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*Environmental Security Technology Certification Program (ESTCP)*

## **Live-Fire Validation of Command-Detonation Residues Testing Using an 81 mm IMX-104 Munition**

Samuel A. Beal, Matthew F. Bigl, and Charles A. Ramsey

April 2023



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# **Live-Fire Validation of Command-Detonation Residues Testing Using an 81 mm IMX-104 Munition**

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## Abstract

Postdetonation energetic residues can have environmental impacts that present a risk to military training-range sustainment. As munitions with new explosive formulations are developed and fielded, quantitative methods for assessing their residues are needed. Command detonation (i.e., static firing) allows residue testing to occur early in the acquisition process; however, its representation of live-fire residue production is uncertain due to differences in the initiation mechanism and cartridge orientation. This study aims to validate residue testing by command detonation through statistical comparison of residue deposition rates between live fire and command detonation. Live-fire residues were collected from fourteen 81 mm IMX-104 mortar cartridges fired onto snow, and deposition rates were compared with previous command-detonation tests of the same munition. Average live-fire deposition rates were 8000 mg NTO (3-nitro-1,2,4-triazol-5-one), 60 mg DNAN (2,4-dinitroanisole), 20 mg RDX (1,3,5-trinitroperhydro-1,3,5-triazine), and 2 mg HMX (1,3,5,7-tetranitro-1,3,5,7-tetrazocane) per cartridge. Compared to command detonation, live fire of the study munition produced significantly greater residues of NTO ( $p < 0.0001$ ) and RDX ( $p = 0.01$ ) but not DNAN ( $p = 0.067$ ). Although absolute deposition rates of some IMX-104 compounds differed, command detonation was successful at predicting the order of magnitude of each IMX-104 compound for the studied 81 mm munition.

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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 SO491, SO492, and SO1021. Dr. Herb Nelson was executive director for SERDP-ESTCP, and Dr. Andrea Leeson was deputy director and project monitor.

This report was prepared by the Biogeochemical Sciences Branch (BSB) and the Engineering Resources Branch (ERB) of the Research and Engineering Division, US Army Engineer Research and Development Center, Cold Regions Research and Engineering Laboratory (ERDC-CRREL). Researchers from ERDC-CRREL collaborated with EnviroStat, Inc., of Vail, Arizona. At the time of publication, Mr. Nathan Lamie was chief, BSB; Dr. Melisa Nallar was chief, ERB; and Dr. Caitlin A. Callaghan was division chief. The acting deputy director of ERDC-CRREL was Dr. Ivan P. Beckman, and the director was Dr. Joseph L. Corriveau.

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As this report captures the continuation of a previous project, portions of this report have been modified and reprinted from S. A. Beal, M.F. Bigl, and C.A. Ramsey, *Live-Fire Validation of Command-Detonation Residues Testing Using a 60 mm IMX-104 Munition*, ERDC/CRREL TR-22-13 (Hanover, NH: US Army Engineer Research and Development Center, Cold Regions Research and Engineering Laboratory, 2022), <http://dx.doi.org/10.21079/11681/45266>. Public domain.

COL Christian Patterson was commander of ERDC, and Dr. David W. Pittman was the director.

# 1 Introduction

## 1.1 Background

Energetic residues from munition firing and detonation have the potential to impact the environment and threaten range sustainment (Jenkins et al. 2002; M. R. Walsh, Walsh, Poulin, et al. 2011). Research by the US Army Engineer Research and Development Center–Cold Regions Research and Engineering Laboratory (ERDC-CRREL), funded primarily by the Strategic Environmental Research Development Program (SERDP), identified that, while conventional munitions produce minimal postdetonation energetic residue (typically less than 10 mg/round),\* new insensitive munitions deposit significantly greater postdetonation residues that could present an environmental risk (M.R. Walsh et al. 2013, 2014; M. R. Walsh, Bigl, et al. 2018). These research findings were determined through command detonation and highlighted the need for munition postdetonation residue data to accurately predict potential environmental impacts on ranges.

While postdetonation gaseous and condensed-phase products are often calculated using thermochemical models, there is no existing theory to support prediction of unreacted residual explosives following detonation (Abdul-Karim et al. 2014). Therefore, the only current method for testing a munition early in the acquisition process, when changes can be made most efficiently, is by command detonation (i.e., static detonation). Command detonation can occur before a munition is certified for live firing from a weapon system and allows the test to be performed in a controlled location. The key uncertainty in this practice, however, is whether residue production is affected by replacing the munition's standard fuze with a fuze simulator and orienting the munition in a static position. Previous studies have shown that fuze booster type and mass affect residue production (Volk 1996; M.R. Walsh et al. 2015), so close replication of a munitions' issued fuze is likely essential for accurate representation of live fire.

The Environmental Security Technology Certification Program (ESTCP) funded this project (ER20-5018) to validate command detonation as a

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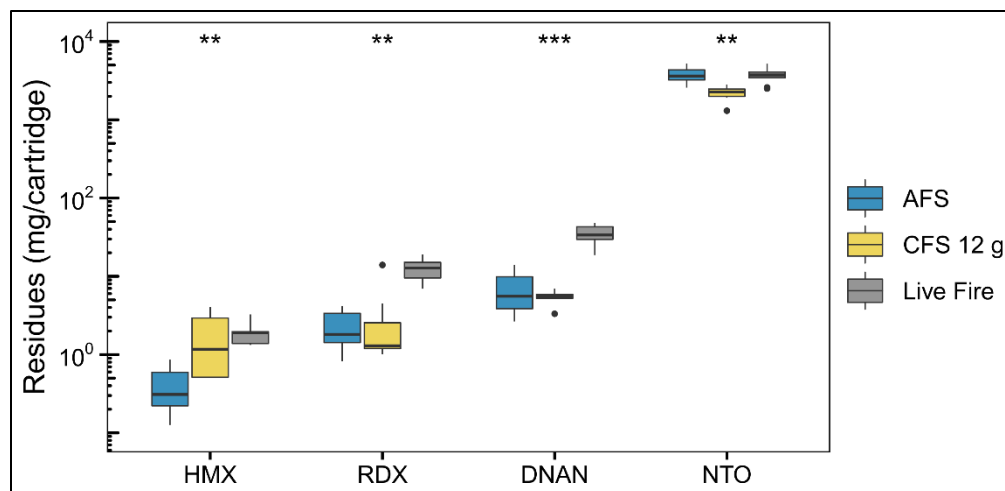
\* For a full list of the spelled-out forms of the units of measure used in this document, please refer to *US Government Publishing Office Style Manual*, 31st ed. (Washington, DC: US Government Publishing Office, 2016), 248–52, <https://www.govinfo.gov/content/pkg/GPO-STYLE-MANUAL-2016/pdf/GPO-STYLEMANUAL-2016.pdf>.

representative means of testing munitions for energetic residues. This validation involved collecting new residue data from live firing, in the same manner as it occurs during training, of two munitions that have existing robust residue datasets by command detonation. The munitions are 60 mm and 81 mm mortar cartridges each filled with the insensitive high explosive IMX-104, which is composed of the compounds 2,4-dinitroanisole (DNAN); 3-nitro-1,2,4-triazol-5-one (NTO); and 1,3,5-trinitroperhydro-1,3,5-triazine (RDX). Both munitions have been well characterized by command detonation (M. R. Walsh, Bigl, et al. 2018) using fuze simulators developed by CRREL and by the Army Combat Capabilities Development Command Armaments Center (DEVCOM AC). The CRREL fuze simulator (CFS) uses a field-adjustable mass of C-4 (RDX and wax) as a booster (M. R. Walsh, Walsh, and Hug 2011), whereas the DEVCOM AC fuze simulator (AFS) uses a booster charge containing 1,3,5,7-tetranitro-1,3,5,7-tetrazocane (HMX) that matches the composition and mass of the munitions' issued fuzes. In the previous command-detonation tests of the study munitions, both types of fuze simulators were initiated with standard (M6 or M7) military blasting caps.

The results of the live-fire validation with the 60 mm IMX-104 mortar munition, Task 1 of this ESTCP project, were discussed in Beal et al. (2022). In brief, the live-fire total-residue-deposition rate for this munition was significantly different from command detonation using the CFS but not significantly different from the AFS. The predominant energetic compound in residues from this munition was NTO, whose mean deposition rate was identical between live fire and command detonation using the AFS. However, deposition rates of the compounds RDX and DNAN were significantly greater by live fire than by command detonation using either of the fuze simulators (Figure 1). Beal et al. (2022) hypothesized that differences in these minor residue compounds were caused by an effect of munition orientation on the surface recovery of fine-particle-size residues.

While the results of Task 1 support representative residue production by command detonation, more complete validation of the method requires extension to other munitions. This report details results of the Task 2 live-fire validation using an 81 mm IMX-104 mortar cartridge. Successful validation would enable confident integration of this technology into the testing process for new munitions, with the data generated informing acquisition and range managers on potential environmental impacts and risks to training-range sustainment.

Figure 1. Residue deposition rates for the 60 mm IMX-104 mortar munition (M720A2) determined by command detonation (Walsh et al. 2014; M. R. Walsh, Bigl, et al. 2018) and live fire (Beal et al. 2022). Boxes represent the median and interquartile range, and whiskers represent no more than 1.5 times the interquartile range (IQR) above and below the hinges. Data beyond the whiskers are marked as outlying points. Asterisks represent Kruskal-Wallis rank sum test significance levels (\*\*  $p \leq 0.01$ ; \*\*\*  $p \leq 0.001$ ).



## 1.2 Objectives

The objectives of this study were to (1) quantify energetic residue deposition rates from live-fire detonations of 81 mm IMX-104 cartridges and (2) compare deposition rates between live-fire and previously conducted command-detonation tests of the same munition. Combined with the prior live-fire study of the 60 mm IMX-104 munition, this project aimed to provide empirical support for the validation of command-detonation residue testing.

## 1.3 Approach

This study leveraged a robust residue dataset from a completed SERDP project (ER-2219) for an 81 mm IMX-104 munition that was collected through command detonations with multiple fuze simulators. As this munition was certified for live fire, the same methods used previously to characterize its residues by command detonation could be applied to actual live-fire training. These methods involved the sampling of snow surrounding the detonation point, which serves as a sensitive and clean medium for collecting deposited solid residues (Jenkins et al. 2002; M. R. Walsh et al. 2007, 2012). The study aimed to quantify residue deposition rates from at least seven live-fire detonations of the study munition. Once collected, statistical comparisons between live fire and command detonation tested the hypothesis of no significant differences in residue deposition rates.

## 2 Methods

### 2.1 Live firing

The study munition was the M889A4 (DODAC:\* 1315-CA63) 81 mm mortar cartridge filled with IMX-104 and using a booster charge composed of PBXW-14, a polymer-bonded explosive containing HMX. This munition is identical to the M821A3 used in previous command-detonation tests, with the exception of the included fuze. While the M821A3 uses the M734A1 multioption fuze, similar to the M720A2 60 mm munition studied in Task 1 of this project, the M889A4 uses the M783 fuze with settings for only point detonating or delay. Both fuzes have the same HMX-based booster composition (PBXN-5, another polymer-bonded explosive containing HMX) and mass, but the DoD generally prefers M783 for training due to its lower cost.

Live firing occurred over four days in January 2022. Cartridges of the study munition were fired from an M252 mortar system onto snow in the Eagle River Flats (ERF) impact area at Joint Base Elmendorf-Richardson, Alaska. The M783 fuzes on all fired cartridges were set to point detonating (i.e., impact). Coordinates that maximized target separation within the target area were provided to the mortar team.

### 2.2 Sampling

The methods for sampling, processing, and analysis in this study were identical to those used in Task 1 and reported in Beal et al. (2022), which, briefly, were as follows.

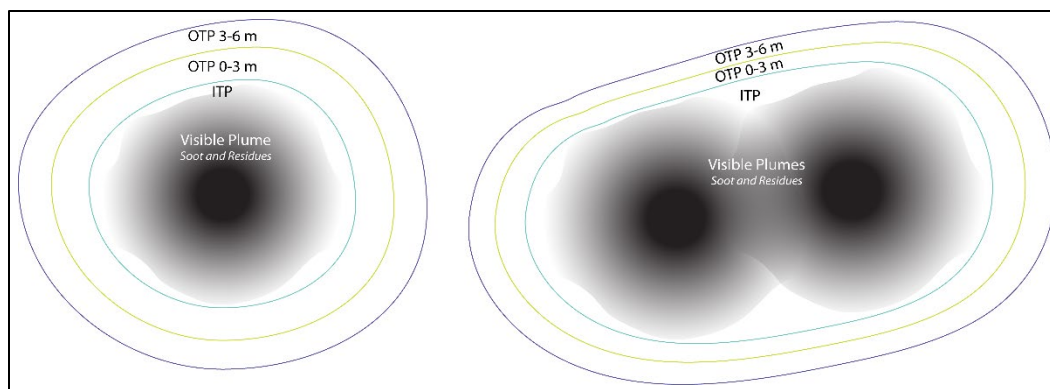
As in previous command-detonation tests of 81 mm IMX-104 mortar cartridges at ERF, the sampling design incorporated Multi-Increment Sampling (MIS) of decision units (DUs) defined by the visible plume of soot distributed on the snow surface. Figure 2 shows the conceptual layout of DUs for each plume or, in the case of two overlapping plumes, plume set: the inside-the-plume (ITP) DU that covers nearly all the deposited residues (M. R. Walsh et al. 2012); the outside-the-plume (OTP) DU from 0 to 3 m outside the edge of the ITP; and the OTP DU from 3 to 6 m outside the

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\* DoD Ammunition Code.

edge of the ITP. The OTPs largely serve as quality-control checks on complete recovery of deposited residues.

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



Samples were collected from the snow surface of each DU using 10 cm × 10 cm × 2.5 cm (depth) aluminum scoops and polyethylene cleanroom bags. Each MIS sample comprised many increments collected at regular spacing across the entire DU, with a target of 100 increments. The ITP of each plume was sampled in triplicate, and OTPs of one plume per day were sampled in triplicate, with the remaining OTPs sampled with a single replicate. DU areas were measured using a high-accuracy, real-time kinematic survey (Trimble R8 GNSS).

Background samples were collected using MIS of the surface snow from 40 m × 40 m areas directly surrounding the target area. These samples were collected before firing, in the middle of firing, and after all cartridges had been fired.

Samples were protected in larger polyethylene overbags and kept frozen until the day prior to processing.

### 2.3 Sample processing and quality control

The samples melted at room temperature (20°C) for approximately 24 hr and were still generally cold prior to processing. The entirety of each sample was filtered through glass microfiber filters (Whatman GF/A) using glass vacuum filtration (Kontes). Filters with attached particulates were stored in glass jars, and the filtrate was mixed and then aliquoted for direct analysis (40 mL glass vial), solid phase extraction (SPE; 500 mL glass bottle), and archive (500 mL glass bottle). Conditioned SPE cartridges

(Waters Sep-Pak Vac RDX 6 cc 500 mg) were loaded with 500 mL of aqueous sample and then eluted with 5 mL of acetonitrile for a hundredfold concentration factor. Filters were extracted with 20–50 mL of 1/1 (v/v)\* acetonitrile/water on a shaker table at 170 rpm for 18 hr, then syringe filtered into glass scintillation vials (Millex-FH PTFE 0.45  $\mu\text{m}$ ).

Quality control included filter blanks (before, in the middle of, and after processing all samples), SPE blanks, laboratory control samples (LCS), matrix spikes, and sample triplicates. Blanks and LCS were composed of Type I water. LCS and matrix spikes for HMX, RDX, and DNAN were spiked at 4  $\mu\text{g/L}$  in aliquots for SPE, and those for NTO were spiked at 0.5 mg/L in direct aqueous aliquots. LCS, matrix spikes, and triplicates were performed at a frequency of one set every 20 samples.

## 2.4 Sample analysis

Concentrations of energetic compounds in each sample fraction were analyzed by high-performance liquid chromatography (HPLC; Agilent 1260 series) following methods established by M.E. Walsh (2016). The compounds HMX, RDX, and DNAN were quantified on a C8 column (Waters NovaPak, 150  $\times$  3.9 mm, 4  $\mu\text{m}$ ), eluted with 15/85 (v/v) isopropanol/water at 1.4 mL/min, and detected by absorption at 230 nm for HMX and RDX and at 295 nm for DNAN. The polar compound NTO was quantified on a porous graphitic carbon column (Thermo Scientific Hypercarb, 150  $\times$  4.6 mm, 5  $\mu\text{m}$ ), eluted with 3/1 (v/v) acetonitrile/water and 0.1% trifluoroacetic acid at 1.5 mL/min, and detected by absorption at 321 nm.

Sample concentrations were confirmed by secondary HPLC methods using different separation mechanisms and elution orders. HMX, RDX, and DNAN were confirmed on a cyano column (Agilent Poroshell 120 EC-CN, 150  $\times$  3.0 mm, 2.7  $\mu\text{m}$ ) eluted with 50/50 (v/v) methanol/water at 0.6 mL/min. NTO was confirmed on a hydrophilic interaction column (Waters XBridge BEH Amide, 100  $\times$  4.6 mm, 2.5  $\mu\text{m}$ ) eluted with 97/3 (v/v) acetonitrile/water and 0.1% trifluoroacetic acid at 0.5 mL/min. Detection wavelengths were the same as for the primary methods. Appendix A and Appendix B show detailed chromatographic conditions, chromatograms, and spectra.

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\* Volume per volume.

Quantification used seven-point external calibration curves made from commercially available standards from Restek (HMX and RDX), Absolute Standards (DNAN), and AccuStandard (NTO). Initial calibration verification (ICV) used standards from separate lots (HMX and RDX) and from neat materials (DNAN and NTO). Acceptance of the ICV and the continuing calibration verification (CCV), which was run every 10 samples, was recovery within  $\pm 20\%$ .

## **2.5 Deposition-rate estimation**

Energetic compound masses in plumes were determined by extrapolation of sample masses using the surface area of the MIS sample (determined by the scoop size and number of increments) and the DU area (measured by GPS). Deposition rate was calculated by dividing the plume compound mass (summed ITP and OTP masses) by the number of cartridges represented by the plume. Uncertainty in the deposition-rate estimate was calculated by relative standard deviation (RSD; standard deviation [SD] divided by average) of triplicate MIS samples from each ITP DU.

### 3 Results and Discussion

#### 3.1 Observations and sampled deposition plumes

Postdetonation clouds were generally similar between all cartridges and characterized by a dark component that settled out quickly and a fainter light component that drifted off area along the wind direction (Figure 3; Appendix B). The darker component was assumed in this study to comprise carbon soot and the majority of solid energetic residues and the lighter component predominantly condensed water vapor.

Figure 3. Two examples of postdetonation residue, soot, and gas clouds from live fire of 81 mm IMX-104 (M889A4) cartridges.



Figure 4 shows the impact locations and plume areas, labeled sequentially in order of day of firing and sampling. All plumes covered separate single-cartridge detonations, with the sole exception of Plume 2, which covered two intentionally overlapping cartridge detonations. The plume shapes surrounding the impact location provide a general indication of predominant wind direction and speed.

The ITP plume areas (Table 1) were within the range observed during previous command-detonation tests of 81 mm IMX-104 mortar cartridges (190–890 m<sup>2</sup>; M. R. Walsh, et al. 2015; M. R. Walsh, Bigl, et al. 2018; M. R. Walsh, Gullet, et al. 2018). The number of increments collected from each plume met or exceeded the target range of 70 to 100, and all samples exceeded the minimum of 40 increments (M. R. Walsh et al. 2012).

Figure 4. Plume locations and areas in the target region measured by GPS.



Table 1. GPS-measured areas and Multi-Increment Sampling (MIS) increments collected for each plume. Plume 2 covered residues from two cartridges.

Plume	ITP		OTP 0-3 m		OTP 3-6 m	
	Area (m <sup>2</sup> )	Increments ( <i>n</i> )	Area (m <sup>2</sup> )	Increments ( <i>n</i> )	Area (m <sup>2</sup> )	Increments ( <i>n</i> )
1	540	89-121	288	104	345	99
2	843	119-140	344	73-92	389	95-108
3	324	87-102	239	80-88	273	76-80
4	426	81-125	273	89	314	95
5	451	93-113	275	91	301	96
6	568	99-105	323	91-97	366	73-89
7	711	93-130	370	76	391	75
8	672	92-118	395	90	403	83
9	617	96-150	321	101	346	87
10	445	90-91	294	79-96	332	86-88
11	492	90-142	314	108	342	88
12	553	95-145	380	95	386	85
13	526	101-110	305	98	333	85

Warm weather in the days leading up to the tests flooded parts of the impact area and constrained target locations to the higher-elevation area in the southwest of the target region for Plumes 1–5 (Figure 4). Temperatures during firing and sampling of Plumes 1 through 5 were around  $-1^{\circ}\text{C}$  with a light snow and low to no wind. Subsequent cold weather refroze the remainder of the target region and, along with fresh snow (approximately 1–3 cm), produced ideal conditions for Plumes 6–13 (Figure 5). Temperatures during these later plumes were between  $-9^{\circ}\text{C}$  and  $-6^{\circ}\text{C}$  with generally low wind but some intermittent gusts.

Figure 5. An example sample increment from Plume 10, showing the thin snow layer and subsurface ice typical of Plumes 6–13.



### 3.2 Quality control

Background samples provide a measure of potential preexisting compounds in the surface snow of the target area. Backgrounds 1 and 2 were collected before any firing, Background 3 was collected after firing of Plumes 1 through 5, and Background 4 was collected after all rounds had been fired. Concentrations of IMX-104 compounds were below detection in all background samples, except for Background 4, which had NTO concentrations right at the detection limit (Table 2). The very low concentration of NTO in this sample could not be confirmed by the secondary analysis method, but if true indicates slight distribution of this compound outside the OTP 3–6 m DUs, likely from nearby Plumes 6, 7, or 8. Overall, these background results support the assumption that the target-area

snow was free of preexisting IMX-104 compounds and that the sampled plumes represent deposition from the tested cartridges only.

**Table 2. Energetic compound masses per area in background samples.** Listed sample detection limits are a function of analytical detection limits, decision unit (DU) size, number of increments, and sample volume.

Background Sample	Mass per Area (mg/m <sup>2</sup> )			
	HMX	RDX	DNAN	NTO
1	<0.0011	<0.0011	<0.0011	<0.037
2	<0.0011	<0.0011	<0.0011	<0.035
3	<0.0004	<0.0004	<0.0004	<0.008
4	<0.0004	<0.0004	<0.0004	0.011

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 0.6%–7% (Appendix A) and met the success criteria of less than 20%. Matrix spikes had recoveries of 75%–110% (Appendix A), which met the success criteria of 70%–130%, except for one matrix spike that had enhanced recoveries of RDX and DNAN. This exception was considered an anomaly in the context of otherwise quantitative spike recovery and excellent overall sample replicate precision. Matrix spike relative percent differences were 0%–14% that met, without exception, the success criteria of less than 20%. Three of the four LCS had recoveries (93%–100%) that met the success criteria of 70%–130%. The remaining LCS had recoveries at (70% for HMX) or just below (68% for RDX, 69% for DNAN) the lower recovery limit, which indicated an anomaly during SPE loading or eluting.

### 3.3 Estimates of residue deposition rates

All IMX-104 analytes were detected in all thirteen plumes, presented in Table 3. As observed in previous tests of IMX-104 munitions, NTO was the dominant residual compound by two orders of magnitude. Masses of the other compounds followed a consistent trend of DNAN > RDX > HMX. The recovered HMX could be derived from the filler (impurity in RDX), auxiliary booster, or fuze booster. Only NTO was frequently detected in the OTP decision units and at masses that were negligible compared to the ITPs, which supports complete sampling of deposited residues. Transformation products of DNAN (methoxy nitrophenols, methoxy nitroanilines, and dinitrophenol) were not observed in any of the samples, indicating no degradation of this compound.

**Table 3. Average estimated energetic residue deposition rates within each plume's DUs. Values in parentheses are standard deviations of triplicate MIS estimates. Note that Plume 2 includes two cartridge detonations.**

Plume	DU Type	Residues (mg/plume)			
		HMX	RDX	DNAN	NTO
1	ITP	6.4 (0.7)	80 (9)	240 (30)	11000 (2000)
	OTP 0-3	0.2	0.5	3.4	108
	OTP 3-6	<0.1	0.3	0.8	28
2	ITP	2.5 (0.3)	30 (10)	90 (20)	8000 (2000)
	OTP 0-3	<0.2	0.5 (0.8)	<0.2	44 (4)
	OTP 3-6	<0.1	<0.1	<0.1	28 (4)
3	ITP	1.7 (0.4)	22 (2)	48 (8)	10000 (600)
	OTP 0-3	<0.1	0.1 (0.0)	0.6 (0.1)	66 (9)
	OTP 3-6	0.3 (0.1)	<0.2	<0.2	18 (4)
4	ITP	1.3 (0.2)	9.6 (0.5)	31 (7)	4700 (100)
	OTP 0-3	<0.1	0.2	1.4	110
	OTP 3-6	0.4	<0.2	0.4	47
5	ITP	3.1 (0.4)	23 (3)	100 (10)	4500 (600)
	OTP 0-3	0.5	1.5	10	130
	OTP 3-6	0.3	<0.2	0.8	24
6	ITP	3.0 (0.2)	31 (3)	79 (9)	7600 (700)
	OTP 0-3	<0.2	0.2 (0.1)	0.8 (0.4)	50 (20)
	OTP 3-6	0.3 (0.1)	<0.2	0.2 (0.0)	16 (3)
7	ITP	1.8 (0.3)	23 (4)	42 (9)	12200 (300)
	OTP 0-3	<0.2	<0.2	0.4	47
	OTP 3-6	0.3	<0.2	0.3	31
8	ITP	0.8 (0.1)	7 (1)	17 (2)	5200 (800)
	OTP 0-3	<0.2	<0.2	0.2	32
	OTP 3-6	0.2	<0.2	<0.2	7
9	ITP	1.7 (0.1)	16.8 (0.7)	47 (2)	6600 (400)
	OTP 0-3	<0.2	0.2	1.0	95
	OTP 3-6	0.2	<0.2	0.3	35
10	ITP	0.9 (0.1)	8 (1)	14.3 (0.9)	5800 (800)
	OTP 0-3	<0.2	<0.2	0.2 (0.0)	30 (8)
	OTP 3-6	<0.2	<0.2	<0.2	10 (10)
11	ITP	2.0 (0.3)	25 (3)	50 (10)	9000 (1000)
	OTP 0-3	<0.1	0.1	0.5	30
	OTP 3-6	0.1	<0.1	<0.1	11
12	ITP	2.5 (0.6)	22.8 (0.4)	60 (8)	7600 (800)
	OTP 0-3	<0.2	<0.2	0.4	13
	OTP 3-6	<0.3	<0.3	<0.3	10
13	ITP	1.2 (0.2)	14 (3)	23 (3)	10000 (2000)
	OTP 0-3	<0.2	<0.2	0.6	78
	OTP 3-6	<0.2	<0.2	<0.2	21

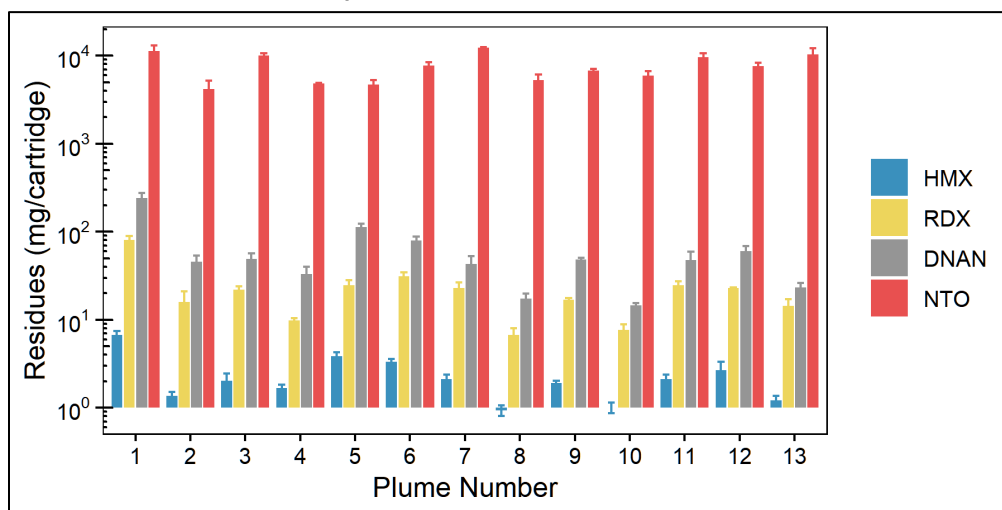
Table 4 and Figure 6 summarize the per-cartridge residue deposition rates and uncertainties in each plume. The total energetic mass in each plume is almost entirely a reflection of the NTO mass due to the compound's predominance. Uncertainty associated with each plume's estimate is shown as RSDs in Table 4 and is derived from the triplicate samples taken from each plume's ITP. The low RSDs associated with each compound's estimate within individual plumes (2%–32%, mean 13%) indicate relatively minimal uncertainty related to sampling and analysis. Uncertainty in deposition-rate estimates among all plumes includes variability related to cartridge-to-cartridge differences in detonation and plume-surface recovery of produced residues. This uncertainty is relatively high for HMX, RDX, and DNAN (65%–96%) but more reasonable if Plume 1 is excluded (41%–56%), though there are no unusual observations from Plume 1 to support its exclusion. The overall uncertainty characterized by NTO and total residues RSD (35%) meets the success criteria of less than 50% and bettered previous command detonation tests of the study munition (RSDs: 46% AFS; 36% CFS 12 g; 45% CFS 18 g; see M. R. Walsh, et al. 2015; M. R. Walsh, Bigl, et al. 2018; M. R. Walsh, Gullet, et al. 2018). Overall, the precision in the live-fire data, both within individual plumes and among all plumes, provides confidence that the live-fire deposition rates are broadly representative of the munition.

Table 4. Average estimated live-fire energetic residue deposition rates and precision.

Plume	Residues (mg/cartridge)					RSD (%)				
	HMX	RDX	DNAN	NTO	Total	HMX	RDX	DNAN	NTO	Total
1	6.7	80	240	11000	12000	11	11	13	16	16
2	1.4	16	46	4000	4000	11	32	17	25	25
3	2.0	22	49	10000	10000	19	10	16	6	6
4	1.7	9.9	33	4800	4900	9	5	21	2	2
5	3.9	25	110	4700	4800	10	14	9	12	12
6	3.3	31	80	7700	7800	7	11	11	9	9
7	2.1	23	43	12300	12300	12	16	22	2	2
8	0.9	7	17	5300	5300	14	19	14	16	16
9	1.9	17	48	6700	6800	7	4	5	6	6
10	1.0	8	14.5	5900	5900	14	15	6	13	13
11	2.1	25	50	10000	10000	13	12	23	12	12
12	2.7	22.8	60	7600	7700	24	2	14	10	10
13	1.2	14	23	10000	10000	13	20	12	19	19
Ave.	2.4	23	63	7700	7800	—	—	—	—	—

SD	1.6	19	60	2700	2800	—	—	—	—	—
RSD (%)	65	81	96	35	35	—	—	—	—	—

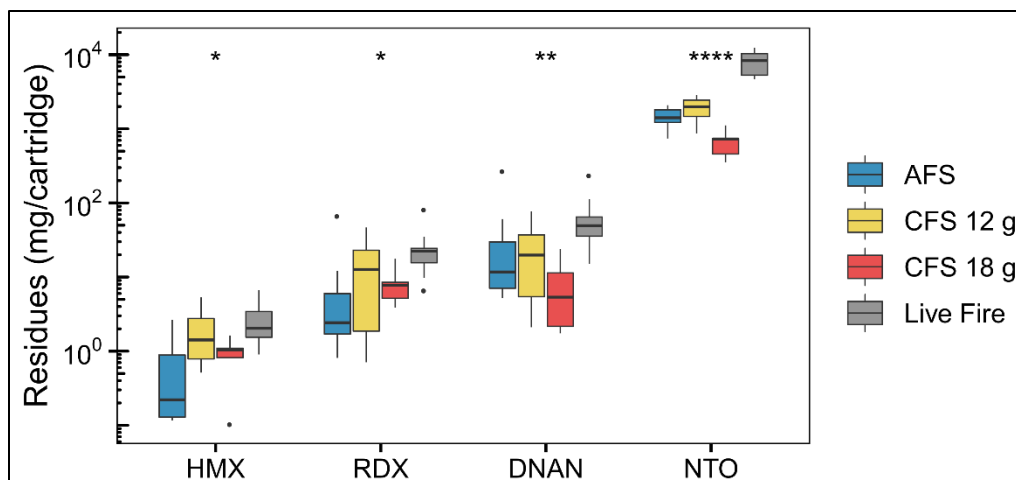
Figure 6. Average estimated energetic residue deposition for each plume. *Error bars* represent one standard deviation.



### 3.4 Comparison with command detonations

Figure 7 compares the live-fire deposition rates for the M889A4 with previously determined command-detonation deposition rates for the M821A3. The boxplots represent the data distribution of each compound for each fuze type. Nonparametric statistical tests (i.e., Mann-Whitney test) were used due to limited sample sizes and nonnormal data distributions for some fuze types (Appendix B). Relative to the command-detonation tests, live-fire deposition rates of HMX, RDX, and DNAN are generally similar if not slightly higher. However, NTO deposition rates are consistently and significantly greater by live fire than by command detonation using any of the fuze simulators. The mean NTO deposition rate by live fire is over 4 times greater than command-detonation tests using the AFS and the CFS with 12 g booster. The high booster mass used in the 18 g CFS is likely unrepresentative of the munition but provides end-member data for potentially overdriven detonations.

Figure 7. The 81 mm IMX-104 deposition rates determined by live fire compared with previous command-detonation tests with the Armaments Center fuze simulator (AFS) and the Cold Regions Research and Engineering Laboratory fuze simulator (CFS; M.R. Walsh et al. 2015; M. R. Walsh, Bigl, et al. 2018; M. R. Walsh, Gullet, et al. 2018). *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. *Asterisks* represent Kruskal-Wallis rank sum test significance levels (\*  $p \leq 0.05$ ; \*\*  $p \leq 0.01$ ; \*\*\*\*  $p \leq 0.0001$ ).

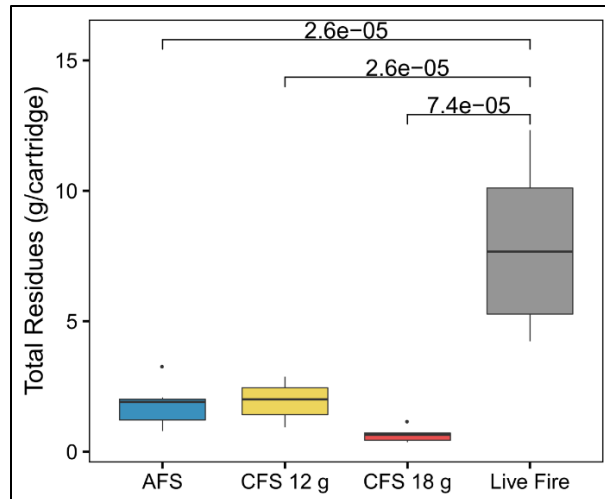


The large differences in NTO deposition rates drive statistical tests to confidently reject the null hypothesis that total residue deposition is not significantly different between each fuze simulator and live fire (Table 5 and Figure 8). However, deposition rates for the other IMX-104 compounds compare more closely. DNAN deposition rates are not significantly different between live fire and either the AFS or 12 g CFS at the 95% confidence level, indicated by  $p$ -values greater than 0.05. Live-fire deposition rates of HMX and RDX are significantly different with the AFS but not with the 12 g CFS, though these compounds could have derived from the fuze boosters and not the IMX-104 main filler.

Table 5. Results of two-sided Mann-Whitney rank sum tests between live-fire and prior command-detonation tests for the M889A4/M821A3 81 mm IMX-104 mortar munition.

Comparison	$p$ -Value				
	HMX	RDX	DNAN	NTO	Total
Live Fire—AFS	0.026	0.014	0.067	$2.6 \times 10^{-5}$	$2.6 \times 10^{-5}$
Live Fire—CFS 12 g	0.503	0.275	0.097	$2.6 \times 10^{-5}$	$2.6 \times 10^{-5}$
Live Fire—CFS 18 g	0.007	0.007	$5.2 \times 10^{-4}$	$7.4 \times 10^{-5}$	$7.4 \times 10^{-5}$

Figure 8. Comparison of total residue deposition rates for the 81 mm IMX-104 between the different tested fuze simulators and live fire. The  $p$ -values for Mann-Whitney tests are shown above comparison group brackets.



Overall, these comparisons show that command detonations of the 81 mm IMX-104 mortar munition significantly underestimated NTO residues as they are produced during live fire, while the compounds DNAN, RDX, and HMX were estimated with relative accuracy. Three main hypotheses for the observed differences in NTO are (1) overdriving of the detonation train during previous command-detonation tests, (2) enhanced residue recovery in a nose-down orientation during impact from live fire, or (3) manufacturing or production differences in studied cartridges.

For practicality in setup, tests with both the AFS and CFS used blasting caps to initiate the detonations, and the explosive masses used in these caps are substantially greater than masses used in the issued fuze's safe and arm (S&A) device. Interestingly, the same fuze simulation setups were applied to the 60 mm IMX-104 munition with no significant difference in total residue production between the AFS and live fire (Beal et al. 2022). It is unclear whether the blasting cap's increased explosive mass could affect filler mass consumption. While an effect of booster mass on residue production is clearly apparent in comparing results from the CFS with 12 g and 18 g C-4, the underestimation in NTO deposition is still observed with the AFS whose booster matches the charge in the issued fuze. These results suggest a potential explanatory variable in the initiation charge simulation but not the booster charge. Future command detonations may prove more representative if initiated with an exploding bridge wire detonator containing a similar charge mass to the fuze S&A device.

Cartridge orientation is another key difference between the previous command-detonation tests and live fire. In Task 1, the nose-down angle during detonation from live fire of the 60 mm IMX-104 cartridge was hypothesized to have potentially led to greater retention of fine particles on the snow surface and corresponding increased live-fire RDX and DNAN deposition rates (Beal et al. 2022). As NTO is present as relatively large crystals in IMX-104, which would tend to deposit locally if unconsumed, it seems unlikely that cartridge orientation alone would explain the greater NTO residues produced by live fire of the 81 mm IMX-104 munition. Examination of compound phase partitioning immediately upon sample melting and filtering (Table 6), which is a proxy for particle size, reveals no major differences for any of the compounds between fuze types. Nose-down command detonations could not be achieved during this study due to weather, scheduling, and ammunition supply constraints. Future command-detonation tests should consider nose-down positioning directly above the sampling surface.

**Table 6. Initial proportions of energetic compounds in the aqueous phase from 81 mm IMX-104 tests. Results are the average of all ITP samples, with standard deviation in parentheses.**

Fuze	Aqueous Fraction (%)			
	HMX	RDX	DNAN	NTO
Live Fire	37 (8)	88 (4)	40 (10)	97 (2)
AFS	20 (20)	80 (20)	20 (5)	100 (0)
CFS 12 g	10 (20)	70 (10)	10 (6)	99 (2)
CFS 18 g	0 (0)	50 (20)	10 (10)	90 (10)

The final hypothesis to explain observed differences in residue deposition rates is potential formulation-specific differences stemming from manufacturing variations. The excellent agreement between the CFS tests (pre-production cartridges) and the AFS tests (production cartridges) suggests this explanation may be unlikely; however, minor differences in manufacturing, possibly related to NTO crystal size (Smith and Cliff 1999), could have downstream effects on residue production.

Unlike the Task 1 study with the 60 mm IMX-104 munition, the success criterion of no significant difference in total residue deposition rates between live fire and command detonation was not met for the 81 mm IMX-104 munition. However, the reproduction of each IMX-104 compound's deposition rates to the same order of magnitude as live fire (Table 7)

supports a coarser-scale representation of this munition's residues. In context of trends observed across all previously tested munition types, where postdetonation residues ranged from less than 1 mg to over 100 g per round (M. R. Walsh, Bigl, et al. 2018), the command detonations of this 81 mm IMX-104 munition provide sufficiently accurate data to inform environmental assessment.

Table 7. Summary of mean deposition rates from all live-fire and command-detonation tests of the study munitions.

Munition Type	Fuze	Mean Residues (mg/cartridge)					Cartridges ( <i>n</i> )
		HMX	RDX	DNAN	NTO	Total	
81 mm IMX-104	Live Fire	2	20	60	8000	8000	14
	AFS	1	10	50	1700	1800	7
	CFS 12 g	2	20	30	1900	1900	7
	CFS 18 g	0.9	8	8	600	700	6
60 mm IMX-104	Live Fire	1.9	12	34	3800	3800	11
	AFS	0.4	2	7	3800	3800	7
	CFS 12 g	2	4	5	2200	2200	7

Given the methodological deviations imposed in previous command-detonation tests (i.e., blasting cap initiator and nose-up orientation), the general accuracy in their representations of live-fire residues is remarkable. The observed contrasting differences in compound-specific representation by the two different munitions highlight room for improvement in the command-detonation setups. Potential follow-on tests could include examining the effect of initiation with a representative exploding bridge wire, the effect of cartridge orientation with nose-down command detonations, and the effect of manufacturing differences through multiple lot testing. The results of such follow-on tests would further refine the current understanding of residue production and more confidently transition the residue testing method to stakeholders.

## 4 Conclusions

The results of this study with the 81 mm IMX-104 munition complement the results of the previous 60 mm IMX-104 study in support of validating command detonation for residues testing. While the success criteria were not met for all residue compounds across the two studied munitions, the results highlight the practical limits on residue representation to be expected when using command detonation. In the broader context of trends observed in residue production by other munitions, the ability shown here for command detonation to predict actual live-fire deposition rates within approximately the correct order of magnitude demonstrates the utility this technology can have for new, in-development munitions. Integrating this technology into the testing process for new munitions would aid in identifying environmental impacts that can inform acquisition decisions and range management strategies.

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## Appendix A: Supplementary Tables

Table A-1. Results of the processing-quality-control samples. All were below the method detection limit. NTO was not analyzed in solid-phase extraction (SPE) samples.

Quality-Control Sample	Concentration (mg/L)			
	HMX	RDX	DNAN	NTO
Prefiltering Blanks ( <i>n</i> = 2)	<0.0001	<0.0001	<0.0001	<0.004
Midfiltering Blanks ( <i>n</i> = 2)	<0.0001	<0.0001	<0.0001	<0.004
Postfiltering Blanks ( <i>n</i> = 2)	<0.0001	<0.0001	<0.0001	<0.004
SPE Blanks ( <i>n</i> = 4)	<0.0001	<0.0001	<0.0001	—

Table A-2. Precision for inside-the-plume (ITP) and outside-the-plume (OTP) aqueous-sample triplicates. (RSD is the relative standard deviation.)

Sample ID	Type	Mean Concentration (µg/L)				RSD (%)			
		HMX	RDX	DNAN	NTO	HMX	RDX	DNAN	NTO
22FRA018	Plume 2 ITP	0.546	11.2	19.7	3260	0.6	0.7	0.9	0.6
22FRA040	Plume 3 OTP 0–3 m	<0.1	0.17	0.84	90.6	—	7	4	0.2
22FRA060	Plume 3 ITP	0.37	15.2	14.1	7890	4	3	2	0.8
22FRA082	Plume 11 ITP	0.41	11.2	8.5	5470	3	2	5	0.6

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 for 22FRA040 and 5 mg/L for 22FRA018, 22FRA060, and 22FRA082.

Quality-Control Sample ID	Recovery (%)				Relative Percent Difference (%)			
	HMX	RDX	DNAN	NTO	HMX	RDX	DNAN	NTO
22FRA018 MS	101	95	75	83	4	2	0	2
22FRA018 MSD	96	86	76	87	—	—	—	—
22FRA040 MS	101	102	110	98	4	6	12	2
22FRA040 MSD	97	96	94	101	—	—	—	—
22FRA060 MS	97	109	83	82	2	2	2	2
22FRA060 MSD	99	98	92	87	—	—	—	—
22FRA082 MS	111	154	135	80	11	14	8	5
22FRA082 MSD	98	99	110	90	—	—	—	—

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	93	95	95	96
LCS-2	99	100	100	96
LCS-3	70	68	69	100
LCS-4	98	95	96	99

Table A-5. Chromatographic conditions. IPA is isopropanol, ACN is acetonitrile, MeOH is methanol, RT is retention time, and  $\lambda$  is the detection wavelength.

Column	Mobile Phase (v/v)	Flow Rate (mL/min)	Temperature (°C)	Injection Volume (µL)	Injection Matrix (v/v)	RT/ $\lambda$ (min/nm)
NovaPak C8 150 × 3.9 mm, 4 µm	15/85 IPA/Water	1.4	30	100	1/3 ACN/Water	HMX: 1.5/230 RDX: 2.8/230 DNAN: 6.7/295
Hypercarb 150 × 4.6 mm, 5 µm	3/1 ACN/Water 0.1% TFA	1.5	28	100	3/1 ACN/Water	NTO: 1.7/321
Poroshell 120 EC-CN 150 × 3.0 mm, 2.7 µm	50/50 MeOH/Water	0.6	30	10	1/3 ACN/Water	HMX: 6.7/230 RDX: 4.1/230 DNAN: 2.9/295
XBridge BEH Amide 100 × 4.6 mm, 2.5 µm	97/3 ACN/Water 0.1% TFA	0.5	28	20	3/1 ACN/Water	NTO: 3.3/320

## Appendix B: Supplemental Figures

Figure B-1. Example 1 of a postdetonation cloud immediately following impact of an 81 mm IMX-104 cartridge from this study.



Figure B-2. Example 2 of a postdetonation cloud immediately following impact of an 81 mm IMX-104 cartridge from this study.



Figure B-3. Example 3 of a postdetonation cloud immediately following impact of an 81 mm IMX-104 cartridge from this study.



Figure B-4. Example 4 of a postdetonation cloud immediately following impact of an 81 mm IMX-104 cartridge from this study.



Figure B-5. Example 5 of a postdetonation cloud immediately following impact of an 81 mm IMX-104 cartridge from this study.



Figure B-6. Progression (*top to bottom*) of a postdetonation cloud immediately following impact of an 81 mm IMX-104 cartridge from this study.



Figure B-7. Example chromatography and UV spectra of a standard and samples using the C8 primary method for HMX, RDX, and DNAN.

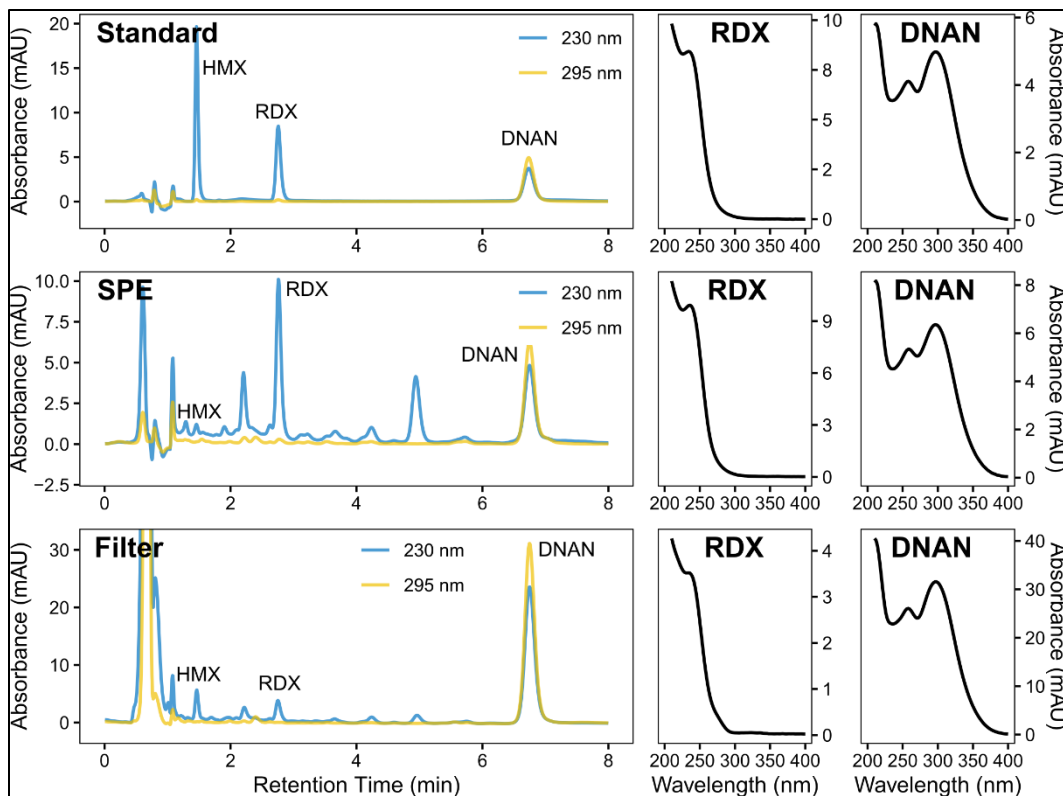


Figure B-8. Example chromatography and UV spectra of a standard and samples using the Hypercarb primary method for NTO.

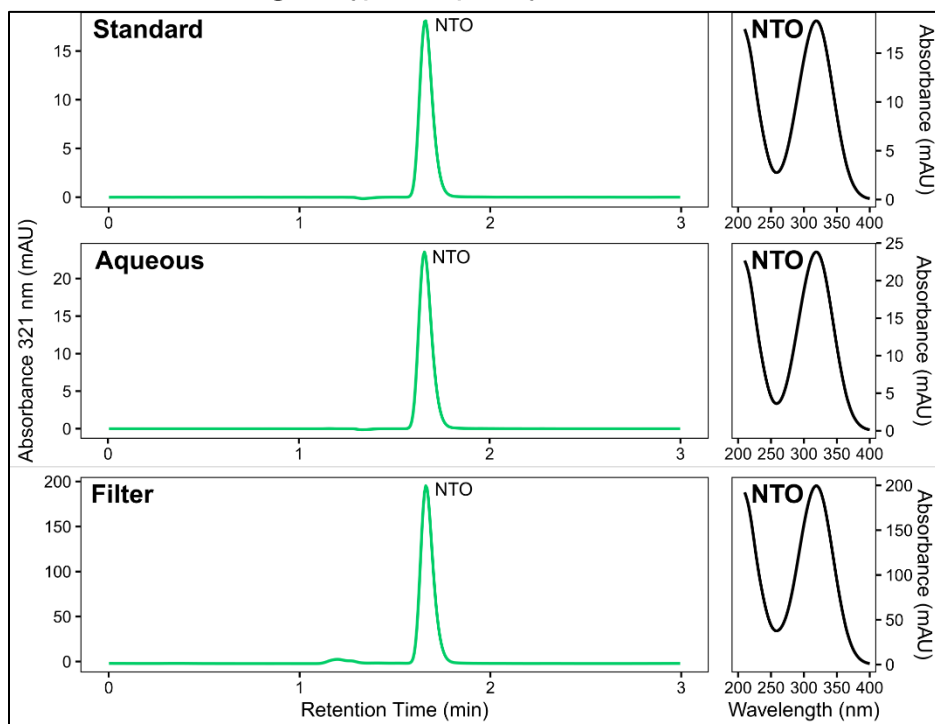


Figure B-9. Example chromatography and UV spectra of a standard and samples using the cyano confirmation method for HMX, RDX, and DNAN.

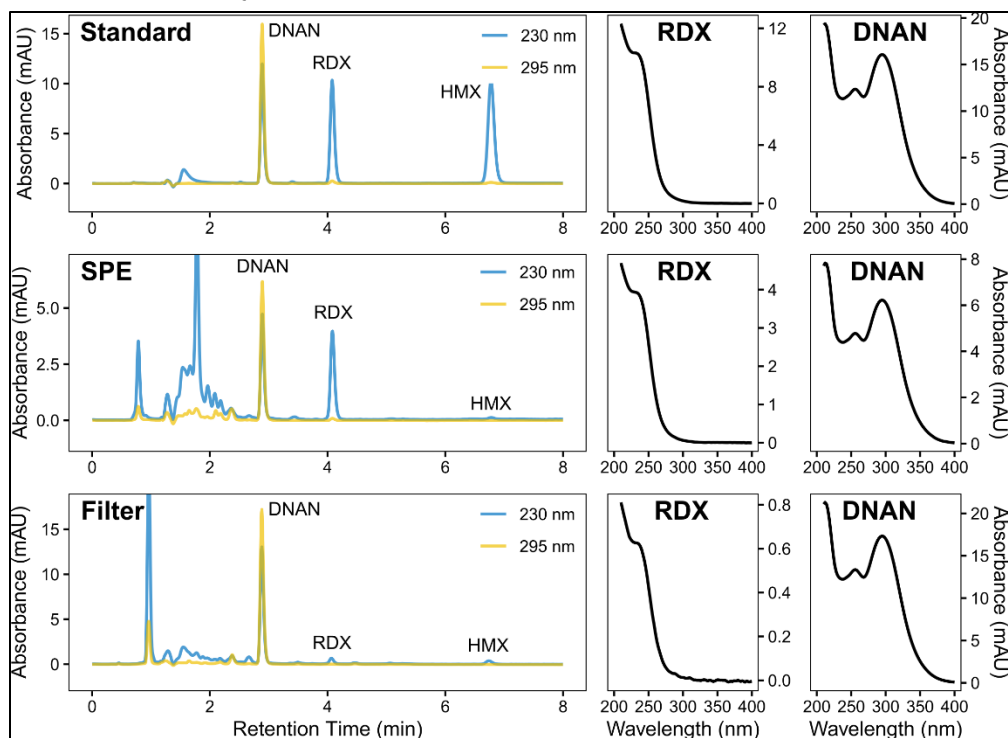


Figure B-10. Example chromatography and UV spectra of a standard and samples using the hydrophilic interaction confirmation method for NTO.

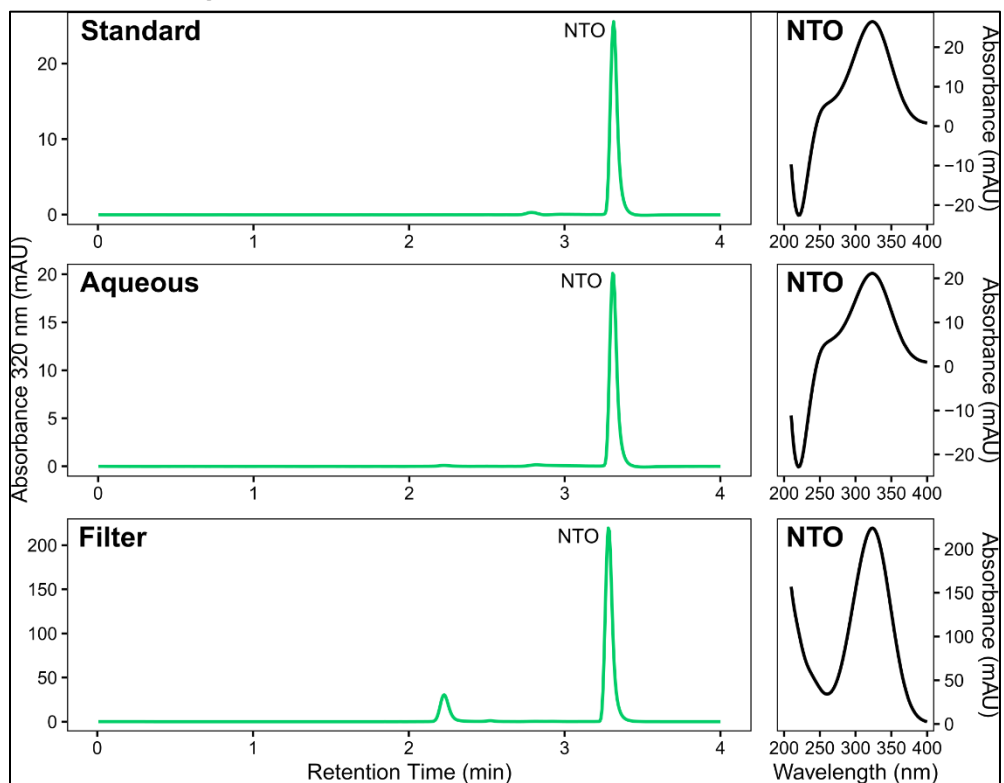
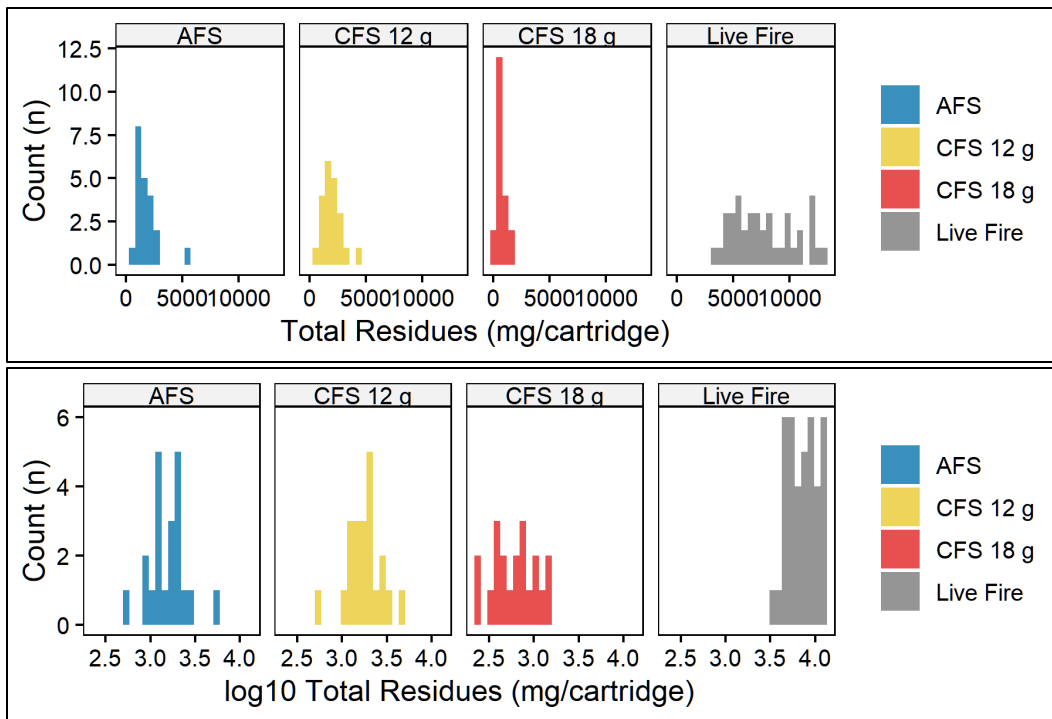


Figure B-11. Histograms of untransformed (*top*) and log10-transformed (*bottom*) total residue deposition rates by fuze type.



## Appendix C: Complete Dataset

Table C-1. Sampling and processing data for the 2022 live-fire test of 81 mm IMX-104 cartridges. (NA = not applicable.)

Sample ID	Type	Plume Area (m <sup>2</sup> )	Increments ( <i>n</i> )	Filtrate Vol (mL)	SPE Factor	Filter Extract (mL)
22FRA001	Background 1	1543.7	98	8958	100	20.0
22FRA002A	Background 2	1577.7	117	8000	100	30.0
22FRA002B	Background 2	1577.7	NA	2110	100	NA
22FRA003	Plume 1 OTP 3-6	344.9	99	1978	100	20.0
22FRA004	Plume 2 OTP 3-6	389.0	105	2010	100	20.0
22FRA005	Plume 2 OTP 3-6	389.0	95	1455	100	20.0
22FRA006	Plume 2 OTP 3-6	389.0	108	1935	100	20.0
22FRA007	Plume 1 OTP 0-3	287.8	104	2540	100	20.0
22FRA008	Plume 2 OTP 0-3	344.1	73	1265	100	20.0
22FRA009	Plume 2 OTP 0-3	344.1	73	1748	100	20.0
22FRA010	Plume 2 OTP 0-3	344.1	92	1505	100	20.0
22FRA013	Plume 1 ITP	540.5	121	5075	100	30.0
22FRA014	Plume 1 ITP	540.5	89	3280	100	30.0
22FRA015	Plume 1 ITP	540.5	94	2785	100	20.0
22FRA016	Plume 2 ITP	843.4	119	2410	100	30.0
22FRA017	Plume 2 ITP	843.4	140	5630	100	50.0
22FRA018	Plume 2 ITP	843.4	122	3270	100	50.0
22FRA019	Background 3	1674.0	97	1960	100	20.0
22FRA020	Background 4	1654.9	99	2590	100	20.0
22FRA021	Plume 3 OTP 3-6	273.2	80	2595	100	30.0
22FRA022	Plume 3 OTP 3-6	273.2	76	2230	100	20.0
22FRA023	Plume 3 OTP 3-6	273.2	79	2550	100	20.0
22FRA024	Plume 4 OTP 3-6	313.6	95	3475	100	20.0
22FRA025	Plume 5 OTP 3-6	300.9	96	3430	100	20.0
22FRA026	Plume 6 OTP 3-6	365.6	87	2330	100	20.0
22FRA027	Plume 6 OTP 3-6	365.6	73	2390	100	20.0
22FRA028	Plume 6 OTP 3-6	365.6	89	2890	100	20.0
22FRA029	Plume 7 OTP 3-6	390.8	75	2630	100	20.0
22FRA030	Plume 8 OTP 3-6	403.3	83	2810	100	20.0
22FRA031	Plume 9 OTP 3-6	346.2	87	3410	100	20.0
22FRA032	Plume 10 OTP 3-6	332.0	86	3010	100	20.0
22FRA033	Plume 10 OTP 3-6	332.0	87	2900	100	20.0
22FRA034	Plume 10 OTP 3-6	332.0	88	2855	100	30.0
22FRA035	Plume 11 OTP 3-6	342.0	88	3115	100	20.0
22FRA036	Plume 12 OTP 3-6	386.2	85	2600	100	30.0

Table C-1 (cont.). Sampling and processing data for the 2022 live-fire test of 81 mm IMX-104 cartridges. (NA = not applicable.)

Sample ID	Type	Plume Area (m <sup>2</sup> )	Increments ( <i>n</i> )	Filtrate Vol (mL)	SPE Factor	Filter Extract (mL)
22FRA037	Plume 13 OTP 3-6	333.4	85	2410	100	20.0
22FRA038	Plume 3 OTP 0-3	238.8	88	2390	100	20.0
22FRA039	Plume 3 OTP 0-3	238.8	87	2215	100	20.0
22FRA040	Plume 3 OTP 0-3	238.8	80	2810	100	20.0
22FRA041	Plume 4 OTP 0-3	273.4	89	2425	100	20.0
22FRA042	Plume 5 OTP 0-3	275.1	91	3215	100	20.0
22FRA043	Plume 6 OTP 0-3	322.5	94	2580	100	20.0
22FRA044	Plume 6 OTP 0-3	322.5	91	2420	100	20.0
22FRA045	Plume 6 OTP 0-3	322.5	97	2250	100	20.0
22FRA046	Plume 7 OTP 0-3	369.6	76	2210	100	20.0
22FRA047	Plume 8 OTP 0-3	395.3	90	2200	100	20.0
22FRA048	Plume 9 OTP 0-3	321.0	101	2850	100	20.0
22FRA049	Plume 10 OTP 0-3	293.5	80	2000	100	20.0
22FRA050	Plume 10 OTP 0-3	293.5	96	2730	100	30.0
22FRA051	Plume 10 OTP 0-3	293.5	79	2150	100	20.0
22FRA052	Plume 11 OTP 0-3	313.8	108	2960	100	20.0
22FRA053	Plume 12 OTP 0-3	379.9	95	2770	100	20.0
22FRA054	Plume 13 OTP 0-3	304.6	98	3185	100	20.0
22FRA058	Plume 3 ITP	323.7	87	3070	100	30.0
22FRA059	Plume 3 ITP	323.7	88	3225	100	30.0
22FRA060	Plume 3 ITP	323.7	102	3820	100	30.0
22FRA061	Plume 4 ITP	425.7	92	2665	100	30.0
22FRA062	Plume 4 ITP	425.7	81	2725	100	30.0
22FRA063	Plume 4 ITP	425.7	125	4520	100	30.0
22FRA064	Plume 5 ITP	451.3	113	3860	100	30.0
22FRA065	Plume 5 ITP	451.3	93	3140	100	30.0
22FRA066	Plume 5 ITP	451.3	106	3835	100	30.0
22FRA067	Plume 6 ITP	567.7	104	3700	100	30.0
22FRA068	Plume 6 ITP	567.7	99	3810	100	30.0
22FRA069	Plume 6 ITP	567.7	105	3990	100	30.0
22FRA070	Plume 7 ITP	711.4	130	5955	100	40.0
22FRA071	Plume 7 ITP	711.4	96	4165	100	30.0
22FRA072	Plume 7 ITP	711.4	93	4160	100	30.0
22FRA073	Plume 8 ITP	672.3	118	3600	100	30.0
22FRA074	Plume 8 ITP	672.3	95	3190	100	30.0
22FRA075	Plume 8 ITP	672.3	92	2780	100	30.0
22FRA076	Plume 9 ITP	616.6	150	6305	100	40.0

Table C-1 (cont.). Sampling and processing data for the 2022 live-fire test of 81 mm IMX-104 cartridges. (NA = not applicable.)

Sample ID	Type	Plume Area (m <sup>2</sup> )	Increments ( <i>n</i> )	Filtrate Vol (mL)	SPE Factor	Filter Extract (mL)
22FRA077	Plume 9 ITP	616.6	96	3650	100	30.0
22FRA078	Plume 9 ITP	616.6	117	5210	100	30.0
22FRA079	Plume 10 ITP	444.7	90	3520	100	30.0
22FRA080	Plume 10 ITP	444.7	91	3170	100	40.0
22FRA081	Plume 10 ITP	444.7	90	3085	100	30.0
22FRA082	Plume 11 ITP	492.1	142	5010	100	40.0
22FRA083	Plume 11 ITP	492.1	90	3080	100	20.0
22FRA084	Plume 11 ITP	492.1	99	3810	100	30.0
22FRA085	Plume 12 ITP	553.4	145	4535	100	30.0
22FRA086	Plume 12 ITP	553.4	95	3230	100	30.0
22FRA087	Plume 12 ITP	553.4	96	3225	100	30.0
22FRA088	Plume 13 ITP	525.6	110	3905	100	30.0
22FRA089	Plume 13 ITP	525.6	101	3245	100	30.0
22FRA090	Plume 13 ITP	525.6	103	3045	100	20.0

Table C-2. Analysis data for the filtrate fraction of samples from the 2022 live-fire test of 81 mm IMX-104 cartridges.

Sample ID	Aqueous NTO (mg/L)	Aqueous SPE (mg/L)		
		HMX	RDX	DNAN
22FRA001	<0.004	<0.01	<0.01	<0.01
22FRA002A	<0.004	<0.01	<0.01	<0.01
22FRA002B	<0.004	<0.01	<0.01	<0.01
22FRA003	0.039	<0.01	0.04	0.12
22FRA004	0.038	0.02	<0.01	0.02
22FRA005	0.051	0.02	<0.01	0.02
22FRA006	0.034	0.02	<0.01	0.02
22FRA007	0.150	0.03	0.08	0.45
22FRA008	0.066	0.02	<0.01	0.04
22FRA009	0.056	0.02	<0.01	0.04
22FRA010	0.076	<0.01	<0.01	0.03
22FRA013	5.538	0.14	3.68	6.48
22FRA014	5.518	0.14	3.66	6.62
22FRA015	5.675	0.16	4.28	7.76
22FRA016	3.500	0.05	1.35	1.68
22FRA017	3.059	0.05	1.23	2.14
22FRA018	3.262	0.05	1.12	1.97
22FRA019	<0.004	<0.01	<0.01	<0.01

Table C-2 (cont.). Analysis data for the filtrate fraction of samples from the 2022 live-fire test of 81 mm IMX-104 cartridges.

Sample ID	Aqueous NTO (mg/L)	Aqueous SPE (mg/L)		
		HMX	RDX	DNAN
22FRA020	0.004	<0.01	<0.01	<0.01
22FRA021	0.025	0.03	<0.01	0.02
22FRA022	0.021	0.04	<0.01	<0.01
22FRA023	0.017	0.03	<0.01	0.01
22FRA024	0.040	0.03	<0.01	0.03
22FRA025	0.022	0.02	<0.01	0.06
22FRA026	0.017	0.02	<0.01	0.02
22FRA027	0.016	0.04	<0.01	0.01
22FRA028	0.011	0.03	<0.01	0.01
22FRA029	0.022	0.02	<0.01	0.02
22FRA030	0.005	0.01	<0.01	<0.01
22FRA031	0.026	0.02	<0.01	0.02
22FRA032	0.006	0.02	<0.01	<0.01
22FRA033	0.006	0.01	<0.01	<0.01
22FRA034	0.028	<0.01	<0.01	<0.01
22FRA035	0.009	0.01	<0.01	<0.01
22FRA036	0.009	0.01	<0.01	0.01
22FRA037	0.023	<0.01	<0.01	<0.01
22FRA038	0.096	<0.01	0.02	0.09
22FRA039	0.096	<0.01	0.02	0.10
22FRA040	0.091	<0.01	0.02	0.08
22FRA041	0.138	<0.01	0.03	0.14
22FRA042	0.132	0.02	0.14	0.96
22FRA043	0.082	<0.01	0.04	0.14
22FRA044	0.057	<0.01	0.02	0.09
22FRA045	0.044	<0.01	0.01	0.06
22FRA046	0.044	<0.01	0.01	0.03
22FRA047	0.033	<0.01	<0.01	0.02
22FRA048	0.104	<0.01	0.02	0.09
22FRA049	0.045	<0.01	<0.01	0.03
22FRA050	0.042	<0.01	<0.01	0.02
22FRA051	0.025	<0.01	<0.01	0.03
22FRA052	0.035	<0.01	0.01	0.05
22FRA053	0.012	<0.01	<0.01	0.03
22FRA054	0.079	<0.01	<0.01	0.04
22FRA058	8.813	0.05	1.92	1.87

Table C-2 (cont.). Analysis data for the filtrate fraction of samples from the 2022 live-fire test of 81 mm IMX-104 cartridges.

Sample ID	Aqueous NTO (mg/L)	Aqueous SPE (mg/L)		
		HMX	RDX	DNAN
22FRA059	7.558	0.04	1.51	1.55
22FRA060	7.961	0.04	1.52	1.41
22FRA061	3.560	0.04	0.70	0.87
22FRA062	2.960	0.03	0.58	0.85
22FRA063	2.923	0.02	0.51	0.51
22FRA064	3.185	0.09	1.58	3.48
22FRA065	2.462	0.05	1.10	2.50
22FRA066	2.514	0.04	1.13	2.13
22FRA067	3.440	0.05	1.28	1.30
22FRA068	3.756	0.04	1.37	1.23
22FRA069	3.286	0.04	1.19	1.28
22FRA070	3.672	0.03	0.71	0.49
22FRA071	3.843	0.02	0.68	0.42
22FRA072	3.877	0.02	0.52	0.24
22FRA073	2.749	0.02	0.35	0.36
22FRA074	2.486	0.02	0.26	0.16
22FRA075	2.005	0.01	0.23	0.19
22FRA076	2.438	0.02	0.51	0.56
22FRA077	2.643	0.03	0.66	0.65
22FRA078	2.515	0.02	0.50	0.49
22FRA079	3.164	0.02	0.37	0.17
22FRA080	3.296	0.02	0.38	0.23
22FRA081	4.302	0.03	0.49	0.23
22FRA082	5.474	0.04	1.12	0.85
22FRA083	4.857	0.04	1.35	0.94
22FRA084	5.447	0.03	1.17	0.91
22FRA085	4.279	0.04	1.12	1.24
22FRA086	4.322	0.05	1.08	1.21
22FRA087	3.627	0.04	1.09	1.08
22FRA088	6.389	0.03	0.85	0.48
22FRA089	4.783	0.03	0.60	0.36
22FRA090	6.617	0.04	0.82	0.50

**Table C-3. Analysis data for solid filter extractions of samples from the 2022 live-fire test of 81 mm IMX-104 cartridges.**

Sample ID	Filter Extract (mg/L)			
	NTO	HMX	RDX	DNAN
22FRA001	<0.004	<0.01	<0.01	<0.01
22FRA002A	<0.004	<0.01	<0.01	<0.01
22FRA002B	NA	NA	NA	NA
22FRA003	0.054	<0.01	<0.01	<0.01
22FRA004	0.073	<0.01	<0.01	<0.01
22FRA005	0.075	<0.01	<0.01	<0.01
22FRA006	0.031	<0.01	<0.01	<0.01
22FRA007	0.457	<0.01	<0.01	0.05
22FRA008	0.108	<0.01	<0.01	<0.01
22FRA009	0.122	<0.01	0.15	<0.01
22FRA010	0.167	<0.01	<0.01	<0.01
22FRA013	23.773	0.29	0.41	8.34
22FRA014	22.112	0.21	0.26	6.75
22FRA015	13.563	0.26	0.29	6.76
22FRA016	22.439	0.06	0.07	2.15
22FRA017	4.861	0.04	0.05	1.11
22FRA018	13.285	0.03	0.02	1.38
22FRA019	<0.004	<0.01	<0.01	<0.01
22FRA020	<0.004	<0.01	<0.01	<0.01
22FRA021	0.020	<0.01	<0.01	<0.01
22FRA022	0.053	<0.01	<0.01	<0.01
22FRA023	0.054	<0.01	<0.01	<0.01
22FRA024	0.200	<0.01	<0.01	0.01
22FRA025	0.059	<0.01	<0.01	0.02
22FRA026	0.014	<0.01	<0.01	<0.01
22FRA027	0.009	<0.01	<0.01	<0.01
22FRA028	0.005	<0.01	<0.01	<0.01
22FRA029	0.034	<0.01	<0.01	<0.01
22FRA030	<0.004	<0.01	<0.01	<0.01
22FRA031	0.019	<0.01	<0.01	<0.01
22FRA032	<0.004	<0.01	<0.01	<0.01
22FRA033	<0.004	<0.01	<0.01	<0.01
22FRA034	0.010	<0.01	<0.01	<0.01
22FRA035	0.007	<0.01	<0.01	<0.01
22FRA036	<0.004	<0.01	<0.01	<0.01
22FRA037	0.008	<0.01	<0.01	<0.01

Table C-3 (cont.). Analysis data for solid filter extractions of samples from the 2022 live-fire test of 81 mm IMX-104 cartridges.

Sample ID	Filter Extract (mg/L)			
	NTO	HMX	RDX	DNAN
22FRA038	0.085	<0.01	<0.01	<0.01
22FRA039	0.133	<0.01	<0.01	0.01
22FRA040	0.038	<0.01	<0.01	<0.01
22FRA041	1.150	<0.01	<0.01	0.06
22FRA042	0.257	0.04	0.03	0.13
22FRA043	0.015	<0.01	<0.01	0.01
22FRA044	0.023	<0.01	<0.01	<0.01
22FRA045	0.034	<0.01	<0.01	<0.01
22FRA046	0.010	<0.01	<0.01	<0.01
22FRA047	0.007	<0.01	<0.01	<0.01
22FRA048	0.044	<0.01	<0.01	0.03
22FRA049	0.031	<0.01	<0.01	<0.01
22FRA050	0.012	<0.01	<0.01	<0.01
22FRA051	0.022	<0.01	<0.01	<0.01
22FRA052	0.008	<0.01	<0.01	0.01
22FRA053	0.020	<0.01	<0.01	<0.01
22FRA054	0.025	<0.01	<0.01	0.04
22FRA058	43.029	0.14	0.21	3.16
22FRA059	34.593	0.09	0.21	2.61
22FRA060	32.478	0.11	0.26	2.47
22FRA061	22.454	0.06	0.11	1.84
22FRA062	18.811	0.06	0.08	1.37
22FRA063	22.173	0.08	0.13	1.51
22FRA064	21.422	0.18	0.22	4.83
22FRA065	26.255	0.16	0.25	4.34
22FRA066	14.339	0.17	0.28	4.41
22FRA067	9.763	0.12	0.19	2.66
22FRA068	14.250	0.14	0.29	3.53
22FRA069	17.266	0.12	0.23	3.12
22FRA070	7.221	0.06	0.12	1.54
22FRA071	3.368	0.06	0.14	1.46
22FRA072	6.067	0.04	0.10	1.06
22FRA073	4.243	0.03	0.06	0.73
22FRA074	4.035	0.02	0.04	0.59
22FRA075	7.301	0.02	0.04	0.53
22FRA076	5.379	0.06	0.20	1.93

Table C-3 (cont.). Analysis data for solid filter extractions of samples from the 2022 live-fire test of 81 mm IMX-104 cartridges.

Sample ID	Filter Extract (mg/L)			
	NTO	HMX	RDX	DNAN
22FRA077	5.925	0.06	0.11	1.76
22FRA078	8.006	0.08	0.16	1.98
22FRA079	6.062	0.03	0.08	0.70
22FRA080	6.939	0.02	0.04	0.58
22FRA081	10.330	0.04	0.10	0.76
22FRA082	13.384	0.07	0.14	1.80
22FRA083	7.070	0.14	0.20	2.40
22FRA084	15.152	0.10	0.33	2.88
22FRA085	15.114	0.13	0.27	2.50
22FRA086	9.853	0.14	0.16	2.36
22FRA087	3.613	0.08	0.16	2.59
22FRA088	5.146	0.05	0.11	1.13
22FRA089	5.650	0.05	0.11	1.10
22FRA090	6.078	0.05	0.11	1.14

## Abbreviations

ACN	Acetonitrile
AFS	Armaments Center Fuze Simulator
CCV	Continuing calibration verification
CFS	CRREL fuze simulator
CRREL	Cold Regions Research and Engineering Laboratory
DEVCOM AC	Combat Capabilities Development Command Armaments Center
DNAN	2,4-dinitroanisole
DODAC	Department of Defense Ammunition Code
DU	Decision unit
ERDC	Engineer Research Development Center
ERF	Eagle River Flats
ESTCP	Environmental Security Technology Certification Program
HMX	1,3,5,7-tetranitro-1,3,5,7-tetrazocane
HPLC	High-performance liquid chromatography
ICV	Initial calibration verification
IMX-104	An insensitive munition formulation containing DNAN, NTO, and RDX
IPA	Isopropanol
IQR	Interquartile range
ITP	Inside the plume
LCS	Laboratory control sample

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MeOH	Methanol
MIS	Multi-Increment Sampling
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not applicable
NTO	3-nitro-1,2,4-triazol-5-one
OTP	Outside the plume
PBXN-5	A polymer-bonded explosive containing HMX
PBXW-14	A polymer-bonded explosive containing HMX
RDX	1,3,5-trinitroperhydro-1,3,5-triazine
RSD	Relative standard deviation
RT	Retention time
S&A	Safe and arm device
SD	Standard deviation
SERDP	Strategic Environmental Research and Development Program
SPE	Solid-phase extraction
v/v	Volume per volume

# REPORT DOCUMENTATION PAGE

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<b>14. ABSTRACT</b> Postdetonation energetic residues can have environmental impacts that present a risk to military training-range sustainment. As munitions with new explosive formulations are developed and fielded, quantitative methods for assessing their residues are needed. Command detonation (i.e., static firing) allows residue testing to occur early in the acquisition process; however, its representation of live-fire residue production is uncertain due to differences in the initiation mechanism and cartridge orientation. This study aims to validate residue testing by command detonation through statistical comparison of residue deposition rates between live fire and command detonation. Live-fire residues were collected from fourteen 81 mm IMX-104 mortar cartridges fired onto snow, and deposition rates were compared with previous command-detonation tests of the same munition. Average live-fire deposition rates were 8000 mg NTO (3-nitro-1,2,4-triazol-5-one), 60 mg DNAN (2,4-dinitroanisole), 20 mg RDX (1,3,5-trinitroperhydro-1,3,5-triazine), and 2 mg HMX (1,3,5,7-tetranitro-1,3,5,7-tetrazocane) per cartridge. Compared to command detonation, live fire of the study munition produced significantly greater residues of NTO ( $p < 0.0001$ ) and RDX ( $p = 0.01$ ) but not DNAN ( $p = 0.067$ ). Although absolute deposition rates of some IMX-104 compounds differed, command detonation was successful at predicting the order of magnitude of each IMX-104 compound for the studied 81 mm munition.					
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