte ^{a)} | Est. total mass ^{b)} [mg] | Range [mg] | % of Original analyte mass ^{c)} |
|-----------|---------------|-------------|-----------------------|---------------------------------------|---------------|--|
| 60 mm 250 | 250 | 200 | DNAN | 5.3 | 3–6 | 0.005% |
| | | | RDX & HMX | 4.5 | 1–18 | 0.006% |
| | | | NTO | 2,200 | 1,300-2,800 | 1.2% |
| 81 mm | 350 | 230 | DNAN | 27 | 2.1-71 | 0.001% |
| | | RDX & HMX | 16 | 0.6-47 | 0.001% | |
| | | | NTO | 1,900 | 900–2,800 | 0.4% |

a) RDX includes some HMX as well (>9% of original mass); b) Mean of the combined plume, OTP, and subsurface estimated masses; c) For 60 mm tests: 107 g DNAN, 75 g RDX & HMX, and 179 g NTO ($\pm2\%$); For 81-mm tests: 256 g DNAN, 147 g RDX & HMX, and 428 g NTO ($\pm2\%$).

Table 3. Mean BIP detonation IM deposition results.

| Munition | Plume [m²] | OTP [m²] | Analyte ^{a)} | Est. total mass ^{b)} [mg] | Range [mg] | % of Original analyte mass ^{c)} |
|-------------------------|---------------|-------------|-----------------------|---------------------------------------|---------------|--|
| 60 mm 580 | 300 | DNAN | 20.000 | 2,700-34,000 | 19% | |
| | | RDX & HMX | 8,300 | 1,000-17,000 | 1.4% | |
| | | NTO | 89,000 | 54,000-124,000 | 50% | |
| 81 mm ^{d)} 610 | 320 | DNAN | 29,000 | 20,000-49,000 | 11 % | |
| | | RDX & HMX | 11,000 | 7,100–19,000 | 1.6% | |
| | | NTO | 190,000 | 160,000–250,000 | 45 % | |

a) RDX includes some HMX as well (< 9% of original mass); b) Mean of the combined plume, OTP, and subsurface estimated masses; c) For 60 mm tests: 107 g DNAN, 75 g RDX & HMX, and 179 g NTO (2%); For 81 mm tests: 256 g DNAN, 147 g RDX & HMX, and 428 g NTO (\pm 2%); d) Excludes Plume 4, which almost failed to detonate.

an average of 16% of the total detonation residues. This is higher than the 3% we normally consider a goal. Two 3–6 m OTPs were collected for the 60 mm detonations. Neither contained any residues.

Subsurface residues were quite low for both tests, with no residues detected for the 60 mm high-order detonation and 0.06% of the total plume residues detected for the 81 mm detonation. The goal for subsurface residues recovery is < 1.5%.

3.2 BIP Detonations

Results of the blow-in-place detonation residues analyses extrapolated over their respective sampling units and totaled for each test are presented in Table 3. Where appropriate, the plume, OTP, and subsurface results are combined to derive the mass estimate for the plumes. One 81 mm round nearly failed to detonate (based on NTO residues) and is not included in the overall data. The overall mean detonation efficiency (consumption) of the DNAN and RDX/HMX for two types of rounds averaged 96%. For the NTO, the consumption efficiencies averaged only 50% for the 60 mm detonations and 55% for the 81 mm detonations. For the 81 mm round that nearly failed to detonate, 77% of the DNAN and RDX and >98% of the NTO was estimated to have been deposited on the snow surface.

The average residues plume sizes were $580 \, \text{m}^2$ and $610 \, \text{m}^2$ for the $60 \, \text{mm}$ and $81 \, \text{mm}$ detonations, respectively. The OTPs averaged $300 \, \text{m}^2$ for the $60 \, \text{mm}$ detonations, 52% of the plume areas. For the $81 \, \text{mm}$ detonations, the OTPs averaged $320 \, \text{m}^2$, 52% of the original plume areas. OTP residues averaged 0.6% of the total residues mass for the $60 \, \text{mm}$ detonations and 1% for the $81 \, \text{mm}$ detonations. One $3-6 \, \text{m}$ OTP each was collected for the $60 \, \text{mm}$ and $81 \, \text{mm}$ detonations. They contained < 0.3% of the total residues for the $60 \, \text{mm}$ detonation and < 0.7% of the total residues for the $81 \, \text{mm}$ detonation. Subsurface residues were low for both tests with 0.17% of the total plume residues estimated for the $81 \, \text{mm}$ BIP and 0.47% of the total plume residues estimated for the $81 \, \text{mm}$ BIP subsurface samples.

3.3 Quality Assurance

Field quality assurance results are covered for the most part in the previous two subsections. Analyses of the two background samples taken prior to testing indicated no detectable levels of the COCs. OTPs averaged 4.3% of overall residues (0.6% without the 81 mm high-order plumes for which we had demarcation difficulties) and subsurface samples averaged 0.2% of the overall residues, indicating that most of the plumes were correctly demarcated and those that were not were compensated for by the OTP samples. In the lab, the SPE triplicates had an average relative standard deviation of < 0.3 %, indicating reproducibility for the SPE concentration process and lab analyses. The SPE blank had no detectable COCs. The mean recovery values for the SPE control samples were 100% for HMX and 101% for RDX and DNAN, again indicating successful processing and analysis techniques for these tests. Matrix spikes on a sample aqueous aliquot showed no influence on the original concentrations of the COCs due to the spiked solution. The SPE filtration blanks indicated very low (at detection limit) concentrations of DNAN for three of the four aqueous fractions and no detectable COCs for the filter extracts, indicating no carryover from the glassware cleaning process. All QA indicates the data are robust.

4 Discussion

The findings from this research on IMX-104 munitions are similar to those derived from testing the insensitive high explosive PAX-21 [4]. Like the PAX-21 detonations, the 60-mm and 81-mm mortar rounds filled with IMX-104 demonstrated lower detonation efficiency than for conventional munitions when measured by the consumption of the explosive compounds in the insensitive high explosive filler. Also like the PAX-21 rounds there is a single problematic IHE compound. For PAX-21, it was AP and for IMX-104, it is the NTO. We suspect the reason for the low efficiency of these two compounds lies with their particle sizes. The NTO particles recovered from post-detonation material were large (>50 μ m). Smaller particles are more efficient in detonation but are more difficult to initiate. Using small

Table 4. Comparison of three 60 mm mortar munitions.

| Explosive filler High order detonations | Plume [m²] | OTP [m²] | Analyte ^{a)} | Estimated total mass ^{b)} [mg] | % of Original analyte mass ^{c)} |
|--|---------------|-------------|-----------------------|--|--|
| Comp-B [10] | 214 | 193 | RDX + HMX | 0.073 | 0.00003% |
| PAX-21 [4] | 330 | 250 | RDX + HMX | 7.1 | 0.007% |
| | | | DNAN | 9.2 | 0.006% |
| | | | AP | 14,000 | 15% |
| IMX-104 | 250 | 200 | RDX + HMX | 4.5 | 0.006% |
| | | | DNAN | 5.3 | 0.005 % |
| | | | NTO | 2,200 | 1.2% |
| BIP detonations | | | | | |
| Comp-B [10] | 500 | 230 | RDX + HMX | 200 | 0.027% |
| PAX-21 [4,5] | 330 | 250 | RDX + HMX | 880 | 0.13% |
| | | | DNAN | 740 | 0.61% |
| | | | AP | 35,000 | 38% |
| IMX-104 | 580 | 300 | RDX + HMX | 20,000 | 1.4% |
| | | | DNAN | 8,300 | 19% |
| | | | NTO | 89,000 | 50% |

a) RDX includes some HMX as well (<9% of original mass); b) Mean of the combined plume, OTP, and subsurface estimated masses; c) 107 g DNAN, 75 g RDX & HMX (plus 520 g RDX/HMX for BIP donor block), and 179 g NTO. Based on mid-range value of formulation specifications.

particles in melt cast formulations present a challenge as they will increase the viscosity of the mixture, making casting without voids or other flaws difficult. The tradeoff may thus be between formulation efficiency and manufacturability [14].

A comparison between the IM formulations and conventional high explosives is illuminating. Detonation efficiencies for the two 60 mm IM and a conventional 60 mm round are shown in Table 4. As can be seen, the efficiencies of the two IM formulations and the Comp-B rounds differ by two orders of magnitude. Interestingly, the RDX and HMX efficiencies of the two IM formulations are quite close. Although the mass of RDX and HMX is higher for the IM formulations than for Comp B, the mass and concentrations that will be found on an impact area will be quite small and should not be of concern from a range contamination standpoint [10]. However, the results for the AP and NTO are a matter for concern. Both these compounds are highly soluble in water and deposited in gram quantities during high-order detonations. AP is very toxic and does not readily break down in the environment. NTO toxicity studies are on-going but incomplete. The acidity of the NTO (pH 3-4) combined with its rapid dissolution from the detonation residues may lead to the mobilization of metals on an impact area.

The 81 mm high-order detonation residues rounds follow a similar deposition trend. There are no data for 81 mm detonations containing PAX-21 as they were never manufactured in that configuration. However, Table 5 compares 81 mm rounds containing Comp-B and IMX-104. As with the 60 mm rounds, there is a significant difference between the conventional munition and the IMX-104 munition stemming from the inefficiency of the IHE compound NTO. The mass deposition per detonation for the DNAN and RDX/

HMX are quite low and are not critical from a range sustainment perspective.

Where the results differ the most between the energetic compounds is for the BIP detonations. For both rounds, the IHE compound residues are not only high; their low detonation efficiencies seem to have affected the efficiency of the RDX and HMX. Consumption of NTO is especially inefficient, with about half of the mass not detonating during the BIP operations. The results in Table 5 do not include one of the seven BIP detonations, as that round almost did not function. That detonation resulted in an estimated 75 g of RDX and HMX (11.2%), 135 g of DNAN (47%), and over 420 g of NTO (99%) in residues for an overall efficiency of



Figure 2. 81 mm BIP residues on a 90 mm Ø filter. Bright particles are IMX-104.

Table 5. Comparison of two 81 mm mortar munitions.

| Explosive filler High order detonations | Plume [m²] | OTP [m²] | Analyte ^{a)} | Estimated total mass ^{b)} [mg] | % of Original analyte mass ^{c)} |
|---|---------------|-------------|------------------------|--|--|
| Comp-B ^{d)} [10] IMX-104 | 230 350 | - 230 | RDX + HMX RDX + HMX | 8.5 16 | 0.00014% 0.001% |
| | 330 | 230 | DNAN NTO | 27 1.900 | 0.001 % 0.001 % 0.4 % |
| BIP detonations | 020 | 410 | | <i>V</i> · · · | |
| Comp-B [10] IMX-104 ^{e)} | 820 590 | 410 310 | RDX + HMX RDX + HMX | 150 11,000 | 0.0014 <i>%</i> 1.6 <i>%</i> |
| | | - 1 - | DNAN | 29,000 | 11 % |
| | | | NTO | 193,000 | 45 % |

a) RDX includes some HMX as well (<9% of original mass); b) Mean of the combined plume, OTP, and subsurface estimated masses; c) 147 g RDX & HMX (plus 520 g RDX/HMX for BIP donor block), 256 g DNAN, and 428 g NTO. Based on mid-range value of formulation specifications; d) 14 rounds in various multiple detonation groupings. No OTPs; e) Excludes Plume 4, which almost failed to detonate.

53%. The plume area for this round was 28% smaller than the mean of the other six plumes and 12% smaller than the next smallest plume, indicating an incomplete detonation. The snow was discolored yellow in the plume from NTO leaching out of the residues, and particles of IMX-104 were actually recovered on the filters of the processed samples (Figure 2). Two other BIP detonations had NTO consumptions of less than 50%, but the other explosive compounds detonated at 93% or higher. This indicates that one block of C4 is not sufficient to reliably dispose of an 81 mm round containing IMX-104. Additional BIP tests with this round are necessary to prove out this hypothesis. We are currently planning to conduct such trials in the future.

Although initial studies for NTO indicate it is "practically nontoxic," the toxicological data are not complete [15, 16]. It is worth noting at this point the preliminary results of the ongoing toxicology studies. Research being conducted by Sunahara's group at the National Research Council Canada indicates a relatively high phytotoxicity of NTO towards selected plant species. Spiked soil studies conducted with earthworms demonstrate an avoidance behavior by the earthworms, which may be indicative of toxicity (Sunahara, personal communication). Toxicology studies in the US have been delayed because of the budget sequestration in effect at the time of this manuscript (Johnson, personal communication). Preliminary results indicate there are lowlevel health effects on rats but the connection with human health is not firmly established. A further discussion of this critical topic is best left to the experts in the field.

5 Conclusion

The detonation performance of the IMX-104 rounds with respect to the reaction of the explosive components of the IHE filler is lower than with the current common explosive filler, Comp-B. The NTO component was slightly inefficient during high-order detonations, leaving 2 g of residues per detonation for both rounds. Significant problems occurred

during BIP operations, with very low consumption of NTO for both rounds, affecting the consumption of the other explosive compounds. It is likely that two C4 donor blocks will be required to efficiently dispose of an 81 mm round during a BIP operation. As with the PAX-21 insensitive high explosive filler field tested previously, the problem occurred mostly with a single explosive component that occurs as large crystals in the IHE matrix, in this case NTO.

Toxicological studies need to be finalized before a determination can be made if this and related rounds containing NTO should be fielded. Testing of alternative blow in place techniques combined with systematic deposition rate trials must be conducted prior to the fielding of future insensitive munitions to avoid adverse environmental impacts that could in turn lead to a loss of range sustainability. Once again, these tests show the importance of field-testing munitions prior to their acceptance and release.

References

- C. Patel, Common Low-Cost IM Explosive Program, SERDP and ESTCP Partners in Technology Technical Symposium and Workshop, Washington, DC, USA, 28 November–1 December, 2011.
- [2] A. R. Clairmont Jr., I. Jaffe, D. Price, The Detonation Behavior of Ammonium Perchlorate as a Function of Charge Density and Diameter, NOLTR 67–71, 1967, US Naval Ordnance Laboratory, White Oak, MD, USA.
- [3] D. Price, A. R. Clairmont, I. Jaffe, Particle Size Effect on Explosive Behavior of Ammonium Perchlorate, NOLTT 67–112, 1967, US Naval Ordnance Laboratory, White Oak, MD, USA.
- [4] M. R. Walsh, M. E. Walsh, S. Taylor, C. A. Ramsey, D. B. Ringelberg, S. Thiboutot, G. Ampleman, E. Diaz, Characterization of PAX-21 Insensitive Munition Detonation Residues, *Propellants Explos. Pyrotech.* 2013, 38, 399–409.
- [5] M. R. Walsh, M. E. Walsh, C. A. Ramsey, S. Brochu, S. Thiboutot, G. Ampleman, Perchlorate Contamination from the Detonation of Insensitive High-Explosive Rounds, J. Hazard. Mater. 2013, 262, 228–233.
- [6] V. Fung, M. Ervin, B. Alexander, C. Patel, P. Samuels, Development and Manufacture of an Insensitive Composition B Replacement Explosive IMX-104 for Mortar Applications, *Insensi-*

- tive Munitions and Energetic Materials Technology Symposium, Munich, Germany, 11–13 October, **2010**.
- [7] S. Thiboutot, M. R. Walsh, Characterization of Munitions from the Detonation of Insensitive Munitions, SERDP Project ER-2219, 2012, US Department of Defense Strategic Environmental Research and Development Program, Environmental Restoration Program, A. Leeson Program Manager (Accessed: May 2013): http://www.serdp-estcp.org/Program-Areas/Environmental-Restoration/Contaminants-on-Ranges/Characterizing-Fate-and-Transport/ER-2219.
- [8] M. R. Walsh, M. E. Walsh, C. A. Ramsey, Measuring Energetic Contamination Deposition Rates on Snow, Water Air Soil Pollut. 2012, 223, 3689–3699.
- [9] M. R. Walsh, M. E. Walsh, G. Ampleman, S. Thiboutot, S. Brochu, T. F. Jenkins, Munitions Propellants Residue Deposition Rates on Military Training Ranges, *Propellants Explos. Pyrotech.* 2012, 37, 393–406.
- [10] M. R. Walsh, M. E. Walsh, I. Poulin, S. Taylor, T. A. Douglas, Energetic Residues from the Detonation of Common US Ordnance, Int. J. Energ. Mater. Chem. Prop. 2011, 10, 169–186.
- [11] USEPA, Nitroaromatics and Nitramines by High-Pressure Liquid Chromatography, EPA SW846 Method 8330B, 2006, US Department of Environmental Protection, Washington, DC, USA (Accessed May 2013): http://www.epa.gov/osw/hazard/testmethods/pdfs/8330b.pdf.
- [12] M. Hable, Analysis of the Components of IMX-101 by Liquid Chromatography and Gas Chromatography, 2013, U.S. Army Public Health Command, Personal Communication.

- [13] L. Le Campion, M. T. Adeline, J. Ouazzani, Separation of NTO Related 1,2,4-Trizaole-3-One Derivatives by a High Performance Liquid Chromatography and Capillary Electrophoresis, Propellants Explos. Pyrotech. 1997, 22, 233–237.
- [14] R. Vijayalakshmi, S. Radhakrishnan, P. S. Rajendra, G. M. Giri, S. K. Arun, Particle Size Management Studies of Spherical 1-Nitro-1,2,4-triazol-5-one, *Part. Part. Syst. Charact.* **2011**, *28*, 57–63.
- [15] M. V. Haley, R. G. Kuperman, R. T. Checki, Aquatic Toxicity of 3-Nitro-1,2,4-Triazol-5-One, ECBC-TR-726, 2009, US Army Research, Development, and Engineering Command, Edgewood Chemical Biological Center, Aberdeen Proving Ground, MD, USA.
- [16] G. Reddy, J. Song, P. Kirby, E. M. Lent, L. C. B. Crouse, M. S. Johnson, Genotoxicity Assessment of an Energetic Propellant Compound, 3-Nitro-1,2,4-triazol-5-one (NTO), Mut. Res. Genetic Toxicol. Environ. Mutagen. 2011, 719, 35–40.
- [17] M. W. Smith, M. D. Cliff, NTO-Based Explosive Formulations: A Technology Review, DSTO-TR-0796, 1999, Weapon Systems Division, DSTO Aeronautical and Maritime Research Laboratory, Salisbury, SA, Australia.

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