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Live-Fire Validation of Command-Detonation Residues Testing Using a 60 mm IMX-104 Munition

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Abstract

Command detonation (i.e., static firing) provides a method of testing munitions for their postdetonation residues early in the acquisition process. However, necessary modifications to the firing train and cartridge orientation raise uncertainty whether command detonation accurately represents residue deposition as it occurs during live-fire training. This study collected postdetonation residues from live-fired 60 mm IMX-104 mortar cartridges and then compared estimated energetic-compound deposition rates between live fire and prior command detonations of the same munition. Average live-fire deposition rates of IMX-104 compounds determined from 11 detonations were 3800 mg NTO (3-nitro-1,2,4-triazol-5-one), 34 mg DNAN (2,4-dinitroanisole), 12 mg RDX (1,3,5-Trinitroperhydro-1,3,5-Triazine), and 1.9 mg HMX (1,3,5,7-Tetranitro-1,3,5,7-Tetrazocane) per cartridge. Total live-fire residue deposition (mean ± standard deviation: 3800 ± 900 mg/cartridge) was not significantly different from command detonation using a representative fuze simulator (3800 ± 900 mg/cartridge, $n = 7$, $p = 0.76$) but was significantly different from command detonation using a simplified fuze simulator (2200 ± 500 mg/cartridge, $n = 7$, $p < 0.01$). While the dominant residue compound NTO was broadly similar between live fire and command detonation, the minor residue compounds RDX and DNAN were underestimated during command detonation by a factor of approximately three to seven.

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Preface

This research was conducted for the Strategic Environmental Research and Development Program (SERDP) Environmental Security and Technology Certification Program (ESTCP) under Environmental Restoration Program project number ER20-5018, “Validation of Command Detonation Munitions Residues Testing,” through MIPRs W74RDV00831813 and W74RDV00841816 and Repositions S0491 and S0492. Dr. Herb Nelson was executive director for SERDP-ESTCP, and Dr. Andrea Leeson was deputy director and project monitor.

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1 Introduction

1.1 Background

Live-fire training with munitions is an essential component of warfighter readiness. However, live-fire training releases energetic residues that may accumulate in high concentrations at the soil surface or mobilize off-site. Environmental impacts from energetic residues have resulted in training restrictions on installations in the past (M. R. Walsh et al. 2014). Controlling source loading of energetic residues is fundamental to sustaining training ranges, especially as the military transitions from using conventional munitions to insensitive munitions (IM).

Conventional munitions containing Composition B (Comp B; 2,4,6-trinitrotoluene [TNT] and 1,3,5-trinitroperhydro-1,3,5-triazine [RDX]) and TNT have been found to deposit minute quantities of energetic residue after high-order detonation (Table 1; Hewitt et al. 2005; M. R. Walsh et al. 2011). The majority of conventional munition residues on ranges are derived from occasional low-order detonations and duds that slowly release their energetic filler over months to decades, respectively (Chendorain, Steward, and Packer 2005; M. E. Walsh et al. 2010). However, for IM, command-detonation tests have found significant deposition of energetic residues from high-order detonations (Table 1).* The two current predominant IM filler formulations are IMX-101 (containing the compounds 3-nitro-1,2,4-triazol-5-one [NTO]; 2,4-dinitroanisole [DNAN]; and nitroguanidine [NQ]) and IMX-104 (containing the compounds NTO, DNAN, and RDX). The relatively high water solubility and low soil affinity of NTO and NQ compared to their conventional counterparts raise concerns of their potential mobility into groundwater and surface water (Taylor et al. 2015; Arthur et al. 2018).

Although new munitions are subjected to environmental assessment prior to large-scale production and usage on ranges, testing for postdetonation residues is not common (M. R. Walsh, Bigl, et al. 2018). There is no current alternative, modeling or otherwise, for determining energetic residues

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* For a full list of the spelled-out forms of the units of measure used in this document and their conversions, please refer to *US Government Publishing Office Style Manual*, 31st ed. (Washington, DC: U.S Government Publishing Office, 2016), 245–252, https://www.govinfo.gov/content/pkg/GPO-STYLEMANUAL-2016/pdf/GPO-STYLEMANUAL-2016.pdf.

other than postdetonation residues testing. While live-fire testing reflects the actual conditions in which residues are produced, this method of testing is logistically difficult as it requires operating in a dudded impact area, and it cannot occur early in the acquisition process when munitions are not yet certified for live fire. Command detonation provides a method of quantifying residue loading from new munitions early in the acquisition process.

Table 1. Summarized total energetic residues from US Army Cold Regions Research and Engineering Laboratory (CRREL) high-order detonation tests (Hewitt et al. 2005; M. R. Walsh et al. 2011, 2013, 2014; M. R. Walsh, Bigl, et al. 2018). AFS is the Armaments Center Fuze Simulator, and CFS is the CRREL Fuze Simulator. Munitions for live-fire testing in this project are in *bold*.

Caliber	Munition	Filler	Fuze	Total Residues (mg/cartridge)	Tested Cartridges (*n*)
60 mm	M888	Comp B	Live Fire	0.07	12
	M768	PAX-21	CFS	14,000	7
	M720	IMX-104	CFS	2,200	7
	M720	IMX-104	AFS	3,800	7
81 mm	M374	Comp B	Live Fire	9	17
	M374	Comp B	CFS	10	3
	M889	Comp B	CFS	<0.8	6
	M821	IMX-104	CFS	1,300	16
	M821	IMX-104	AFS	1,800	7
120 mm	M933	Comp B	Live Fire	21	8
155 mm	M107	Comp B	Live Fire	0.3	7
	M107	TNT	Live Fire	<0.1	7
	M1122	IMX-101	CFS	154,000	9
	M1122	IMX-101	AFS	78,000	7

Command detonation is a method of initiating a munition in a static configuration. Critically, command detonation must accurately represent the firing train of the study munition's issued fuze, as booster type and mass have been shown to affect the quantity of residues observed following detonation (Volk 1996; M. R. Walsh et al. 2015). Blow-in-place detonation, as is commonly performed by Explosive Ordnance Disposal, involves initiating a munition with an external charge and has been found to produce significantly greater residues than fuze simulation (M. R. Walsh et al. 2006), so therefore is not representative of live-fire detonation.

The US Army Engineer Research and Development Center's Cold Regions Research and Engineering Laboratory (CRREL) has conducted munition residue tests using two different types of fuze simulators, both of which replaced the study munitions' issued fuzes. The CRREL Fuze Simulator (CFS) is a machined aluminum plug (Figure 1) that can be filled with field-variable masses of C-4 (M. R. Walsh, Walsh, and Hug 2011). A fuze simulator developed by the Army Combat Capabilities Development Command Armaments Center (AFS) used an acrylic body and a pellet booster that matched the booster used in the study munitions' issued fuzes. Both types of fuze simulators were initiated with military blasting caps (i.e., M6 and M7). Cartridges to be tested were oriented in a nose-up orientation to make connection with the blasting cap easier for the operator, whereas the ballistic trajectory of a live-fired cartridge would typically have impact in an approximately nose-down orientation. Additionally, command-detonation tests typically used solid barriers, such as a block of ice or steel plate, placed underneath the study cartridge to minimize potential cratering effects.

Figure 1. The CRREL Fuze Simulator (CFS) packed with C-4 (*left*) and threaded onto a 60 mm mortar cartridge (*right*).

Given these differences imposed by command detonation, there remains uncertainty that this method of testing accurately represents the residues produced during training. Validating command-detonation residues testing would support integrating this testing methodology into the acquisition process for new munitions.

1.2 Objectives

The objective of this study was to quantify energetic residue deposition rates from live-fired cartridges of a 60 mm IMX-104 munition and then to use these data in validating prior command-detonation tests of the same munition. In context of the broader project, this study aimed to provide initial evidence to support validation of command-detonation residue testing for new munitions.

1.3 Approach

The approach for this study focused on a 60 mm IMX-104 munition with a robust existing residue dataset from command-detonation tests using two different fuze simulators. This munition had completed the certification process and was therefore available for live-fire residue testing. The study aimed to collect residue data from at least seven detonations of live-fired 60 mm IMX-104 mortar cartridges by using procedures for sampling, processing, and analysis that were identical to prior command-detonation tests. The sampling procedures used snow as a clean and sensitive medium for collection of deposited solid residues (M. R. Walsh, Walsh, and Ramsey 2007; Jenkins et al. 2002). Statistical comparisons were applied to examine whether residues deposited from command detonation were significantly different from actual residue deposition rates derived from live fire.

2 Methods

2.1 Live firing

Cartridges of the study munition were fired from an M224A1 60 mm mortar system over the course of 3 days at Joint Base Elmendorf-Richardson (JBER), Alaska, in the winter of 2021. The study munition was the M720A2 60 mm mortar cartridge (DODAC 1310-BA44) with IMX-104 insensitive high explosive fill. The munition used the M734A1 multioption fuze with settings for impact (IMP), delay, and proximity (PRX). The first two fired cartridges compared the plume characteristics of the IMP and PRX fuze settings. The setting with the optimal plume characteristics, evidenced by visible soot and residues on the snow surface, was used for firing the remaining cartridges. Target coordinates were provided to the mortar team to maximize separation within the allotted target area in Area C of the Eagle River Flats (ERF) impact area on JBER.

2.2 Sampling

The sampling design and techniques used, including the study location, were identical to prior command-detonation tests of the study munition. Area C is tidally flooded and frozen during winter months, which creates a clean barrier between soil and overlying snow. The visible plumes of deposited residue and soot on snow were first demarcated by walking their perimeter to form *inside-the-plume* (ITP) decision units (DUs). The ITPs included all visible discoloration and disturbance to the snow surface so that they likely contained the vast majority of deposited residues. To ensure complete residue collection, two DUs outboard of each ITP, termed here as *outside the plume* (OTP), were also demarcated with measuring tape from the edge of the ITP to 3 m from the edge (OTP 0–3 m) and from 3 to 6 m from the edge (OTP 3–6 m). Figure 2 conceptualizes these DUs. In the case of overlapping visible plumes from multiple detonations, these plumes were combined into a single set of ITP and OTP DUs.

Samples were collected from each ITP and OTP using *MULTI INCREMENT*® sampling (MIS)[*] of the snow surface, 10 cm × 10 cm × 2.5 cm (depth) aluminum scoops, and polyethylene cleanroom bags. The MIS method requires each sample to comprise many increments collected at regular spacing across the entire DU. Spacing between each increment was

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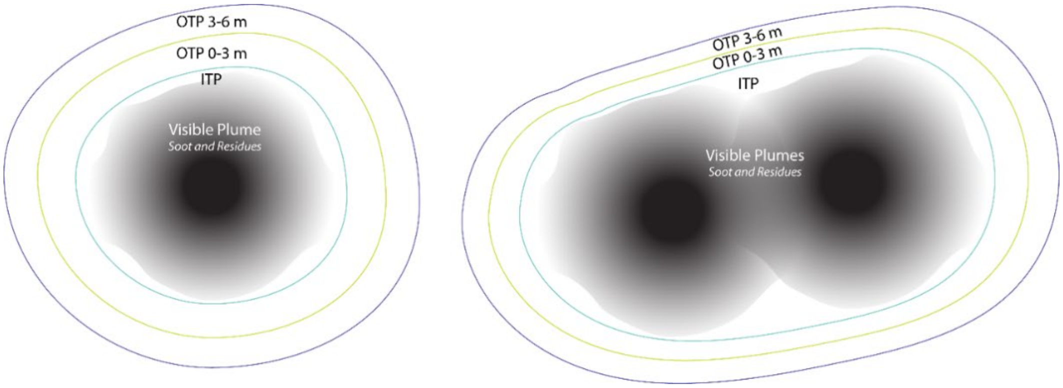
[*] *MULTI INCREMENT*® is a registered trademark of EnviroStat, Inc.

maintained by individual pacing, with a target of collecting 70 to 100 increments per sample; and the number of increments was tracked using a mechanical counter. Each ITP was sampled in triplicate, whereas only select OTPs were sampled in triplicate and the rest sampled with a single replicate. Plume areas were measured using a high-accuracy real-time kinematic survey (Trimble R8 GNSS).

Figure 2. Schematics of sampling areas for a single separated plume (*left*) and two overlapping plumes (*right*). Not to scale.



Prior to live fire, background samples were collected from three 40 m × 40 m areas located between the firing point and the target area. These background samples were collected in an identical manner to the study samples.

At the end of each sampling day, all collected samples were placed in larger polyethylene overbags to prevent sample loss during handling. Background and OTP samples were stored in a tarpaulin-lined clean CONEX box, and ITP samples were stored outside wrapped in a reflective tarpaulin inside a locked yard. Boots, pants, and sampling scoops were cleaned at the end of each sampling day.

2.3 Sample processing

The double-bagged samples were moved indoors and placed in clean plastic containers to melt at room temperature (20°C) for approximately 24 hours prior to processing. Generally, samples were fully melted but still cold prior to processing. Each sample was filtered in its entirety using glass vacuum filtration and glass microfiber filters (Whatman GF/A). Solid material stuck to the interior of the bag was rinsed free into the filtration apparatus with Type I water (Millipore Elix/IQ7000). Filters and solid material were transferred using clean tweezers to new glass sample jars. Aqueous fractions were collected in 2 L batches from the large (~6 L) samples by using 2000 mL volumetric cylinders and combined in large 9 L glass jugs to mix. Total aqueous volumes were recorded for each sample. Aqueous aliquots were collected for NTO analysis (40 mL in glass vials), for solid-phase extraction (SPE; 500 mL in amber glass bottles), and for an archive (500 mL in amber glass bottles). All sample fractions (i.e., solid filters and aqueous aliquots) were refrigerated prior to further processing or shipment.

SPE was performed on a vacuum manifold using Waters Sep-Pak Vac 6 cc (500 mg) RDX cartridges. Each new cartridge was conditioned with 10 mL of acetonitrile and then 20 mL of Type I water. Each 500 mL aqueous aliquot was loaded over approximately an hour using large sample adapters (Supelco Visiprep). Following loading, each cartridge was dried under vacuum for at least 45 minutes and then eluted with 5.00 mL of acetonitrile at slight positive pressure into graduated cylinders. Recovered volumes (4.00 to 4.75 mL) were adjusted to 5.00 mL with acetonitrile, and then aliquots of each SPE sample were stored in a 7 mL amber glass vial for shipment and a 2 mL amber glass vial for archive. SPE samples were stored in a freezer prior to shipping.

All samples were shipped in coolers on blue ice from JBER to the CRREL analytical laboratory in Hanover, New Hampshire. Aqueous fractions and filters were refrigerated, and SPE extracts were stored in a freezer upon receipt. Filter samples were extracted in their jars by adding 20–30 mL of 1/1 (v/v)* acetonitrile/water, vortexing the mixture, and placing it on a shaker table at 170 rpm for 18 hours. Filter extracts were syringe filtered with 0.45 µm Millex-FH PTFE filters.

2.4 Quality assurance and quality control

Quality assurance in sampling included division of sampling teams by DU. Three two-person sampling teams collected all MIS samples. One team collected exclusively ITP samples, one team collected only OTP 3–6 m samples, and one team started each day with OTP 0–3 m samples before moving to collect ITP samples. This order of operation prevented cross contamination between plumes and between DUs. During processing, samples were processed in order of expected ascending residue concentration, beginning with background samples, then OTP samples, and finally

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* Volume/volume.

ITP samples. All used glassware was cleaned with Micro-90 detergent solution followed by Type I water.

Quality control in processing included filter blanks for each filtration apparatus before any samples were run, at the midpoint in sample processing, and after all samples were processed. The filter blanks used 2000 mL of Type I water that was passed through the filtration apparatus, and the filters and aqueous fractions were treated identically to samples. Three SPE blanks each consisted of 500 mL of Type I water and were processed through SPE identically to samples. Matrix spikes, matrix spike duplicates, and sample triplicates were performed by SPE all on the same field sample every 20 samples. Laboratory control samples (LCS) were made from 500 mL Type I water and performed by SPE every 20 samples. Both matrix spikes and LCSs were made by spiking 500 mL samples with 1.00 mL of an intermediate solution containing 2.00 mg/L of the analytes in acetonitrile.

Similar to quality control in SPE, aqueous NTO samples were prepared in triplicate every 20 samples. NTO matrix spikes, matrix spike duplicates, and LCS were made on 2.00 mL aqueous aliquots spiked with 100 µL of 10.0 mg/L NTO in acetonitrile.

2.5 Sample analysis

Each processed sample fraction was analyzed using two different primary chromatographic methods on a high-performance liquid chromatography instrument with a diode array detector (Agilent 1260 Infinity II). For 1,3,5,7-tetranitro-1,3,5,7-tetrazocane (HMX); RDX, and DNAN; the primary method followed US Environmental Protection Agency Method 8330B using a reverse-phase C8 column (Waters NovaPak, 150 × 3.9 mm, 4 µm) at 28°C eluted with 15/85 (v/v) isopropanol/water and 0.1% acetic acid at 1.4 mL/min. The sample injection volume was 100 µL, and the sample matrix was 1/3 (v/v) acetonitrile/water. HMX and RDX were detected at 230 nm and DNAN at 295 nm. The primary method for NTO followed M. E. Walsh (2016) and used a porous graphitic carbon column (Thermo Scientific Hypercarb 150 × 4.6 mm, 5 µm) at 28°C eluted with 3/1 (v/v) acetonitrile/water and 0.1% trifluoroacetic acid at 1.5 mL/min. The sample injection volume was 100 µL, and the sample matrix was 3/1 (v/v) acetonitrile/water. NTO was detected at 321 nm. Appendix B shows chromatograms and spectra.

Confirmation analyses were performed on select samples by using two different secondary chromatographic methods. HMX, RDX, and DNAN were confirmed by using the same conditions for the NTO primary method (i.e., Hypercarb). NTO was confirmed by using a hydrophilic interaction column (Waters XBridge BEH Amide, 100 × 4.6 mm, 2.5 µm) at 28°C eluted with 97/3 (v/v) acetonitrile/water and 0.1% acetic acid at 0.5 mL/min. The injection volume was 20 µL, and the sample matrix was 3/1 (v/v) acetonitrile/water.

Quantification used 8-to-9-point external calibration curves made from commercially available standards from Restek (8095A mix containing HMX, RDX, TNT, and others), Absolute Standards (DNAN), and AccuStandard (NTO). Detection limits were 10 µg/L for HMX, RDX, and DNAN and were 4 µg/L for NTO. Initial calibration verification (ICV) was performed using standards from a separate lot of 8095A mix and from neat materials from Picatinny Arsenal (DNAN and NTO). A mixed standard containing the DNAN transformation products 2,4-dinitrophenol; 2-methoxy-5-nitroaniline; 4-methoxy-3-nitroaniline; 2-methoxy-5-nitrophenol; and 4-methoxy-3-nitrophenol (all from Sigma Aldrich) was run along with the ICV on each day to establish retention times for these degradation products. A continuing calibration verification (CCV) composed of a middle of the calibration range standard was analyzed every 10 samples. Acceptance criteria for the ICV and CCV were ±20% following DoD *Quality Systems Manual* version 5.3 for Method 8330B (DoD and DOE 2019).

2.6 Deposition rate estimation

The residue deposition rate (R) for each IMX-104 compound (i: NTO, DNAN, RDX, and HMX) was determined through the equation below:

$$R_i = \frac{A_\mathrm{p}}{n\,A_\mathrm{s}}\left(C_{i,\mathrm{aq}}V_\mathrm{aq} + C_{i,\mathrm{e}}V_\mathrm{e}\right),$$

where

A_p = the plume area,
n = the number of increments,
A_s = the area of the sampling tool,
C_aq = the measured concentration of the aqueous fraction,
V_aq = the volume of the aqueous fraction,
C_e = the measured concentration in the solid fraction extract, and
V_e = the volume of the solid fraction extract.

The total residue deposition rate was the sum of the rates for each IMX-104 compound. As three replicates were collected from each plume, an average and a standard deviation (SD) were calculated for each plume to assess uncertainty. In the cases of multiple cartridges with overlapping plumes included in a single DU, the deposition rates were divided by the number of cartridges to determine per-cartridge deposition rates. Overall, estimated deposition rates were calculated as the average of deposition rates from all DUs, and uncertainties in the estimates were calculated as SDs and relative standard deviations (RSDs; SD divided by the average).

3 Results and Discussion

3.1 Observations of fuze-setting effect

A total of sixteen M720A2 60 mm IMX-104 were fired, and all cartridges exhibited high-order detonation functioning. The first two fired cartridges were used to compare the residue plumes produced by the IMP and PRX settings on the M734A1 multioption fuze. Observed through telephoto lens, the dark residue cloud produced with the IMP setting was low-lying, and solid material appeared to settle out immediately (Figure 3, Appendix B). Comparatively, the residue cloud from the PRX setting was produced above the ground surface and ultimately drifted and diffused away from the point of detonation.

Figure 3. Residue and soot clouds immediately after detonation of M720A2 using the impact fuze setting (IMP; *left*) and proximity fuze setting (PRX; *right*) on the M734A1 multioption fuze.

At the detonation site, the IMP setting produced a well-defined dark residue and soot plume on the snow surface (Figure 4) with minimal to no cratering, whereas there was little evidence for any residues or soot on the snow surface below the PRX detonation. From these observations, the IMP setting was decided as optimum in preserving residues on the snow surface and the most comparable to prior command-detonation tests. Nevertheless, the comparison indicates a critical role of fuze setting in the distribution of unconsumed energetics on ranges, with implications for their subsequent fate and transport.

Figure 4. Depositional plume from live fire of a study cartridge with IMP fuze setting.

3.2 Sampled depositional plumes

The 14 remaining cartridges were set to the IMP fuze setting and fired into the target area. Targets were adjusted in the field to avoid areas with bare ice cover. Nonetheless, some targeting issues resulted in plumes that were not sampled due to impacting areas of bare ice. Only plumes that encompassed only snow (i.e., no ice patches) were sampled as residues on ice cannot be reliably recovered using the snow-sampling scoop. Figure 5 numbers these demarcated plumes sequentially by order of sampling. Plumes 1 through 7 covered single-cartridge detonations, whereas Plumes 8 and 9 were each the combination of two closely landing cartridge detonations.

For Plume 5, OTP samples were not collected due to the overlap of the OTP areas with bare ice. For Plume 2, its OTP overlapped with what would have been an OTP for an unsampled IMP fuze-setting test cartridge. For double-cartridge Plumes 8 and 9, the ITPs and OTPs covered the combined residue plumes at each site.

Table 2 lists the actual plume areas and number of increments. The demarcated ITP plume areas (Table 2) were greater than previous ones determined in command-detonation studies (190–450 m^2; M. R. Walsh, Bigl, et al. 2018). This difference may have resulted from a difference in residue and soot dispersion or from the subjective determination of visible plume edge.

Figure 5. Plume locations and areas in the target region measured by GPS.

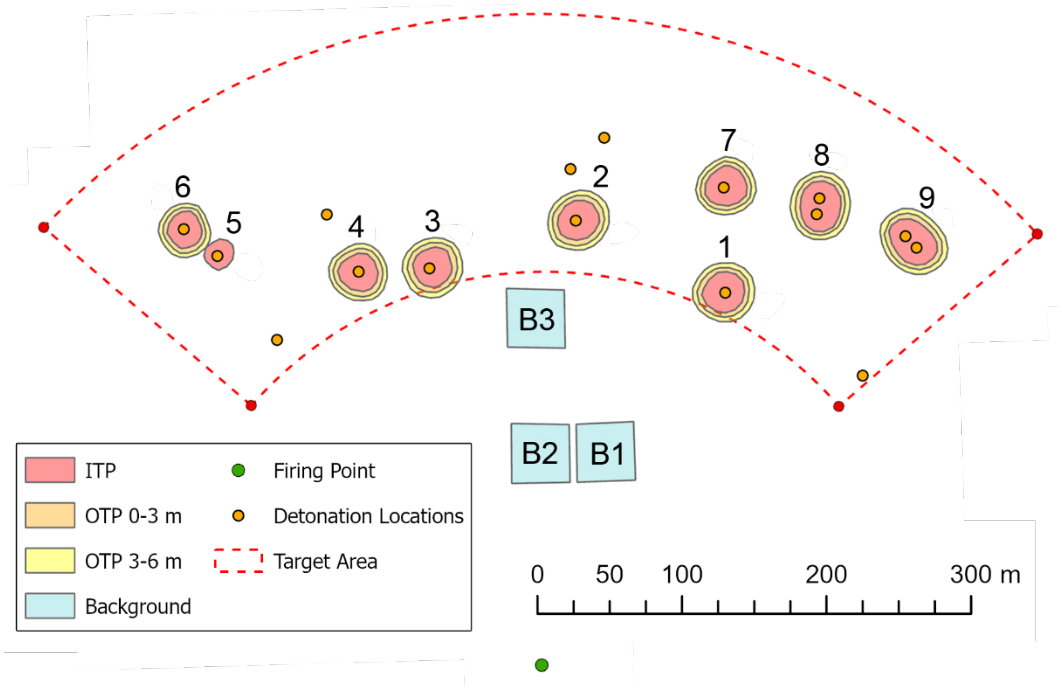
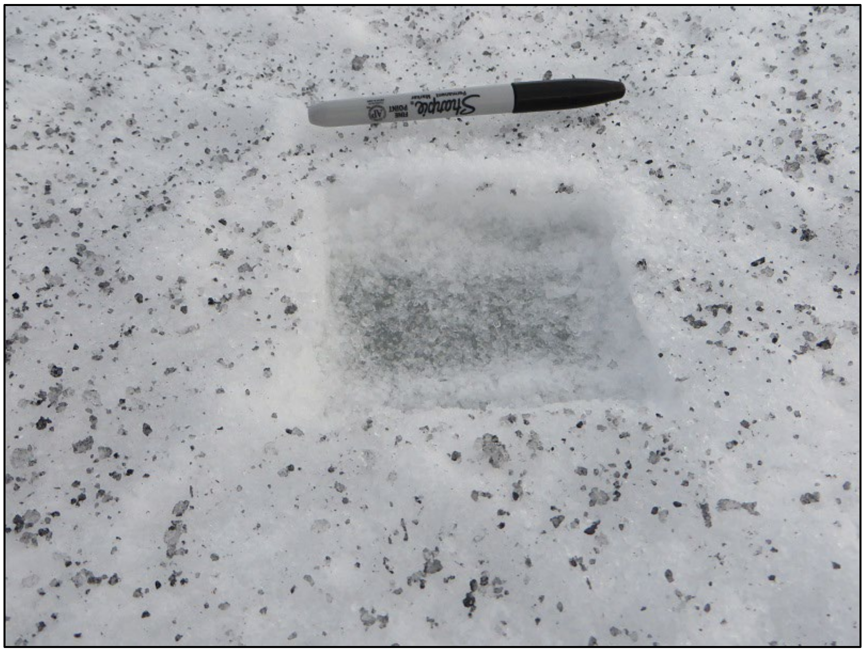


Table 2. GPS-measured areas and *MULTI INCREMENT®* sampling (MIS) increments collected for each plume. Plume 5 did not have OTP samples due to overlap with bare ice. Plumes 8 and 9 each cover residues from two cartridges.

Plume	ITP		OTP 0–3 m		OTP 3–6 m	
	Area (m²)	Increments (*n*)	Area (m²)	Increments (*n*)	Area (m²)	Increments (*n*)
1	632	82–90	327	76	409	97
2	613	86–89	363	97	401	102
3	571	77–114	360	83–101	412	81–97
4	547	84–88	334	85-94	350	96–115
5	322	81–88	—	—	—	—
6	460	84–156	256	93	310	93
7	614	91–105	292	83–98	356	84–102
8	787	78–99	315	88	438	85
9	772	82–120	372	81	442	102

Increments were collected from the surface to the base of the thin (~1–2 cm) snowpack over subsurface ice (Figure 6). The crusted surface required initial outline chiseling using the right-angle corners of the scoop. Normally, the testing method involves the collection of subsurface quality-control samples from below 2.5 cm depth. In this case, the thin snow cover over ice prevented the collection of subsurface samples but also ensured that that no particles were missed below the depth of the scoop (2.5 cm).

Figure 6. An example sample increment showing a thin snow layer and subsurface ice.



3.3 Quality control

Background samples were collected prior to firing just outside the target area to ensure there were no existing energetic compounds in the surface snow. The background sample closest to the target area (Background 3; Figure 5) had no detectable HMX, RDX, DNAN, or NTO (Table 3). However, Backgrounds 1 and 2, which were located closer to the firing point, did have detectable RDX and TNT but no HMX, DNAN, or NTO. There was no evidence of recent use of the impact area. Any prior deposition on the site appeared isolated to Backgrounds 1 and 2 as TNT was absent in all other samples and OTPs generally had RDX near or below the detection limit. These background results provided assurance that the target area was unaffected by prior activity.

Table 3. Energetic-compound masses per area in background samples. Except for detectable RDX and TNT in Backgrounds 1 and 2, all other analytes in background samples were below method detection limits.

Background Sample	Mass per Area (mg/m^2)				
	HMX	RDX	DNAN	NTO	TNT
1	<0.0009	0.0845	<0.0009	<0.03	0.0179
2	<0.0009	0.0777	<0.0009	<0.03	0.0117
3	<0.0009	<0.0009	<0.0009	<0.03	<0.0009

All filtration blanks and SPE blanks met the success criteria of below the method detection limit (Appendix A), indicating no prior or cross contamination of processing glassware and equipment. Triplicate process samples had a precision of 0.8%–19% (Appendix A) and met the success criteria of less than 20%. Matrix spikes had recoveries of 81%–111% (Appendix A) that met the success criteria of 70%–130% and a relative percent difference of 0%–4% that met the success criteria of less than 20%. LCS recoveries (Appendix A) were 94%–110% and met the success criteria of 70%–130%. Altogether these quality-control results indicate acceptable and high-quality data for the study samples.

3.4 Residue deposition rate estimates

All IMX-104 analytes were detected in all nine plumes, presented in Table 4. Consistent across all plumes, residual compound masses follow the trend of NTO >> DNAN > RDX > HMX. The OTP DUs for each plume contained negligible residues relative to those of the ITP DUs, which supports complete sampling of all deposited residues. No transformation products of DNAN were found in any of the samples, indicating negligible degradation of this compound. The pH of aqueous samples ranged from 6.5 to 6.9, so the absence of hydrolysis products was expected. Table 5 and Figure 7 summarize the per-cartridge residue deposition rates and uncertainties in each plume. NTO deposition rates were two orders of magnitude greater than the next highest compound, DNAN. HMX and RDX deposition rates were on the order of 1 to 10 mg per cartridge, approaching the detection limit, similar to high-order detonation residues of Comp B (Table 1). Total residue deposition rates largely reflect the rates of NTO as it was the predominant residual compound. Variability related to sampling is evident in the precision of total residue deposition rates derived from triplicate measurements within each plume, with RSDs of less than or equal to 20%. Broader uncertainties in the estimates, to include variability between all tested cartridges, are characterized in the precision of individual compounds and total residue across all sampled plumes. Precision across all plumes ranged from 24% to 33% RSD, which met the demonstration plan's success criteria of less than 50% RSD and bettered the precision of prior command-detonation tests. The overall high levels of precision provide confidence that the estimated live-fire deposition rates are broadly representative of the munition.

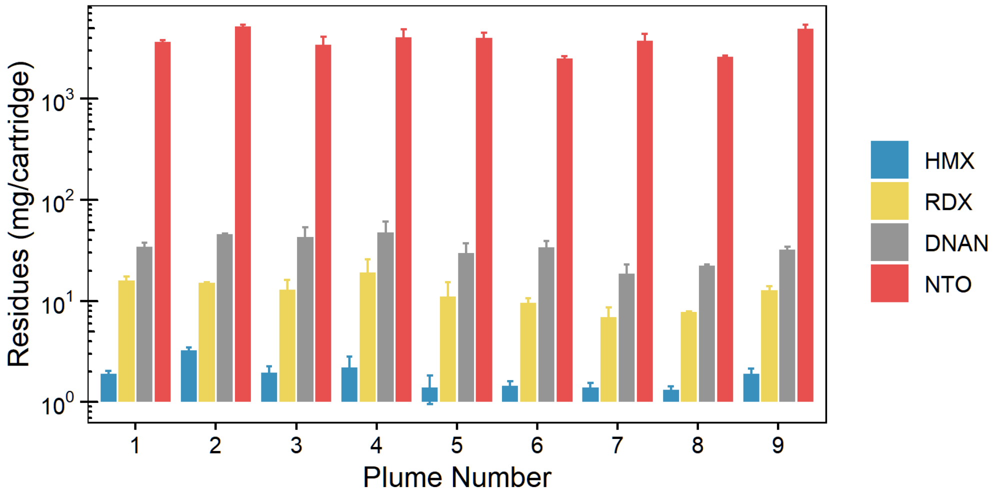
Table 4. Average estimated energetic residue deposition rates within each plume's DUs. Values in parentheses are standard deviations of triplicate MIS estimates. Note that Plumes 8 and 9 each include two cartridge detonations.

Plume	DU Type	Residues (mg/plume)			
		HMX	RDX	DNAN	NTO
1	ITP	1.6 (0.1)	12 (2)	35 (3)	3700 (100)
	OTP 0–3	0.3	<0.3	<0.3	20
	OTP 3–6	<0.4	3.7	<0.4	<11
2	ITP	2.1 (0.2)	14.0 (0.3)	44.8 (0.9)	5200 (200)
	OTP 0–3	0.8	0.4	0.6	29
	OTP 3–6	0.4	0.7	<0.4	<11
3	ITP	1.8 (0.3)	13 (3)	40 (10)	3400 (600)
	OTP 0–3	<0.4	<0.4	<0.4	<11
	OTP 3–6	<0.4	<0.4	<0.4	<12
4	ITP	2.2 (0.6)	19 (7)	50 (10)	4100 (800)
	OTP 0–3	<0.3	<0.3	<0.3	<10
	OTP 3–6	<0.4	<0.4	<0.4	<12
5	ITP	1.4 (0.4)	11 (4)	30 (7)	4000 (500)
6	ITP	1.4 (0.2)	10 (1)	33 (5)	2500 (100)
	OTP 0–3	<0.3	<0.3	0.4	12
	OTP 3–6	<0.3	<0.3	<0.3	<8
7	ITP	1.3 (0.2)	7 (2)	19 (4)	3700 (600)
	OTP 0–3	<0.3	<0.3	<0.3	<10
	OTP 3–6	<0.3	<0.3	<0.3	<10
8	ITP	2.1 (0.2)	14.0 (0.3)	44.8 (0.9)	5200 (200)
	OTP 0–3	<0.3	1.5	<0.3	11
	OTP 3–6	<0.4	<0.4	<0.4	<12
9	ITP	3.5 (0.5)	21 (2)	65 (4)	9800 (1000)
	OTP 0–3	<0.4	4.7	<0.4	17
	OTP 3–6	<0.4	<0.4	<0.4	16

Table 5. Average estimated live-fire energetic residue deposition rates and precision.

	Residues (mg/cartridge)					RSD (%)
Plume	HMX	RDX	DNAN	NTO	Total	Total
1	1.9	16	35	3700	3700	4
2	3.3	15.0	46	5200	5300	3
3	2.0	13	40	3500	3500	19
4	2.2	19	50	4100	4100	20
5	1.4	11	30	4000	4100	12
6	1.4	10	34	2500	2600	6
7	1.4	7	19	3700	3800	17
8	1.3	7.7	22.4	2600	2600	3
9	1.9	14	32	4900	5000	10
Ave.	1.9	12	34	3800	3800	—
SD	0.6	4	10	900	900	—
RSD (%)	33	32	29	24	24	—

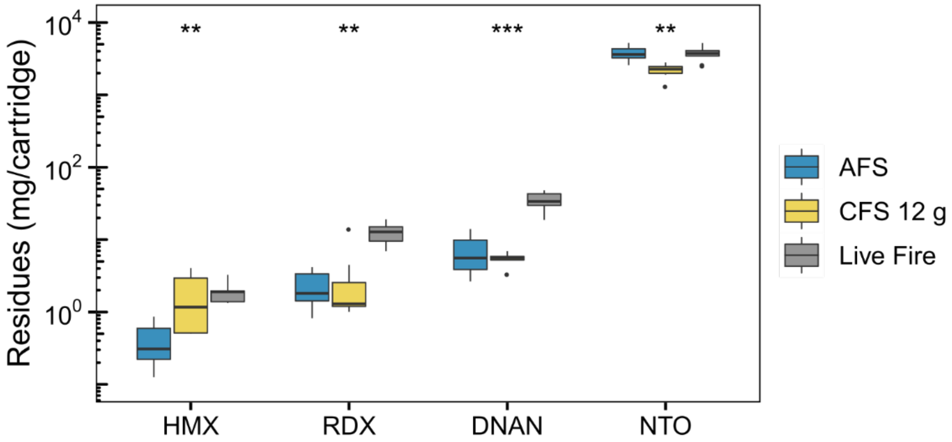
Figure 7. Average estimated energetic residue deposition for each plume. *Error bars* represent one standard deviation.



3.5 Comparison with command detonations

Figure 8 compares the live-fire deposition rates with command-detonation deposition rates for the 60 mm IMX-104 study munition. The box-plots represent the plume-average data distribution of each compound for each fuzing type. Live-fire deposition rates of NTO are largely similar to those using the AFS and generally greater than the CFS. RDX and DNAN deposition rates by live fire are significantly greater than by either fuze simulator. HMX is a minor constituent for all fuzes but is generally greater by live fire than for the AFS and the CFS, the latter of which had some nondetect concentrations.

Figure 8. Boxplots comparing deposition rates from the M720A2 between live-fire tests and previous command-detonation tests using the AFS and CFS (M. R. Walsh, Bigl, et al. 2018). *Boxes* represent the median and interquartile range (IQR), and *whiskers* represent no more than 1.5 times the IQR above and below the hinges. Data beyond the whiskers are marked as outlying points. *Asterisks* represent Kruskal-Wallis rank sum test significance levels (** $p \leq 0.01$; *** $p \leq 0.001$).



Histograms showing the distributions of total residue data (Appendix B) have varying degrees of skew, with only the CFS producing a normal distribution confirmed by the Shapiro-Wilk test. Log10 transformation of the data allowed the live-fire data to pass this normality test; however, the AFS data remained nonnormal. Given these data distributions and the relatively small number of samples, the nonparametric Mann-Whitney test was selected to provide the most robust comparison of means. Applying this test reveals that the total residue deposition rate is significantly different between the CFS and live fire ($p < 0.01$) but is not significantly different between the AFS and live fire ($p = 0.76$; Figure 9).

The live-fire tests confirm that command detonation using the AFS accurately reflects total energetic residue deposition as it occurs during training with the 60 mm IMX-104 munition. The significantly lower total deposition rates using the CFS indicate that the 12 g C-4 booster mass applied to this munition underestimates actual residue production.

While the total deposition rates are similar between live fire and command detonation, they are dominated by NTO and overshadow differences observed in the relatively minor deposition rates of HMX, RDX, and DNAN (Figure 8 and Table 6). The compound HMX is a component of the munition’s fuze and supplemental charge, as well as an impurity in the munition’s IMX-104 filler and in the C-4 booster used by the CFS. HMX deposition rates approached or exceeded the detection limit but tended to be consistently greater by live fire. Deposition rates of RDX and DNAN were significantly greater, by factors of up to 3 and 7, respectively, for live fire compared with the fuze simulators. This underestimation by command detonation of RDX and DNAN would likely have minimal effect on any management decision as the deposition rates of these compounds by live fire are still relatively minimal. However, these observed differences highlight a potential bias in the command-detonation testing method.

Figure 9. Comparison of total residue deposition rates for the M720A2 between the different tested fuzes. *P*-values for Mann-Whitney tests are shown. *Boxes* represent the median and IQR, and *whiskers* represent no more than 1.5 times the IQR above and below the hinges. Data beyond the whiskers are marked as outlying points.

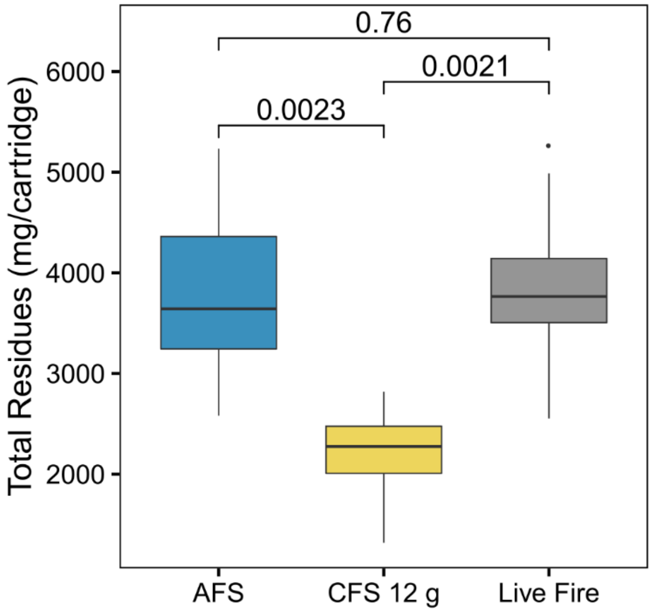


Table 6. Summarized average residue deposition rates using all tested fuzing types for the M720A2. Values are rounded to appropriate significant figures based on plume-average standard deviations shown in parentheses.

Fuze	Mean Residues (mg/cartridge)				
	HMX	RDX	DNAN	NTO	Total
AFS	0.4 (0.3)	2 (1)	7 (4)	3800 (900)	3800 (900)
CFS 12 g	2 (2)	4 (5)	5 (1)	2200 (500)	2200 (500)
Live Fire	1.9 (0.6)	12 (4)	30 (10)	3800 (900)	3800 (900)

There are only two major variables between the live-fire and command-detonation tests: the initiation method and the physical orientation of the cartridges prior to detonation. The similar deposition rates of NTO between the AFS and live-fire tests support the assumption that these equivalent booster charges detonated identically and therefore should have produced similar residues across all IMX-104 compounds. The underestimation of DNAN and RDX by the AFS and CFS suggests loss of some of these residues outside of the sampled deposition areas during the command-detonation tests. Comparison of the phase partitioning between fuze types in the melted samples (Table 7) shows greater aqueous fractionation for DNAN and RDX by live fire. Although the live-fire samples had greater aqueous volumes than the command-detonation tests, due to differences in snow density, measured concentrations were far below each compounds' solubility in water. The greater aqueous partitioning and deposition rates of DNAN and RDX by live fire suggest that the live-fire impact configuration retained fine particles that have relatively increased surface area and solubility. In positioning the cartridges nose-up and on 20 cm thick blocks of solid ice, the command-detonation tests may have promoted the air emission of fine particles composed of RDX and DNAN. The effect of cartridge orientation on residue deposition requires further investigation.

Table 7. Initial proportion of energetic compounds in an aqueous phase from the different M720A2 tests. Results are the average of all ITP measurements with standard deviation in parentheses.

	Aqueous Fraction (% of Total)			
Fuze	HMX	RDX	DNAN	NTO
AFS	24 (6)	76 (10)	15 (6)	100 (0)
CFS 12 g	0 (0)	60 (12)	12 (4)	99 (2)
Live Fire	58 (9)	94 (4)	70 (6)	97 (2)

Quantitative recovery of all residues from detonations, including those deposited in the area immediately surrounding the detonation and those emitted for potential longer-range atmospheric transport, is methodologically challenging to capture. Previous studies have collected air samples following open-air detonations, but typically these collections occurred distal from the point of detonation and relied on carbon monoxide and carbon dioxide emission factors (Aurell et al. 2015) that may not accurately reflect the combustion products of explosives with negative oxygen balance such as IMX-104 (Samuels et al. 2018). One study that was able to position air collections somewhat close to detonations of C-4 plastic explosive found that estimated air emissions accounted for only 7% of the deposited energetic mass (M. R. Walsh, Gullet, et al. 2018). This previous finding, combined with the observations of detonations using the PRX setting, stress the importance of interpreting results from deposited residues as minimum estimates of the total energetic residue from detonations.

Conclusions

Total residue deposition rates for the 60 mm IMX-104 munition determined by live fire are in good agreement with rates from command detonation using the AFS. The underestimation of total residue deposition rates by the CFS highlights the importance of representative fuze simulation in assessing residue loading as it occurs during training. While NTO was generally similar between live fire and command detonation, differences in the minor residue components RDX and DNAN suggest a potential effect of cartridge orientation on residue retention, requiring further investigation. Near quantitative recovery of released residues from detonations was generally supported by live fire with the IMP fuze setting; however, a clear dependency of residue distribution on fuze setting was observed during the live-fire test with the PRX fuze setting that produced no visual deposition.

Altogether, the results of this study support the broader validation of command-detonation testing for other new munitions. This validation will be confirmed in subsequent live-fire testing and comparison of an 81 mm IMX-104 munition during the second task this project. Future integration of this testing methodology into the acquisition process will help the DoD sustain training ranges as improved munitions are developed.

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Appendix A: Quality-Control Data

Table A-1. Processing quality-control sample results. All were below the method detection limit. NTO was not analyzed in SPE samples.

Quality-Control Sample	Concentration (mg/L)			
	HMX	RDX	DNAN	NTO
Prefiltering Blanks (*n* = 2)	<0.0001	<0.0001	<0.0001	<0.004
Midfiltering Blanks (*n* = 2)	<0.0001	<0.0001	<0.0001	<0.004
Postfiltering Blanks (*n* = 2)	<0.0001	<0.0001	<0.0001	<0.004
SPE Blanks (*n* = 3)	<0.0001	<0.0001	<0.0001	—

Table A-2. Precision for aqueous sample triplicates.

Sample ID	Type	Mean Concentration (mg/L)				RSD (%)			
		HMX	RDX	DNAN	NTO	HMX	RDX	DNAN	NTO
21FRA020	Plume 3 OTP 0–3 m	<0.0001	<0.0001	<0.0001	0.004	—	—	—	14
21FRA040	Plume 3 ITP	0.0002	0.0020	0.0048	0.621	10	16	19	0.8
21FRA058	Plume 9 ITP	0.0004	0.0039	0.0091	1.52	8	3	2	1.5

Table A-3. Recoveries and precision for aqueous sample matrix spike (MS) and matrix spike duplicates (MSD). HMX, RDX, and DNAN were spiked at 0.004 mg/L prior to SPE; and NTO was spiked into a direct aqueous aliquot at 0.5 mg/L.

Quality-Control Sample ID	Recovery (%)				Relative Percent Difference (%)			
	HMX	RDX	DNAN	NTO	HMX	RDX	DNAN	NTO
21FRA020 MS	104	101	98	98	0.49	0.03	1.4	0.43
21FRA020 MSD	103	101	96	99	—	—	—	—
21FRA040 MS	104	104	111	96	2.0	3.5	0.23	2.1
21FRA040 MSD	102	98	111	91	—	—	—	—
21FRA058 MS	96	95	98	93	0.29	2.4	1.5	2.9
21FRA058 MSD	96	100	103	81	—	—	—	—

Table A-4. Laboratory control sample (LCS) recoveries. HMX, RDX, and DNAN were spiked at 0.004 mg/L prior to SPE; and NTO was spiked into direct aqueous aliquots at 0.5 mg/L.

Quality-Control Sample	Recovery (%)			
	HMX	RDX	DNAN	NTO
LCS-1	110	96	99	95
LCS-2	106	98	100	96
LCS-3	97	101	96	94

Appendix B: Supplemental Figures

Figure B-1. Example 1 of a depositional and aerial plume immediately following detonation of a 60 mm IMX-104 cartridge from this study using the IMP fuze setting.

Figure B-2. Example 2 of a depositional and aerial plume immediately following detonation of a 60 mm IMX-104 cartridge from this study using the IMP fuze setting.

Figure B-3. Example 3 of a depositional and aerial plume immediately following detonation of a 60 mm IMX-104 cartridge from this study using the IMP fuze setting.

Figure B-4. Evolution of a depositional and aerial plume immediately following detonation of a 60 mm IMX-104 cartridge from this study using the IMP fuze setting.

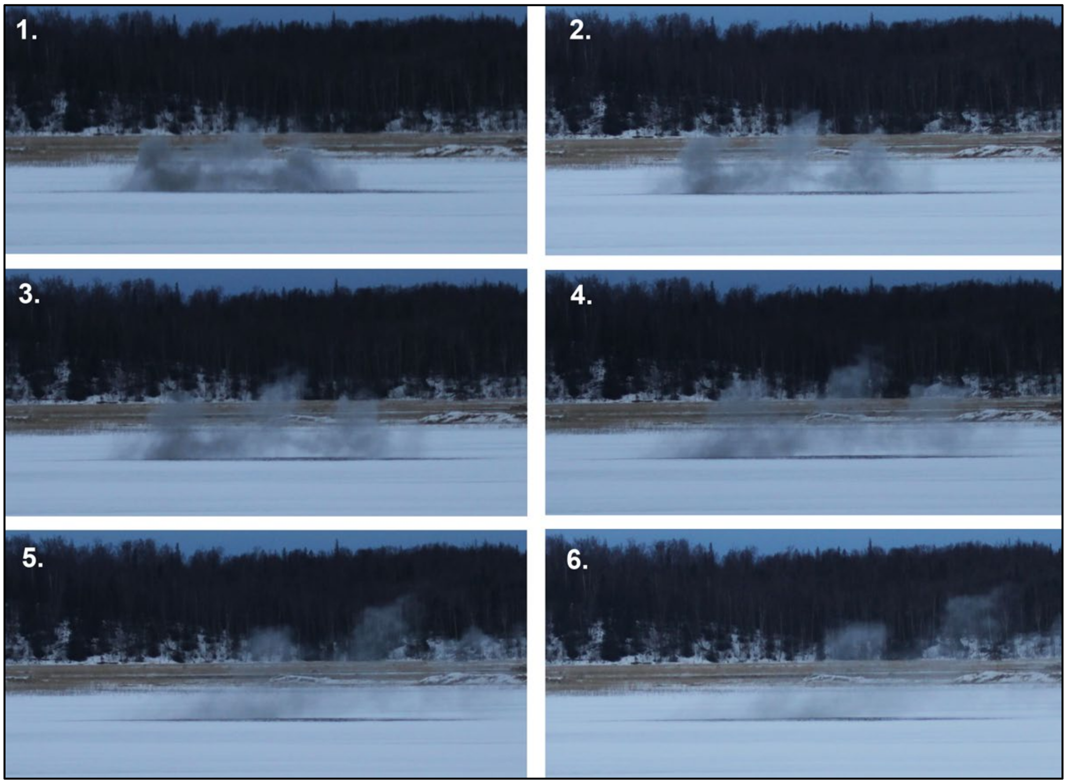


Figure B-5. Example chromatography of standards (1 mg/L) and samples using the NovaPak C8 method for HMX, RDX, DNAN, and other energetic compounds.

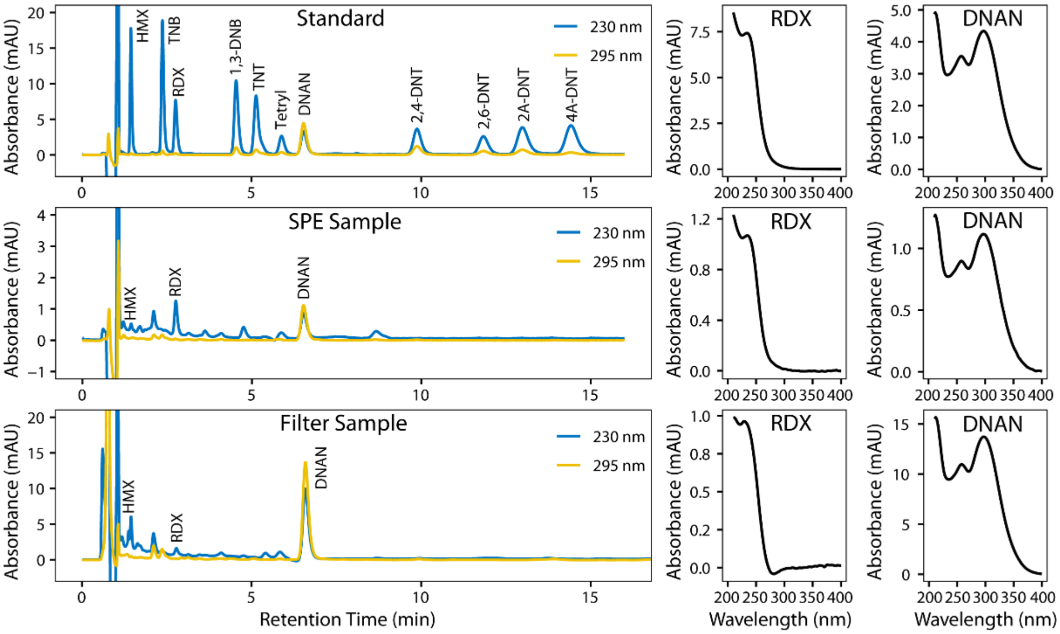


Figure B-6. Example chromatography of a standard (1 mg/L) and samples using the Hypercarb method for NTO.

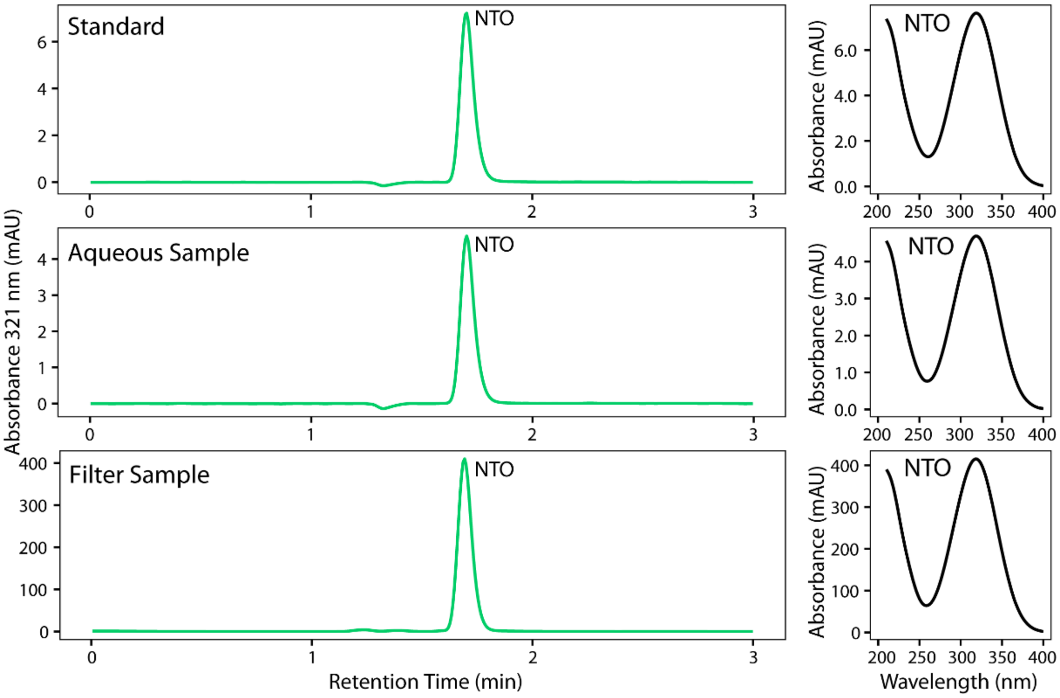
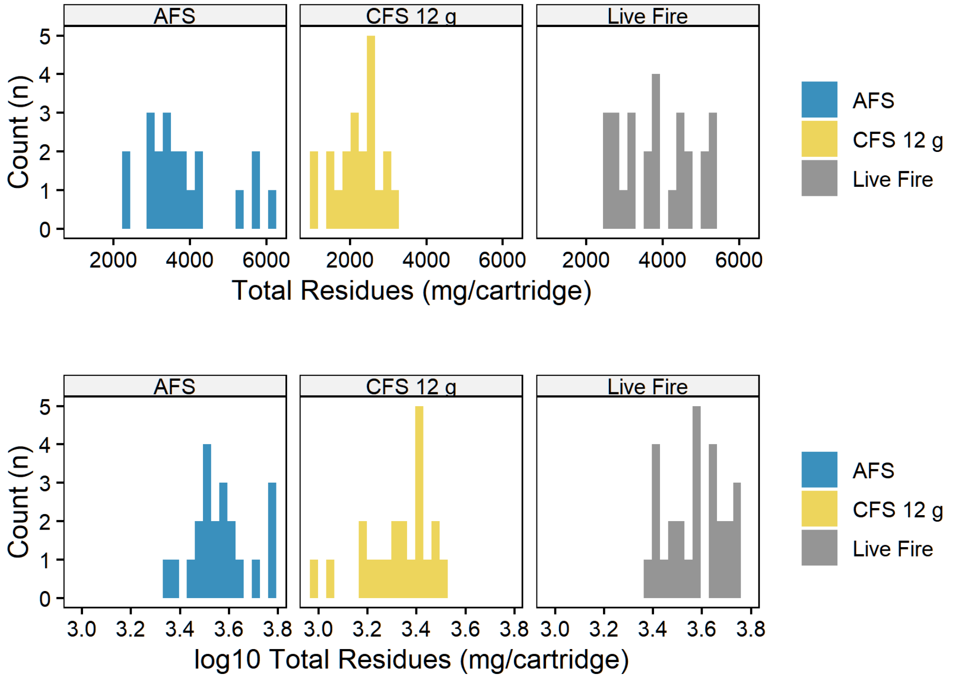


Figure B-7. Histograms of untransformed (*top*) and log10-transformed (*bottom*) total residue deposition rates by fuze type.



Appendix C: Complete Dataset

Table C-1. Sampling and processing data for the 2021 live-fire test of 60 mm IMX-104 cartridges.

ID	Type	Plume Area (m^2)	Increments (*n*)	Filtrate Vol (mL)	SPE Factor	Filter Extract (mL)
21FRA001	Background 1	1611.5	104	6850	100	20.0
21FRA002	Background 2	1634.6	106	7340	100	20.0
21FRA003	Background 3	1613.3	100	6460	100	20.0
21FRA004	Plume 1 OTP 3–6 1/1	409.0	97	6390	100	20.0
21FRA005	Plume 2 OTP 3–6 1/1	401.2	102	7070	100	20.0
21FRA006	Plume 3 OTP 3–6 1/3	412.1	81	5630	100	20.0
21FRA007	Plume 3 OTP 3–6 2/3	412.1	97	6645	100	20.0
21FRA008	Plume 3 OTP 3–6 3/3	412.1	97	6945	100	20.0
21FRA009	Plume 4 OTP 3–6 1/3	349.9	115	7840	100	20.0
21FRA010	Plume 4 OTP 3–6 2/3	349.9	96	7160	100	20.0
21FRA011	Plume 4 OTP 3–6 3/3	349.9	105	7210	100	20.0
21FRA012	Plume 6 OTP 3–6 1/1	310.3	93	6460	100	20.0
21FRA013	Plume 7 OTP 3–6 1/3	356.0	102	6450	100	20.0
21FRA014	Plume 7 OTP 3–6 2/3	356.0	84	5521	100	20.0
21FRA015	Plume 7 OTP 3–6 3/3	356.0	91	6510	100	20.0
21FRA016	Plume 8 OTP 3–6 1/1	437.8	85	5680	100	20.0
21FRA017	Plume 9 OTP 1/1	442.4	102	6320	100	20.0
21FRA018	Plume 1 OTP 0–3 1/1	326.5	76	5480	100	20.0
21FRA019	Plume 2 OTP 0–3 1/1	362.6	97	7480	100	20.0
21FRA020	Plume 3 OTP 0–3 1/3	359.5	101	7810	100	20.0
21FRA021	Plume 3 OTP 0–3 2/3	359.5	83	6260	100	20.0
21FRA022	Plume 3 OTP 0–3 3/3	359.5	92	6640	100	20.0
21FRA023	Plume 4 OTP 0–3 1/3	334.0	94	7500	100	20.0
21FRA024	Plume 4 OTP 0–3 2/3	334.0	85	7260	100	20.0
21FRA025	Plume 4 OTP 0–3 3/3	334.0	93	8200	100	20.0
21FRA026	Plume 6 OTP 0–3 1/1	255.7	93	7175	100	20.0
21FRA027	Plume 7 OTP 0–3 1/3	291.8	98	7245	100	20.0
21FRA028	Plume 7 OTP 0–3 2/3	291.8	83	6720	100	20.0
21FRA029	Plume 7 OTP 0–3 3/3	291.8	88	7568	87.4	20.0
21FRA030	Plume 8 OTP 0–3 1/1	314.8	88	7550	100	20.0
21FRA031	Plume 9 OTP 0–3 1/1	371.8	81	6200	100	20.0
21FRA032	Plume 1 ITP Rep 1/3	631.5	82	5580	100	20.0
21FRA033	Plume 1 ITP Rep 2/3	631.5	90	6375	100	20.0
21FRA034	Plume 1 ITP Rep 3/3	631.5	90	6310	100	20.0
21FRA035	Plume 2 ITP Rep 1/3	613.2	86	6680	100	30.0

Table C-1 (cont.). Sampling and processing data for the 2021 live-fire test of 60 mm IMX-104 cartridges.

ID	Type	Plume Area (m^2)	Increments (*n*)	Filtrate Vol (mL)	SPE Factor	Filter Extract (mL)
21FRA036	Plume 2 ITP Rep 2/3	613.2	89	6890	100	20.0
21FRA037	Plume 2 ITP Rep 3/3	613.2	86	6765	100	30.0
21FRA038	Plume 3 ITP Rep 1/3	571.0	80	5640	100	20.0
21FRA039	Plume 3 ITP Rep 2/3	571.0	114	8480	100	20.0
21FRA040	Plume 3 ITP Rep 3/3	571.0	77	6190	100	20.0
21FRA041	Plume 4 ITP Rep 1/3	547.3	84	6660	100	20.0
21FRA042	Plume 4 ITP Rep 2/3	547.3	88	7220	100	20.0
21FRA043	Plume 4 ITP Rep 3/3	547.3	86	7125	100	20.0
21FRA044	Plume 5 ITP Rep 1/3	322.0	88	6410	100	20.0
21FRA045	Plume 5 ITP Rep 2/3	322.0	86	6000	100	20.0
21FRA046	Plume 5 ITP Rep 3/3	322.0	81	5320	100	20.0
21FRA047A	Plume 6 ITP Rep 2/3 Bag 1/2	460.4	156	7340	100	20.0
21FRA047B	Plume 6 ITP Rep 2/3 Bag 2/2	460.4	156	4120	100	20.0
21FRA048	Plume 6 ITP Rep 1/3	460.4	84	5580	100	20.0
21FRA049	Plume 6 ITP Rep 3/3	460.4	110	7000	100	20.0
21FRA050	Plume 7 ITP Rep 1/3	613.9	105	8140	100	30.0
21FRA051	Plume 7 ITP Rep 2/3	613.9	95	7860	100	20.0
21FRA052	Plume 7 ITP Rep 3/3	613.9	91	7940	100	20.0
21FRA053	Plume 8 ITP Rep 1/3	786.6	78	6320	100	20.0
21FRA054	Plume 8 ITP Rep 2/3	786.6	99	8000	100	20.0
21FRA055	Plume 8 ITP Rep 3/3	786.6	87	7050	100	20.0
21FRA056	Plume 9 ITP Rep 1/3	771.8	102	6760	100	20.0
21FRA057	Plume 9 ITP Rep 3/3	771.8	82	5965	100	20.0
21FRA058	Plume 9 ITP Rep 2/3	771.8	120	8680	100	20.0

Table C-2. Analysis data for the filtrate fraction of samples from the 2021 live-fire test of 60 mm IMX-104 cartridges.

	Aqueous (mg/L)	Aqueous SPE (mg/L)			
ID	NTO	HMX	RDX	TNT	DNAN
21FRA001	<0.004	<0.01	1.150	0.239	<0.01
21FRA002	<0.004	<0.01	0.867	0.162	<0.01
21FRA003	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA004	<0.004	<0.01	0.124	<0.01	<0.01
21FRA005	<0.004	0.013	0.025	<0.01	<0.01
21FRA006	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA007	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA008	<0.004	<0.01	0.010	<0.01	<0.01
21FRA009	<0.004	<0.01	<0.01	<0.01	<0.01

Table C-2 (cont.). Analysis data for the filtrate fraction of samples from the 2021 live-fire test of 60 mm IMX-104 cartridges.

ID	Aqueous (mg/L) NTO	Aqueous SPE (mg/L)			
		HMX	RDX	TNT	DNAN
21FRA010	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA011	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA012	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA013	<0.004	<0.01	0.012	<0.01	<0.01
21FRA014	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA015	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA016	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA017	0.006	<0.01	<0.01	<0.01	<0.01
21FRA018	0.008	<0.01	<0.01	<0.01	<0.01
21FRA019	0.010	<0.01	0.014	<0.01	0.023
21FRA020	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA021	<0.004	<0.01	<0.01	<0.01	0.011
21FRA022	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA023	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA024	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA025	<0.004	<0.01	0.015	<0.01	<0.01
21FRA026	0.006	<0.01	<0.01	<0.01	0.019
21FRA027	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA028	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA029	0.005	<0.01	<0.01	<0.01	<0.01
21FRA030	0.004	0.011	0.056	<0.01	<0.01
21FRA031	0.006	<0.01	0.165	<0.01	<0.01
21FRA032	0.787	0.012	0.202	<0.01	0.461
21FRA033	0.765	0.018	0.249	<0.01	0.587
21FRA034	0.837	0.016	0.251	<0.01	0.574
21FRA035	1.012	0.028	0.271	<0.01	0.691
21FRA036	1.084	0.029	0.281	<0.01	0.676
21FRA037	1.053	0.026	0.270	<0.01	0.658
21FRA038	1.005	0.028	0.363	<0.01	0.943
21FRA039	0.738	0.024	0.257	<0.01	0.677
21FRA040	0.620	0.022	0.216	<0.01	0.539
21FRA041	1.046	0.041	0.557	<0.01	0.997
21FRA042	0.691	0.024	0.263	<0.01	0.556
21FRA043	0.951	0.032	0.403	<0.01	0.836
21FRA044	1.869	0.034	0.486	<0.01	0.835
21FRA045	1.589	0.025	0.287	<0.01	0.631

Table C-2 (cont.). Analysis data for the filtrate fraction of samples from the 2021 live-fire test of 60 mm IMX-104 cartridges.

	Aqueous (mg/L)	Aqueous SPE (mg/L)			
ID	NTO	HMX	RDX	TNT	DNAN
21FRA046	1.665	0.045	0.674	<0.01	1.000
21FRA047A	0.623	0.016	0.192	<0.01	0.529
21FRA047B	1.010	0.043	0.472	<0.01	1.093
21FRA048	0.778	0.029	0.268	<0.01	0.724
21FRA049	0.808	0.033	0.324	<0.01	0.888
21FRA050	0.858	0.014	0.156	<0.01	0.254
21FRA051	0.692	0.013	0.139	<0.01	0.254
21FRA052	0.564	0.012	0.085	<0.01	0.196
21FRA053	1.361	0.041	0.383	<0.01	1.034
21FRA054	1.010	0.034	0.334	<0.01	1.008
21FRA055	1.544	0.033	0.273	<0.01	0.710
21FRA056	2.036	0.047	0.416	<0.01	0.858
21FRA057	1.775	0.035	0.315	<0.01	0.745
21FRA058	1.541	0.037	0.384	<0.01	0.907

Table C-3. Analysis data for solid filter extractions of samples from the 2021 live-fire test of 60 mm IMX-104 cartridges.

	Filter Extractions (mg/L)				
ID	NTO	HMX	RDX	TNT	DNAN
21FRA001	<0.004	<0.01	0.453	0.110	<0.01
21FRA002	<0.004	<0.01	0.940	0.023	<0.01
21FRA003	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA004	0.006	<0.01	0.042	<0.01	<0.01
21FRA005	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA006	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA007	0.005	<0.01	<0.01	<0.01	<0.01
21FRA008	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA009	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA010	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA011	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA012	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA013	0.008	<0.01	<0.01	<0.01	<0.01
21FRA014	0.008	<0.01	<0.01	<0.01	<0.01
21FRA015	0.008	0.035	<0.01	<0.01	<0.01
21FRA016	<0.004	0.028	<0.01	<0.01	<0.01

Table C-3 (cont.). Analysis data for solid filter extractions of samples from the 2021 live-fire test of 60 mm IMX-104 cartridges.

	Filter Extractions (mg/L)				
ID	NTO	HMX	RDX	TNT	DNAN
21FRA017	<0.004	0.034	<0.01	<0.01	<0.01
21FRA018	0.008	0.030	<0.01	<0.01	<0.01
21FRA019	0.004	0.112	<0.01	<0.01	<0.01
21FRA020	0.008	0.031	<0.01	<0.01	<0.01
21FRA021	<0.004	0.028	<0.01	<0.01	<0.01
21FRA022	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA023	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA024	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA025	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA026	0.004	<0.01	<0.01	<0.01	<0.01
21FRA027	0.007	<0.01	<0.01	<0.01	<0.01
21FRA028	0.004	<0.01	<0.01	<0.01	<0.01
21FRA029	0.007	<0.01	<0.01	<0.01	<0.01
21FRA030	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA031	<0.004	<0.01	<0.01	<0.01	<0.01
21FRA032	13.542	0.065	0.123	<0.01	0.718
21FRA033	9.500	0.068	0.136	<0.01	0.783
21FRA034	8.378	0.067	0.151	<0.01	0.710
21FRA035	6.784	0.032	0.033	<0.01	0.536
21FRA036	9.563	0.068	0.062	<0.01	1.005
21FRA037	9.268	0.032	0.049	<0.01	0.584
21FRA038	9.265	0.070	0.134	<0.01	1.205
21FRA039	8.145	0.052	0.086	<0.01	1.021
21FRA040	7.017	0.048	0.048	<0.01	0.658
21FRA041	8.590	0.079	0.125	<0.01	1.089
21FRA042	2.238	0.041	0.029	<0.01	0.615
21FRA043	9.772	0.057	0.050	<0.01	1.229
21FRA044	26.790	0.089	0.063	<0.01	1.721
21FRA045	23.265	0.050	0.025	<0.01	0.973
21FRA046	27.205	0.105	0.072	<0.01	1.784
21FRA047A	6.284	0.051	0.030	<0.01	0.888
21FRA047B	6.577	0.072	0.048	<0.01	1.257
21FRA048	1.925	0.034	0.006	<0.01	0.468
21FRA049	8.309	0.060	0.060	<0.01	1.123
21FRA050	15.267	0.045	0.029	<0.01	0.478
21FRA051	20.450	0.035	0.053	<0.01	0.694

Table C-3 (cont.). Analysis data for solid filter extractions of samples from the 2021 live-fire test of 60 mm IMX-104 cartridges.

ID	Filter Extractions (mg/L)				
	NTO	HMX	RDX	TNT	DNAN
21FRA052	3.534	0.053	0.024	<0.01	0.247
21FRA053	17.908	0.080	0.058	<0.01	1.238
21FRA054	0.274	0.065	0.055	<0.01	1.110
21FRA055	9.496	0.081	0.066	<0.01	1.347
21FRA056	11.102	0.110	0.070	<0.01	1.544
21FRA057	15.370	0.071	0.038	<0.01	0.946
21FRA058	8.359	0.088	0.039	<0.01	1.269

Abbreviations

AFS	Armaments Center Fuze Simulator
C-4	Composition C4, a Formulation Containing RDX
CCV	Continuing Calibration Verification
CFS	CRREL Fuze Simulator
Comp B	Composition B, a Formulation Containing TNT and RDX
CRREL	Cold Regions Research and Engineering Laboratory
DNAN	2,4-Dinitroanisole
DU	Decision Unit
ERF	Eagle River Flats
HMX	1,3,5,7-Tetranitro-1,3,5,7-Tetrazocane
HPLC	High-Performance Liquid Chromatography
ICV	Initial Calibration Verification
IM	Insensitive Munitions
IMP	Impact Fuze Setting
IMX-101	An Insensitive Munition Formulation Containing DNAN, NTO, and Nitroguanidine
IMX-104	An Insensitive Munition Formulation Containing DNAN, NTO, and RDX
IQR	Interquartile Range
ITP	Inside the Plume
JBER	Joint Base Elmendorf-Richardson
LCS	Laboratory Control Sample
MIS	*MULTI INCREMENT* Sampling

MS	Matrix Spike
MSD	Matrix Spike Duplicate
NTO	3-Nitro-1,2,4-Triazol-5-One
NQ	Nitroguanidine
OTP	Outside the Plume
PRX	Proximity Fuze Setting
RDX	1,3,5-Trinitroperhydro-1,3,5-Triazine
RSD	Relative Standard Deviation
SD	Standard Deviation
SPE	Solid-Phase Extraction
TNT	2,4,6-Trinitrotoluene
v/v	Volume/Volume

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14. ABSTRACT

Command detonation (i.e., static firing) provides a method of testing munitions for their postdetonation residues early in the acquisition process. However, necessary modifications to the firing train and cartridge orientation raise uncertainty whether command detonation accurately represents residue deposition as it occurs during live-fire training. This study collected postdetonation residues from live-fired 60 mm IMX-104 mortar cartridges and then compared estimated energetic-compound deposition rates between live fire and prior command detonations of the same munition. Average live-fire deposition rates of IMX-104 compounds determined from 11 detonations were 3800 mg NTO (3-nitro-1,2,4-triazol-5-one), 34 mg DNAN (2,4-dinitroanisole), 12 mg RDX (1,3,5-Trinitroperhydro-1,3,5-Triazine), and 1.9 mg HMX (1,3,5,7-Tetranitro-1,3,5,7-Tetrazocane) per cartridge. Total live-fire residue deposition (mean ± standard deviation: 3800 ± 900 mg/cartridge) was not significantly different from command detonation using a representative fuze simulator (3800 ± 900 mg/cartridge, $n = 7$, $p = 0.76$) but was significantly different from command detonation using a simplified fuze simulator (2200 ± 500 mg/cartridge, $n = 7$, $p < 0.01$). While the dominant residue compound NTO was broadly similar between live fire and command detonation, the minor residue compounds RDX and DNAN were underestimated during command detonation by a factor of approximately three to seven.

15. SUBJECT TERMS
Energetic compounds; Explosives, military--environmental aspects; Explosives, military--residues; Fuze simulation; Impact area; Insensitive munitions; Munitions constituents; Propellants--residues; Range sustainment; Source loading

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