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An Examination of Protocols for the Collection of Munitions-Derived Explosives Residues on Snow-Covered Ice

Michael R. Walsh, Marianne E. Walsh, Charles A. Ramsey, and Thomas F. Jenkins

April 2005

COVER: Sampling in the pre-dawn light at Eagle River Flats, January 2004. Sampling bias is evident in this image as the samplers follow the darkest segment of the plume from the detonation point toward the edge of the plume. The second bag at each sampling location is the subsurface sample. (Photo by C.A. Ramsey)

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Prepared for OFFICE OF THE CHIEF OF ENGINEERS

ABSTRACT

Range contamination and sustainability are major issues for the United States military. Training is a critical factor in force readiness, and the availability of ranges is crucial to this need. To determine the impact of training on ranges, data are required on the deposition of explosives residues from live-fire and blow-in-place detonation of munitions. A method of sampling on snow-covered ranges, the discrete sampling method, was developed by the Army's Cold Regions Research and Engineering Laboratory to determine residues from the detonation of munitions. Although very effective, it requires the collection of many large samples, resulting in labor-intensive field operations and much processing and analysis work in the laboratory. By examining sampled locations within detonation plumes, it appears that collection bias may be affecting the results. There was also no methodology for quality assurance in the collection of the samples. We have examined the process currently in use and carried out a series of experiments to determine whether bias and sample quality issues are present in the sampling technique. Alternative methods of sample collection that afford a greater opportunity for quality control were examined and compared to the discrete sampling method. The recommended alternative sampling protocol is to collect multi-increment samples, and experimental results using this method are presented.

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PREFACE

This report was prepared by Michael R. Walsh, Mechanical Engineer, Engineering Resources Branch, Engineer Research and Development Center, Cold Regions Research and Engineering Laboratory (ERDC-CRREL), Hanover, New Hampshire; Marianne E. Walsh, Chemical Engineer, Environmental Sciences Branch (ESB), ERDC-CRREL; Charles A. Ramsey, EnviroStat, Fort Collins, Colorado; and Dr. Thomas F. Jenkins, Research Chemist, ESB, ERDC-CRREL.

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The Commander and Executive Director of the Engineer Research and Development Center is Colonel James R. Rowan, EN. The Director is Dr. James R. Houston.

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1 INTRODUCTION

Range contamination and sustainability are major issues for the United States military. Training is a critical factor in force readiness, and the availability of ranges is crucial to this need. To determine the impact of training with munitions on military ranges, data are required on the efficiency of both live-fire and blowing in place of munitions. Current lawsuits against the Army claim that residues resulting from the use of these ranges are contaminating local groundwater sources. Reliable data are necessary to assess the merit of these claims.

A method of residues sampling on snow-covered ranges was developed by the U.S. Army Corps of Engineers Cold Regions Research and Engineering Laboratory (CRREL) (Jenkins et al. 2000, 2002). Although very effective, it requires the collection of many large snow samples, resulting in slow, labor-intensive field operations and much processing and analysis work in the laboratory. From an examination of sample locations, it appears that there may be a bias toward sampling in areas where the residue plume is darkest, which may skew the results. There was also little done in the past for quality assurance as the sampling process was so laborious.

Soil sampling on firing points at the Donnelly Training Area in central Alaska between 2001 and 2003 (M.E. Walsh et al. 2005) indicates that multiple-increment sampling for residues is an effective method for characterizing a site for explosives. This work was conducted during the summer, but we hypothesized that the methods used could be effectively applied to winter sampling of residues on snow. Residue sampling on snow following a winter live-fire exercise at the U.S. Army's Fort Richardson, Alaska, Eagle River Flats impact range (Hewitt et al. 2003) indicated that this area would be ideal for testing our hypothesis. In 2004, two sets of tests were designed and carried out.

The first set was designed to compare the then-current method of sampling residues from the surface of snow with sampling methods similar to those used at Donnelly. The second set of tests was used to confirm the validity of our choice for the most effective sampling method.

Snow-covered ice is the ideal medium on which to conduct residues tests. The ice cover isolates past residues deposition from current residues in areas where no recent detonations have occurred. The snow cover provides a highly contrasting surface from which to sample. The general detonation plume area delineation is thus facilitated. The snow and ice also isolate the residues from most vegetation and soils, making sample processing easier.

2 PHYSICAL SETTING

Eagle River Flats (ERF) is an estuarine salt marsh located at the mouth of the Eagle River, along the upper Cook Inlet near Anchorage, Alaska (Fig. 1). The Flats have been used as an artillery and mortar impact range for Fort Richardson since the late 1940s. This small, 865-ha range is periodically flooded by the second-highest tides on Earth. In the winter, the area freezes over and is covered with snow. Temperatures are moderated by the open waters of the inlet throughout the winter months and generally remain below freezing from late November through mid-March.

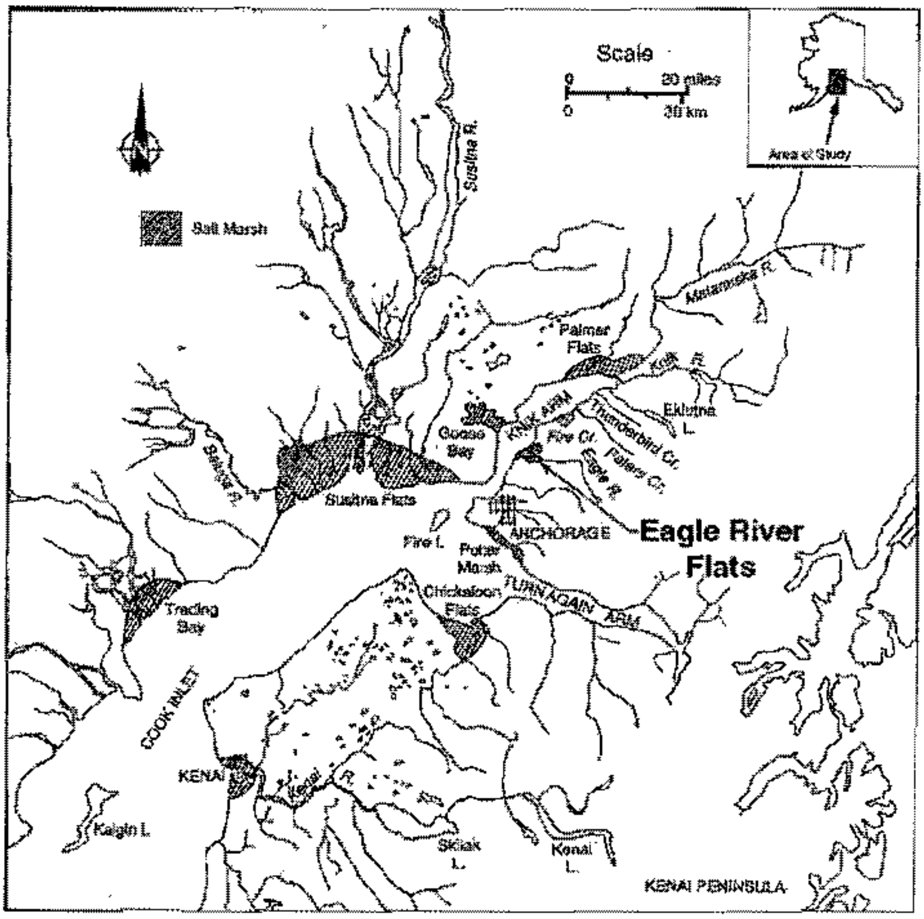


Figure 1. Eagle River Flats, location of the tests.

Ice thickness and snow cover on the flats vary according to several parameters. Temperature is an obvious factor, but snow cover is critical to limiting ice depth and the freezing of the ground beneath the ice sheet. The frequency and severity of the flooding tides thickens the ice sheet. Wind will influence the snow depth and heat transfer. Normally, an ice sheet sufficiently thick to prevent penetration of live-fired rounds up to 105-mm is attained by mid-December (Collins and Calkins 1995).

The ice sheet is grounded in most areas and is of sufficient thickness to allow traverse by heavy vehicles. Vehicular access throughout the eastern half of the Flats in winter allows efficient testing and operations over a large area. Access to post from ERF is via a well-maintained road, and laboratory facilities are located within 10 km of the test area.

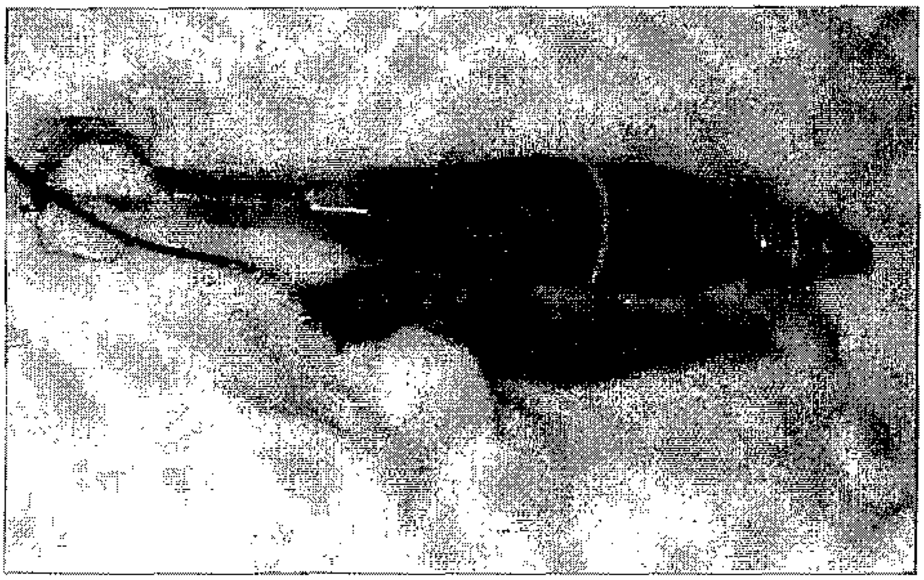
3 METHODS

Sampling protocol tests were carried out in two phases in conjunction with research into the quantification of explosives residues resulting from the detonation of military munitions. Tests in January 2004 on 81-mm mortar rounds and 105-mm artillery rounds focused on the comparison of alternative sampling methods with the discrete sampling method (DSM) currently in use. The tests in March 2004 using 155-mm artillery rounds looked at the application of the proposed new sampling method. All tests were conducted on fuzed static rounds, employing the standard blow-in-place method used by the Army to dispose of dud rounds found on ranges and battlefields. Table 1 outlines these tests. Appendix A contains more detailed information on the munitions.

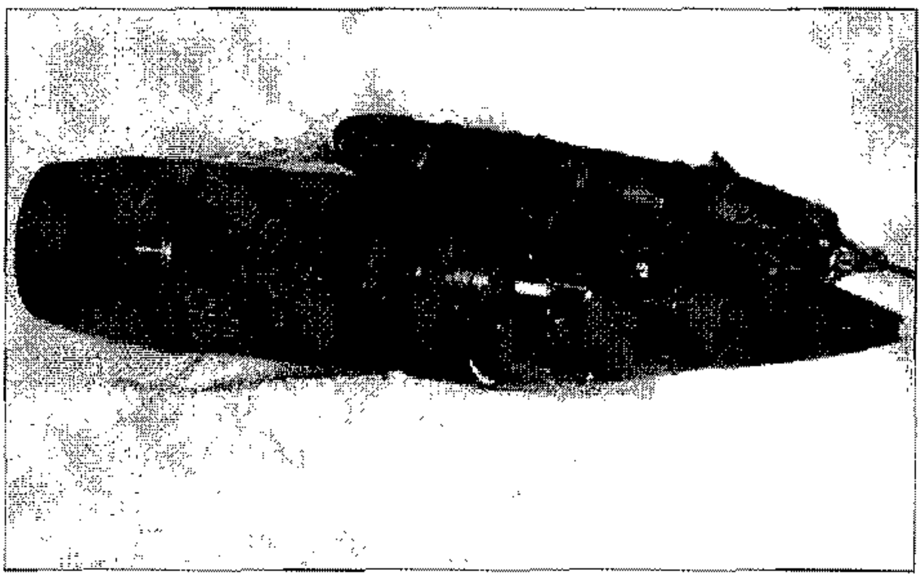
Table 1. Testing conducted for sampling protocol study.

Test/date	Munition	Filler	Objectives
Comparative tests (January 2004)	81-mm mortar rounds with point-detonating fuze and 105-mm artillery rounds with point-detonating fuze	Composition B 60% RDX 39% TNT 1% wax	1) Develop quality assurance methods for winter residues sampling. 2) Verify validity of then-current sampling method (DSM). 3) Develop protocols for alternative sampling methods. 4) Compare sampling method results. 5) Determine best sampling method.
Application and confirmation tests (March 2004)	155-mm howitzer rounds with point-detonating fuze	Composition B and TNT	1) Refine QA techniques. 2) Test sampling method implementation.

All tests used 0.57-kg blocks of C4 (91% RDX, 9% plasticizers) initiated with non-electric blasting caps as the donor charge. The C4 was set alongside the body of the round for the 81-mm detonations and near the fuze on the 105-mm and 155-mm rounds (Fig. 2). Up to seven rounds were detonated and sampled each day.



a. 81-mm fuzed round with C4 donor charge.



b. 105-mm fuzed round with C4 donor charge.

Figure 2. Test setup for detonation of 81-mm mortar rounds and 105-mm howitzer rounds.

Comparative Tests

Comparative tests were conducted from 14 to 17 January. Snow depth was more than normal, 33 cm. Temperatures hovered around −35°C in the mornings with light winds of under 1.3 m/s. Trace amounts of light snow fell sporadically during the first day, but not enough to interfere with the sampling. A weak sun behind a partially overcast sky had no influence on the plumes. Table 2 outlines the test schedule as executed.

Table 2. Test execution for sampling protocol tests.

Date	Munition	Quantity	Tasks
14 January	81-mm mortar	2	Detonation and sampling
15 January	81-mm mortar	3	Detonation and sampling
16 January (morning)	81-mm mortar	2	Detonation and sampling
	105-mm howitzer	2	Detonation and sampling
16 January (afternoon)	105-mm howitzer	5	Detonation and delineation of plumes
17 January	105-mm howitzer	(5)*	Sampling of plumes

* Plumes from the five rounds detonated the afternoon of 16 January were sampled 17 January.

Prior to detonation, locations for the rounds were marked and sampled for background contamination. The rounds were set up by the troops under the supervision of the UXO technician to ensure uniformity of configuration. Following detonation, a GPS technician walked the outline of the plumes, demarcating the estimated residue area based on the observable soot. Sampling of the plumes then commenced using the methods briefly described below.

The Discrete Sampling Method (DSM) entails using a 0.45-m-wide Teflon-lined snow shovel to collect several approximately 1-m^2-sized samples from the surface of the snow to a depth of about 2 cm, plus whatever visible residues may remain in the sampled area. Each sample is placed in its own polyethylene bag for later analysis. The goal is to sample as much of the plume area as is practical, excluding the crater at the detonation point. This will vary between less than 1% of the area for large munitions to 80% for smaller ordnance, resulting in anywhere from five to 25 samples per plume. Plume size is heavily influenced by wind speed, which makes the area sampled difficult to generalize for a particular round. Sampling location is to be random and not influenced by plume coloration. A more thorough treatment of this subject can be found in Jenkins et al. (2002).

In addition to the collection of the DSM samples, three alternative sampling methods were tested. Adjacent sampling (Adjacents) entails taking a 0.04-m^2

sample adjacent to each DSM sample using a 20- × 20-cm hand scoop and combining samples in a single bag for processing and analysis. The objective of this test was to investigate whether smaller samples composed of increments corresponding to DSM locations give results similar to those of DSM samples. This tests both the repeatability of the sampling methods and the validity of multiple-increment sampling.

For the medium-increment sampling method (MIS), increments are taken while walking evenly spaced (1- to 2-m) lanes within the plume. The goal is to collect about 40 systematic-random increments with a 20- × 20-cm hand scoop; these are then combined in a single bag. Systematic-random sampling is the collection of increments in a random location within a rough grid. The objective of MIS sampling is to quantify the plume residues by obtaining a single representative sample composed of increments collected in a systematic-random fashion while covering the entire plume without being influenced by soot deposition darkness or proximity to the detonation point and crater.

Large-increment sampling (LIS) provides more complete coverage of the plume through the collection of a large number of small increments. The goal is to collect approximately 100 random increments while covering the complete plume, including the crater. A 0.01-m^2 (10- × 10-cm) hand scoop is used to collect the samples. A sample bag will hold one LIS. The objective of large-increment sampling is the same as MIS sampling, with the aim of better plume representation through a more distributed sampling pattern that includes the detonation craters.

All samples and increments are taken to a depth of about 2 cm. Visible residue remaining in the sampled area is removed with a small hand scoop and placed in the sample bag. The bags, 38- × 76-cm particle-free polyethylene bags, are sealed with a cable tie that also holds a label describing the sample. DSM samples are left at their sampling locations for later position measurement; the other samples are left near the plume for later transportation to the processing area.

The process of estimating explosives residues from surface snow samples is based on work outlined in Walsh and Ranney (1998), Jenkins et al. (2002), and Hewitt et al. (2003). Essentially, the snow samples are thawed, the filtrate separated from the soot fraction and concentrated using solid-phase extraction, explosives concentrations are determined for each fraction using chromatographic instrumentation, and the concentrations combined and extrapolated over the whole plume to determine residue masses. For the DSM studies, we looked at residues of RDX and HMX, a manufacturing contaminant of RDX. Table C-2 gives the mass estimates for each plume based on the sample type.

Application and Confirmation Tests

Tests implementing the protocol chosen as a result of comparative tests were conducted 16–17 March at Eagle River Flats, north of the location of the January tests. The weather was much milder, with early-morning temperatures ranging from −13° to −6°C. Winds were variable, coming out of the south on the 16th with no wind recorded during detonation on the 17th. This worked out well as the detonation line on the 16th was the northernmost line. A trace amount of snow fell prior to detonation of the rounds on the 16th , with no precipitation during the tests on the 17th. Scattered clouds minimized the effect of the much stronger sun, with some effect on the plumes after noon. However, most sampling was complete by that time. Table 3 outlines the test schedule as executed.

Table 3. Test execution for protocol application tests.

Date	Munition	Quantity	Tasks
16 March	155-mm howitzer	7	Detonation and sampling
17 March	155-mm howitzer	7	Detonation and sampling

The sampling protocol chosen for further testing was the LIS. As described above, this method samples the complete plume, including the crater, using a 10- × 10-cm scoop and collecting around 100 increments. For every plume, at least two LISs were collected and other quality assurance (QA) procedures were implemented. The first set of rounds was filled with Comp-B, the same filler used for the protocol tests described above. The second set of rounds used TNT as the explosive filler. Both sets of rounds were detonated with a single demolition block charge of C4 (DODIC M023) as the donor charge. An M739 point-detonating fuze (DODIC N340) was installed in each round. All rounds contained a supplementary TNT charge in the fuze well below the M739 fuze (see Appendix A). Figure 3 shows the setup common for all the rounds. All seven rounds for each test were detonated within a three-second window. No DSM samples were collected on either date.

Quality Assurance

Quality assurance (QA) was an important part of both series of tests. Field QA procedures were developed and implemented to verify that the data obtained using this sampling method are valid. Some of these procedures were carried over to the protocol application tests, with additional QA procedures conducted to further validate the new protocol. Table 4 outlines the QA tests conducted over the course of the study.

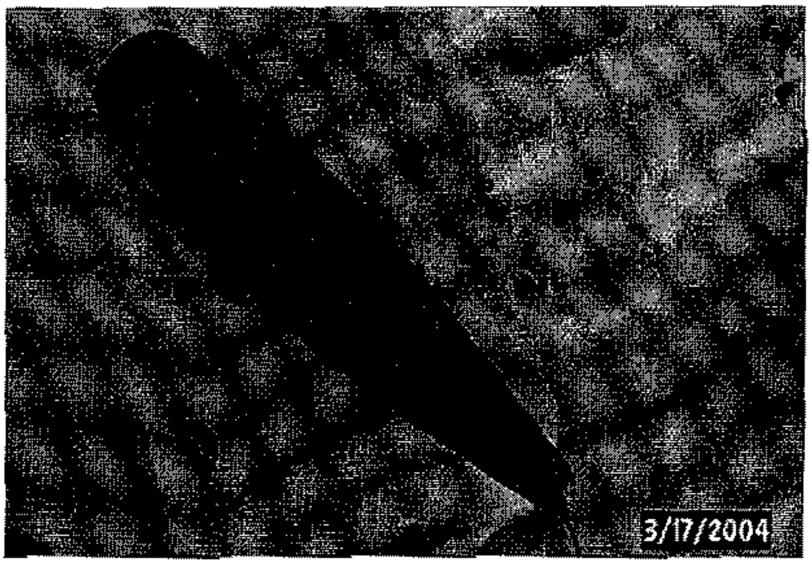
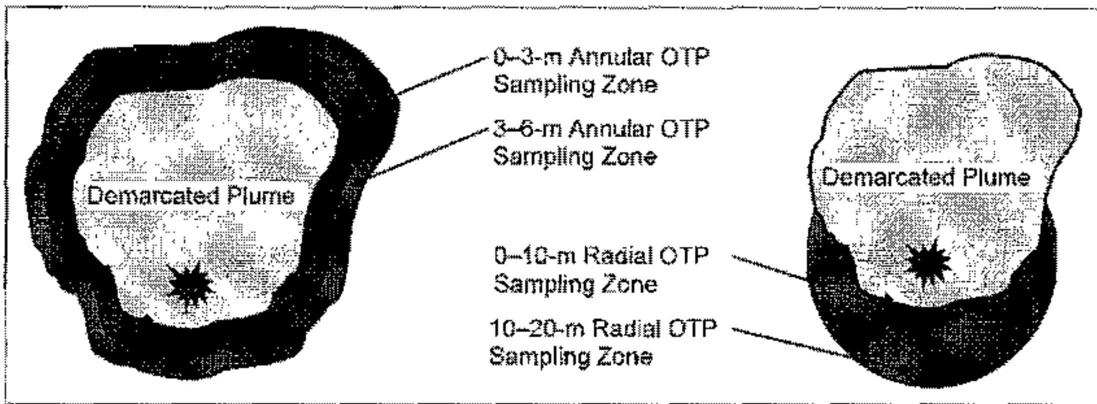


Figure 3. Detonation setup for 155-mm implementation tests.

Table 4. Quality assurance procedures.

Procedure	Description	Objective
Subsurface sampling	Samples are collected beneath areas previously sampled.	To determine whether the sampler is collecting all residue from a location.
Duplicate and triplicate sampling	Sampling method is repeated within a specific plume.	To determine the repeatability of a sampling method.
Paired MIS sampling	Two MIS samples in the same plume consisting of adjacent increments.	To estimate the repeatability of multi-increment sampling through close-proximity replicate sampling. Examine residues heterogeneity.
Radial sampling within the plume	The plume is divided into zones radiating out from the detonation point. LISs collected in each zone.	To determine the influence of sampling in proximity to the detonation point to the overall estimated residue deposition.
Gradient (gray-scale) sampling within the plume	The plume is divided into three zones by the perceived density of the residue soot. LISs are collected in each zone.	To determine the influence of sampling bias toward darker areas.
Radial sampling outside the plume	Sampling outside the demarcated plume and within concentric rings centered on the detonation point.	To determine whether the soot plume correctly models the distribution of explosives residues following a detonation.
Annular sampling outside the plume	Sampling outside the demarcated plume within a concentric ring surrounding the plume or the edge of another annular sampling area.	To determine whether the demarcation of the residues plume is correct.

Subsurface sampling was conducted on DSM samples in January and on one of each paired MIS samples in March. In January, the subsurface sample size matched that of the surface sample, and both samples were collected in the same manner. In March, the subsurface samples were smaller than the surface sample to avoid the possibility of contamination along the edge of the subsample. Duplicate and triplicate sampling was conducted on all other types of samples during tests both months. Paired MIS samples in March were collected with a 0.023-m^2 (15- × 15-cm) hand scoop adjacent to each other and deposited in separate bags. Radial sampling within the plume entailed dividing the plume into three zones, each concentric from the detonation point (0- to 10-m radius, 10- to 20-m radius, and greater-than-20-m radius). A LIS was then taken within each zone. The gradient sampling within the plume entailed dividing the plume into three zones based on the perceived density of the deposited soot. The denser the soot, the darker the area of the plume. A LIS was taken from each zone. Sampling outside the plume was done using two procedures. In one, samples were taken within a band or annulus outside the plume. Up to two concentric bands (0–3 m and 3–6 m) were sampled using the LIS method (Fig. 4). The other procedure entailed sampling outside the plume within a fixed band radiating from the detonation point (0-10 m and 10-20 m). Appendix B lists the QA tests conducted for each detonation.



a. (Left) Annular zones.

b. (Right) Radial zones.

Figure 4. Sampling diagrams for outside-the-plume (OTP) sampling.

4 RESULTS AND DISCUSSION

This section will be divided into three parts: An examination of the DSM protocol, comparison with the other protocols tested in January, and the results of the implementation testing of the new protocol in March. QA methods will be discussed throughout as applicable.

1. Protocol Tests—The Discrete Sampling Method

Protocol testing on the DSM was conducted on 14 rounds. Seven 81-mm plumes and seven 105-mm plumes were sampled. A total of 123 large discrete and five multi-increment samples was taken of the 81-mm detonations, and 128 large discrete and three multi-increment samples were collected from the 105-mm detonations. Detonation crater samples are considered separately. In general, the portion of the total plume area sampled was small, less than 4% ranging down to less than 1%. All surface and subsurface discrete samples were 1 m^2 in size. The OTP sampling was done with 10-cm or 20-cm hand scoops and was composed of 40 to 120 increments. Appendix C, Table C-1, gives the DSM sampling statistics for the plumes and OTP bands. Table 5 summarizes these data.

Table 5. Detonation plume data for DSM tests.

Parameter	Statistics
81-mm plumes	**n=7**
Number of samples: Discretes (total/average)	101/13
Subsurface (Total/# of plumes)	22/2
Outside the plume*	4
Range of plume areas	637 m^2–1506 m^2
Average plume area	820 m^2
Range of OTP areas	310 m^2–490 m^2
Average of OTP areas	410 m^2
Range of sampled areas: Plumes (Area/% of plume)	11 m^2–34 m^2/0.73%–2.2%
OTPs (Area/% of OTP area)	0.80 m^2–1.6 m^2/0.21%–0.46%
Average of sampled areas: Plumes (Area/% of plume)	14 m^2/1.8%
OTPs (Area/% of OTP area)	1.3 m^2/0.32%
OTP area to plume area	50%

Table 5 (cont'd).	
Parameter	**Statistics**
105-mm plumes	**n=7**
Number of samples: Discretes (Total/Average) Subsurface (Total/# of plumes) Outside the plume	113/16 15/1 3
Range of plume areas	440 m^2–1300 m^2
Average plume area	860 m^2
Range of OTP areas	400 m^2–490 m^2
Average of OTP areas	450 m^2
Range of sampled areas: Plumes (area/% of plume) OTPs (Area/% of OTP area)	15 m^2–18 m^2/1.2%–3.4% 0.66 m^2–0.82 m^2/0.14%–0.18%
Average of sampled areas: Plumes (area/% of plume) OTPs (Area/% of OTP area)	16 m^2/1.9% 0.71 m^2/0.16%
OTP area to plume area	52%

* Data for the one 0- to 10-m radius OTP test are not included; there were no detectable residues in this test.

One of the objectives of the DSM protocol is to collect enough samples to derive a valid representation of the plume. However, the average area of the plumes is quite large (840 m^2). To sample a significant portion of the plume, around 10%, 50 to 150 discrete samples would have to have been collected. Even half that number is impractical. The 10% target was not derived in a scientific manner but was set as a goal when the DSM protocol was being developed. However, ongoing work by several researchers at CRREL indicates that to adequately represent a substance heterogeneously deposited over a given area, at least 30 samples (or increments) need to be obtained. The total number is a function of the increment size and sampling area. Taking 10 to 20 DSM samples, in the 1%–3% range, is more practical. Taking fewer samples (or increments) makes it more difficult to obtain representative samples, which will be demonstrated in the section on the distribution of samples that follows.

Prior to sampling, two of the 81-mm plumes were checked from an 8-m-high tower after delineation by the GPS technician to qualitatively determine whether the complete plume was being demarcated. Both plumes looked fully enveloped, and OTPs were done on both to verify this observation.

Using estimates of the residues masses, we looked for sources of sampling error for the DSM tests. The two most obvious places are beneath the areas sampled and outside the demarcated plumes. Residues in these areas will result in an underestimation of the unreacted mass of explosives. Other sources of error are the possible biasing of the sampling toward the detonation point and over-sampling the darker areas of the plume (sample location). These will likely have the opposite effect, resulting in an overestimation of the residue mass. Finally, the contribution and sampling procedure for the craters will be examined.

Subsurface residues

Subsurface samples were taken from three plumes. All three subsurface samples were DSM-type samples (1 m^2 × 2 cm deep) and contained residues. The results are given in Tables 6 and C-2. The subsurface residues were higher than we anticipated and constitute what appears to be a significant source of error (≈10%). Collection conditions may have been the cause of some of the error. The difficulty of working in the extremely low temperatures during the 81-mm tests (≈–35° C) likely contributed to some sampling error while collecting the discretes. The subsample for the 105-mm round was taken on the final day of sampling, when temperatures were more moderate (–10° C) and more time was available for sampling. However, more work needs to be done to get a better indication of the magnitude of the error. Some of this was done as part of the pre-existing sampling plan for the protocol tests and some was built into the application tests to take place in March. From this test it is obvious that care must be taken to ensure that proper sampling depth is achieved and that any residues beneath the sampled area are collected during the sampling process.

Table 6. Underreporting of total mass residues due to sampling depth error.

	Residues recovered (mg)		% of total mass*	
Plume	HMX	RDX	HMX	RDX
81-1	1.4	3.7	11%	16%
81-5	1.0	1.4	12%	11%
105-6	0.68	1.6	5.8%	6.0%
Average	—	—	9.6%	11%

* Subsurface/(subsurface + DSMs)

Residues outside the demarcated plume

The results for the outside-the-plume samples indicate that plume demarcation is adequate. Two types of tests were performed, sampling a 3-m-wide concentric zone outside the plume and sampling within a fixed distance from the detonation point outside the plume. The majority of tests were of the concentric OTP configuration, with one radial test to determine whether our strategy of sampling the visible plume (wind dispersion) rather than in concentric circles from the detonation point (radial dispersion) is valid. The results are given in Table 7. Residues averaged less than 1% of DSM values for HMX and less than 2% of DSM values for RDX. The one test done using the radial OTP strategy came up blank. These tests indicate that we are delineating the plume correctly and that the strategy of sampling within the visible plume for residues is likely sufficient. More data on the radial sampling outside the plume are needed to reinforce the second conclusion, and additional concentric data are needed to confirm the delineation strategy. It is important to note that the OTP samples were multi-increment and not discrete samples.

Table 7. Results of sampling outside the visible plume.

		Residues recovered (mg)		% of total mass*	
Plume	Area sampled	HMX	RDX	HMX	RDX
81-1	0- to 3-m annulus	0.18	ND	1.6%	0%
81-2	0- to 3-m annulus	ND	ND	0%	0%
81-3	0- to 3-m annulus	ND	ND	0%	0%
81-5	0- to 3-m annulus	ND	0.36	0%	1.1%
	0- to 10-m radius	ND	ND	0%	0%
105-3	0- to 3-m annulus	0.15	2.2	2.1%	6.2%
105-5	0- to 3-m annulus	0.25	2.4	2.2%	2.8%
105-7	0- to 3-m annulus	ND	0.43	0%	2.5%
Average				0.70%	1.6%

ND = Not detected

* OTP/(OTP + DSMs)

Sample distribution

As noted above, we looked at the DSM data in relation to the detonation point and the perceived darkness of areas within the plume to try to determine whether these factors influence the samplers' decisions as to where to sample. After completion of the DSM sampling, concentric rings were walked around the detonation points (10-m and 20-m radius) for Plumes 81-5, 105-3, and 105-7. Dark- and medium-density gray zones were also demarcated on Plume 105-4. These boundaries were entered in the GPS database, as were all the DSM locations for these and the other plumes. The 10- and 20-m radial zone boundaries were also determined for the remaining plumes and added to the GPS data to provide a wider statistical base for bias evaluation. The distribution of samples points and the residues in these areas were then examined. Data for the distribution of DSM samples are given in Table C-3. Summary results are in Table 8.

Table 8. Detonation proximity bias in DSM sample location.

Round	Zone	Area (m^2)*	# of samples	% samples	% area	Samples/area†
81-mm (n=8)	0–10 m	178	5.6	44%	24%	2.0
	10–20 m	261	5.0	41%	33%	1.3
	>20 m	381	2.0	15%	44%	0.3
105-mm (n=3)	0–10 m	267	7	42%	30%	1.4
	10–20 m	387	6.3	39%	44%	0.9
	>20 m	244	3.3	19%	26%	0.7

* Average values for plumes.

† Ratio of the % samples to % area.

Sample distribution for these tests is very interesting. The area closest to the craters was sampled on average twice as frequently for the 105-mm tests as compared to the area beyond 20 m from the detonation point, and for the 81-mm tests, the factor is over six times. Sampling in the middle zone, 10 to 20 m out, is more representative, but is still skewed for the 81-mm tests. The distribution improved greatly between the 81-mm tests and the 105-mm tests, but the density of samples near the detonation point was still high. Even though an increasing effort was made to sample in a more distributed manner, a bias still remained.

There are a couple of confounding factors that may be influencing this bias. The plume tends to be darkest in close proximity to the detonation point, and a bias toward sampling the darkest areas may be reflected in a proximity bias. Samplers also tend to start sampling near the crater, as that is where the access path leads. Finally, it is difficult when sampling to keep the size and shape of the plumes in perspective. This leads to a concentration on sampling with respect to the last sample point and not with respect to the plume as a whole.

Another way of looking at the proximity sampling bias is to compare the results for a plume assuming no sampling bias with the results corrected for the sampling patterns found from these tests. To do this, the DSM samples within specific zones of the plumes were mathematically composited and the residue masses estimated for those zones. These were then combined, correcting (weighting) for differences in zonal areas, and compared to the masses derived for the no-bias assumption. Data for these comparisons are given in Table C-4 and the results summarized in Table 9. The values given for HMX and RDX are the percent differences between the unweighted (no-bias assumption: M_u) values for the

DSMs and the values taking the oversampling in proximity to the craters into account (M_w).

$$\text{Bias} = (M_u - M_w)/M_w. \tag{1}$$

A positive percentage indicates the possible overestimation of residues by the standard DSM method.

Table 9. Detonation proximity bias (unweighted vs. weighted) in DSM residues estimates.

Plume	HMX	RDX
81-mm (n=5)	46%	31%
105-mm (n=3)	27%	24%
Overall bias (n=11)	38%	28%

The results of this analysis indicate a proximity bias. The calculated plume residual masses using composited DSM samples are more than 25% greater than when sample location is taken into account and the samples are weighted with respect to area. Again, the results for the 105-mm sampling indicate a better distribution of samples, reflecting the greater effort to obtain more representative samples later in the process. The results show that plume residues masses will be overestimated based on sample location bias with respect to the detonation point.

Data for the gray-scale test are more divergent than for the concentric data. Although we have only one test examining the effect of soot density in the plume on sampling bias, it is worth noting. Table 10 contains the data and analysis for this condition. Bias is measured as the ratio represented in Equation 1.

Table 10. Soot density bias in DSM residues estimates, Plume 105-4.

Condition	HMX (mg)	RDX (mg)
Weighted	4.5	10
Standard DSM	6.6	15
Bias	47%	50%

The soot density biases of 47% and 50% for HMX and RDX are quite different from the concentricity data, especially when compared to the later (105-mm) results. This applies to the results specific to the plume as well, Plume 105-4. A comparison of the collection point zone oversampling (Table C-3) tends to reinforce this observation, as the soot density bias is much larger than the proximity bias (a factor of 2.2 vs. 1.3 for the dark gray area vs. the <10-m zone and a factor of 1.6 vs. 0.94 for the medium gray area vs. the 10- to 20-m zone). Again, a conscious effort was made not to bias sampling. Further data for the proximity and gray-scale bias hypotheses are needed if DSM sampling is to continue to be used.

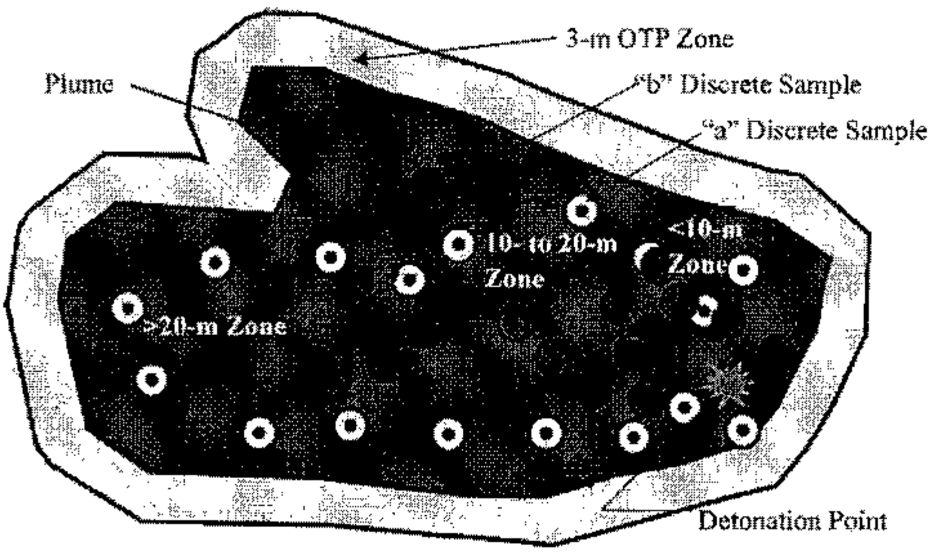


Figure 5. Replicate discrete sampling on Plume 81-3.

We obtained duplicate DSM samples of only one plume during these tests, Plume 81-3. The samples were taken by two different sampling teams, the second set being obtained after the first set was done. Figure 5 shows the sample locations for both sets. It is immediately obvious that the sample distributions are quite different. For the (a) set of samples, the most proximate area (0–10 m) is oversampled by about 50%. For the (b) set of samples, this number rises to 120%. The central area of the plume (10–20 m) is fairly well represented (100% and 120% representation), whereas the difference again widens for the area beyond 20 m: 74% and 26%, respectively. If the area beyond the 20-m line is divided by a 30-m line, the (b) samples have no representation beyond that line.

The second set of samples (b) is more concentrated near the detonation point (see also Table C-3).

Reverting back to the three-zone division of the plume, the overall residues were calculated for the plume (Table C-4). A comparison between the two samples for Plume 81-3 is given in Table 11. In this table, U_a and U_b are unweighted values and W_a and W_b are weighted residue mass values of DSM samples. It is evident that there is a difference between the two samples, with the sample collected closest to the detonation point (b) indicating more contamination than the more evenly distributed sample (a). The attribution of the greater residues for (b) to proximity to the detonation point may be misleading, as the majority of the difference between the samples comes from the poorly sampled zone beyond the 20-m radius. Thus, the difference between the samples may be attributable more to sampling sooty areas than sampling near the detonation point. Without knowing the outline of the soot gradients and having a better grasp of the residue load in the plume, a more precise determination of any sampling bias cannot be made. Although the results are for only one set of data, the agreement between the two sample sets is surprisingly close, indicating a robustness for the DSM sampling protocol not thought to exist.

Table 11. Comparison of DSM samples for Plume 81-3.

Comparison	Relationship	HMX	RDX
Raw data: a	No weighting for zones	1.1 mg	8.1 mg
Raw data: b	No weighting for zones	1.5 mg	10 mg
Unweighted bias: b to a	$(U_b-U_a)/U_a$	+36%	+24%
Weighted bias: b to a	$(W_b-W_a)/W_a$	+21%	+162%
0- to 10-m zone: b to a	$(U_b-U_a)/U_a$	+44%	−31%
10- to 20-m zone: b to a	$(U_b-U_a)/U_a$	−66%	−57%
>20-m zone: b to a	$(U_b-U_a)/U_a$	+460%	+1100%

In summary, there is bias evident in the location of samples within a detonation plume. The source of this bias may be from one or all of the following factors: tendency to sample closest to the detonation point, tendency to start sampling near the detonation point, tendency to sample within the darkest areas of the plumes, and a failure to take the full plume into consideration when choosing sample locations. The overall effect of these biases taken individually and as a group on the residues estimates will need to be determined by comparison with data that are more representative of the plume.

Craters

Missing from the above analyses are the craters. Hewitt et al. (2003) indicate that the craters (detonation points) typically do not significantly alter the overall residue quantities for detonated rounds. In live-fire tests conducted at Eagle River Flats in March 2002, fourteen 81-mm mortar rounds and thirteen 105-mm howitzer rounds were fired onto the snow-covered ice and sampled using the discrete sampling method. The craters were sampled separately. The results from this test showed a measurable contribution of less than 2% in the RDX residue quantity for only one of the 27 detonation points examined. The remainder were below the detection limit. Although the detonation points can have a higher residue concentration than the rest of the plume, their area is very small (<1%) compared to the remainder of the plume. Thus, although residues concentrations may be comparatively high within the craters, their contribution to the overall residue estimate is not significant in most cases.

We evaluated the inclusion of the craters as part of the protocol tests. Craters have always been sampled using incremental sampling. Generally, 5% to 10% of the crater is sampled for analysis. Part of our study was to look at the repeatability of the sampling technique for craters and whether sampling the various parts of the crater separately results in different residue deposition values. Six of the seven 81-mm detonation craters were sampled, as were six of the seven 105-mm craters. During sampling of the craters, the area was undergoing sub-ice interlayer infiltration by water, resulting in water seepage into the craters, especially in the center pits below the round locations. Not all components of each crater were available for sampling.

The crater centers are quite small in relationship to the overall crater and, based on past data, generally contribute little to the residues load in the crater (<10%). We were able to obtain only one good center sample that amounted to 6% of the crater residues. The centers tend to be very difficult to sample (even without the presence of water) as they are full of debris, in this case fractured ice in the form of small chips as well as frag or, in the case of live-fired rounds, mortar tail assemblies. We thus concentrated on two areas within the crater: the annulus, the area generally clear of snow between the edge of the detonation center and the berm; and the berm, the raised rim of snow surrounding the detonation point and outlining the crater. Corresponding berm and annulus samples were obtained for eight of the 14 craters. Table 12 contains the data for these crater components.

Table 12. Data for crater components (RDX).			
Detonation point	Annulus (mg)	Berm (mg)	Difference (mg)
81-1	0.033	0.042	−0.009
81-3	0.069	0.14	−0.072
81-7	0.62	0.24	0.385
105-1	0.96	0.91	0.042
105-2	0.76	2.9	−2.1
105-3	0.14	1.0	−0.88
105-4	0.75	0.67	0.077
105-7	2.0	1.2	0.76

Using the data from Table 12, a paired t-test can be used to test the deposition amounts of the RDX residues for the two crater components to determine whether they differ significantly. From Natrella (1963), the statistics for this test are as follows:

Significance level: 0.05

Average difference: −0.23

Statistical deviation of differences: 0.90

Sample size: 8

Degrees of freedom: 7

t_{Calc}: 0.753

$t_{(0.975)}$: 2.365

In our case, t_{Calc} is much less than $t_{(0.975)}$. This indicates that there is no significant difference in residues between the crater berm and annulus. Thus, no extra care must be exercised when sampling the crater, and if the annulus is inaccessible, sampling the berm will sufficiently characterize the crater as a whole.

We took duplicate annulus and berm samples at three of the craters. For the RDX residues, the relative percent difference between each of the two measurements was around 20%. Repeatability increased with the number of increments, being best with 50–100 increments (0% and 7% difference), increasing to 44% and 56% for the sample with less 40 increments. The third set, taken with 40–60 increments, fell in between with differences of 14% and 16%. The average underreporting of the total residue mass in the plumes due to ignoring the crater is 6% over the 12 samples taken during these tests.

In summary, sampling the crater is not critical in obtaining the overall residues quantities for a plume, although it will add to the accuracy of the results. Taking many increments increased the repeatability of the sample measurements, thus a large-increment sample should give a more accurate representation of the crater if required. The distribution of these increments within the crater is not critical to the repeatability of the results but distributed (representative) sampling is good practice and should be applied.

II. Protocol Tests—Alternative Sampling Methods

Examining the feasibility of replacing DSM sampling with a more efficient sampling method was the primary goals of these tests. Processing of the many large DSM samples is time-consuming and expensive. For large plumes, it is also not very practical. Therefore, we sought to examine the feasibility of using multiple-increment sampling for characterizing the plume.

Multi-increment sampling is already used as part of the DSM method. Craters are sampled using many small increments, and the large plume samples are mathematically averaged over the complete plume to derive the total residues. The ability to characterize a plume with a single sample would greatly increase efficiency and allow replicate sampling and field quality assurance procedures to be conducted.

Adjacent sampling

To determine whether multiple-increment sampling can be used in place of the DSM, we collected 20- × 20- × 2-cm-deep increments adjacent to each DSM sample location. These increments were collected in a single bag in the field and processed in the lab as a single sample. A total of 19 multiple-increment adjacent samples was taken in the 14 plumes over the course of the protocol tests (Table D-1). The results of the adjacent samples were compared to the averaged DSMs to determine the validity of characterizing the plume using smaller samples distributed as with the DSM samples. Table 13 compares these values with those of the averaged DSMs.

Looking first at the proximity of the total residue estimates for the two sampling methods, agreement between the two methods is generally very good. The values are within a factor of two for most of the tests. Averaging the data for all the tests gives a result of 17 mg HMX and 82 mg RDX. The results for the adjacent samples average higher than for the DSMs for both constituents. This indicates that more residues were recovered during the adjacent sampling procedure than with the DSM procedure. This result was predicted because of the ease of obtaining a sample with the small hand scoop over the use of the large shovel with the DSM sample. This results in less spillage during sampling and better penetration into the snow with the sampling tool.

Table 13. Comparison of DSM and adjacent sampling.

Plume #	Method	Mass estimates (mg)		Mass differences* (mg)		Relative % differences	
		HMX	RDX	HMX	RDX	HMX	RDX
81-1	Adjacent	15	23				
	Adjacent	6.8	14				
	DSM	11	20	−0.1	−1.5	0.91%	7.8%
81-2	Adjacent	9.4	4.9				
	DSM	5.6	7.2	3.8	−2.3	51%	38%
81-3	Adjacent	1.8	7.6				
	DSM	1.1	8.1	0.7	−0.5	48%	6.4%
	Adjacent	2.2	16				
	DSM	1.5	10	0.7	6.0	38%	46%
81-4	Adjacent	20	640				
	Adjacent	97	720				
	DSM	57	470	1.5	210	2.6%	37%
81-5	Adjacent	10	45				
	DSM	7.3	31	2.7	14	31%	37%
81-6	Adjacent	67	280				
	DSM	55	220	12	60	20%	24%
81-7	Adjacent	45	130				
	DSM	31	92	14	38	37%	34%
105-1	Adjacent	6.1	11				
	DSM	2.8	13	3.3	−2.0	74%	17%
105-2	Adjacent	6.6	14				
	DSM	5.0	18	1.6	−4.0	28%	25%
105-3	Adjacent	9.6	17				
	DSM	6.9	33	2.7	−16	33%	32%
105-4	Adjacent	8.5	18				
	DSM	6.6	15	1.9	3.0	25%	18%
105-5	Adjacent	28	180				
	Adjacent	24	54				
	DSM	11	82	15.5	35	83%	35%
105-6	Adjacent	17	32				
	DSM	11	25	6.0	7.0	43%	24%

Table 13 (cont'd). Comparison of DSM and adjacent sampling.

Plume #	Method	Mass estimates (mg)		Mass differences* (mg)		Relative % differences	
		HMX	RDX	HMX	RDX	HMX	RDX
105-7	Adjacent	10	23				
	Adjacent	8.5	12				
	DSM	5.3	17	4.0	0.5	55%	3%
Average	Adjacent	19*	94*	5.6	22†		
	DSM	15	71				
	RPD**	24%*	28%*				

* Mass differences are Adjacents − DSM: Adjacent duplicates averaged for these values.

† 7.9 mg difference without 81-4

** Relative Percent Difference of the average values: |Range|/Average

All adjacent increments were taken next to the DSM samples, but replicate samples were not taken adjacent to each other. Relative percent differences (RPDs) in values obtained from replicate adjacent samples range from 12 to 130%, with an average RPD of 60%. The distance between the replicate increments likely accounts for some of the difference between the values, indicating that for small-increment samples (i<30), each increment becomes more important and the sampling location can be critical. A test of this hypothesis was planned for the implementation tests (paired MIS samples with the same size scoop) in March.

The results of the adjacent sampling test indicate that DSM sampling can be replicated by multi-increment sampling. This is a significant finding as the single multi-increment sample has replaced 18 or more DSM samples, making it easier to obtain and process duplicate or triplicate samples for quality assurance. The data indicate somewhat higher residues values on average using the incremental sampling method, which may be due to the ability to obtain better samples with the smaller sampling tool. The other bias factors associated with DSM sampling remained with this exercise, including obtaining enough samples, or in this case increments, to adequately represent the plume residues in a repeatable manner. Craters were not sampled with either test.

An interesting anomaly appears in the data for Table 13 that gives an indication of the difficulties that can be encountered during blow-in-place characterization tests. The data for RDX for two of the five plumes are high when compared to those in the remaining three. Also, the ratio of RDX to HMX differs, especially for Plume 105-5. This may be indicative of less efficient detonation of the unconfined block of C4, used as the donor charge. The C4 block is 91% RDX. Separate tests done in conjunction with the 155-mm BIP tests indicate that following the proper detonation of an undeformed block of C4, the ratio of RDX to HMX residues is between 2:1 and 3:1. The donor charge efficiency and its influence on the residues plume is difficult to determine.

Medium-increment samples

We next tested full-plume incremental sampling that will allow the characterization of a plume with a single multi-increment sample. Previous work by M.E. Walsh et al. (2005) indicated that a minimum of 40 samples is required to accurately characterize a site. The standard sample bag we use for the collection of snow samples will hold 40 samples taken with a 20-cm-square scoop at a depth of 2 cm, so this tool was used.

Table 14. Relative percent differences (RPD*) in calculated residue values between averaged MIS and composited DSM sampling.

	Difference*	
	HMX	**RDX**
81-mm		
1	117%	139%
2	75%	43%
3	16%	8%
4	2%	2%
6	17%	24%
7	64%	72%
Average (n=6)	48%	48%
105-mm		
1	17%	14%
2	8%	10%
3	9%	47%
4	5%	2%
5	14%	97%
6	25%	35%
7	25%	13%
Average (n=7)	17%	37%
Overall (n=13)	33%	43%

* RPD = |Range|/Average

The relative percent differences between the MIS and DSM sampling methods for 13 plumes are shown in Table 14. The MIS samples generally had lower concentrations of residues. However, the average values for the MIS samples are very close overall to those of the DSMs. The lower values were expected, as the MIS increments are more spatially representative of the complete plume. This results in a greater percentage of the sample being collected in areas away from the detonation point and less of a tendency to sample where the plume is darkest. It is interesting to note that as the DSM sampling became more spatially representative of the plume area (105-mm data), the differences between the sampling methods became smaller and more consistent.

For five of the plumes, we took multiple MIS samples (Table 15). Three triplicate and two duplicate MIS samples were collected over five plumes. Repeatability was good, even though many of these data are near the detection limits for the analytical method. The maximum difference from average for the sample groups is 41% for HMX and 53% for RDX. Data for these tests are in Table D-2.

Overall agreement of MIS data with the DSM data is surprisingly close, with the expected lower MIS residue estimates resulting from the more representative sampling of the plume partially offset by the more efficient sample collection method. Repeatability between duplicate and triplicate samples is also good. In the past, order-of-magnitude repeatability was a difficult goal to achieve. For the MIS samples, agreement was generally in the 30% range.

Large-increment samples

Large-increment samples (LIS) comprise a large number of increments, generally 100, taken with a small sampling tool. For the tests, 100 increments from a 10-cm hand scoop fit into the standard sample bag. In these tests, sampling included the crater area, thus fully characterizing the plume. Table D-3 contains the data for these tests. In general, the residues calculated for the plumes from the LIS data were approximately equal to (within 20%) or lower than those for the DSMs (23 of 28 comparisons). For HMX, the LIS method resulted in lower estimates for five plumes and approximately equal estimates for six. For RDX, eight were lower and four approximately equal. Table 16 contains comparative data for the averaged LISs and the DSMs. These data indicate that the differences in the results of the two sampling protocols are not very significant, generally less than a factor of two. An order of magnitude difference was previously thought to be good repeatability for explosives residue sampling.

Table 15. Replicate comparisons for MIS samples.							
		Total residues		RSD*		Maximum difference from mean	
Plume	Replicate	HMX (mg)	RDX (mg)	HMX	RDX	HMX	RDX
81-2	1	2.2	3.2				
	2	2.9	6.0				
	Average	2.5	4.6	20%	45%	13%	30%
81-6	1	75	360				
	2	55	290				
	3	65	220				
	Average	65	280	15%	25%	15%	29%
81-mm average						14%	30%
105-4	1	5.1	10				
	2	8.9	23				
	3	4.9	11				
	Average	6.3	15	36%	48%	41%	53%
105-5	1	9.9	100				
	2	9.2	74				
	Average	9.6	87	5%	21%	4%	15%
105-7	1	7.7	14				
	2	7.6	19				
	3	5.1	12				
	Average	6.8	15	22%	24%	25%	27%
105-mm average	6.6	15				23%	32%
Overall average						19%	31%

* RSD = Relative Standard Deviation

Replicate sampling was conducted on six of the 14 plumes, concentric zone sampling on three, and gray-zone sampling on one (see Methods section of this report for test descriptions). The objectives of these tests were to examine the repeatability of the LIS method and determine whether the residue levels are influenced by distance from the plume or the soot density of the plume. The latter tests have a direct impact on the bias analysis of the DSM protocol.

Replicate sampling consisted of three duplicate samples and three triplicate samples. The range of values about the mean for each plume is more consistent and slightly lower overall than for the MIS samples, averaging around 30% for both residue constituents. Triplicate samples and plumes with higher residue levels tended to have smaller ranges (17% vs. 47% on average for HMX and 19% vs. 55% on average for RDX). This likely reflects the difficulty in measuring residues at concentrations that are at or below the detection limits. The average of the plume LISs was compared with the DSM results.

Table 16. Relative percent differences in calculated residue values between averaged LIS and composited DSM sampling.

	Difference with DSM	
81-mm	**HMX**	**RDX**
1	54%	53%
2	65%	71%
3*	22%	75%
4	1%	2%
5	77%	32%
6	2%	20%
7	70%	80%
Average (n=7)	42%	48%
105-mm	**HMX**	**RDX**
1	44%	38%
2	57%	26%
3*	18%	27%
4†	80%	65%
5	82%	75%
6	14%	4%
7*	21%	12%
Average (n=7)	45%	35%
Overall (n=14)	43%	41%

* Weighted average of 3-zone radial sampling

† Weighted average of 3-zone gray-scale sampling

The calculated residues totals for each zone in the concentric zone tests were added to obtain the total residues for each of the three plumes (Table 17). These totals were used for comparison purposes with the DSM results in Table 16 (see values for 81-3 and 105-3, and –7). The results of the concentric-zone tests are shown in Table 17. For all three plumes, the difference in residues between adjacent zones approaches an order of magnitude. This is not surprising if the assumption is that the residue concentration is highest near the detonation point and falls off non-linearly to the edge of the plume. These results indicate that by oversampling the plume near the detonation point, the results will be skewed toward higher levels of residues, all other factors being equal. Note that even in this test, there was a tendency to collect twice as many increments in the area closest to the detonation point as in the two areas farther out from the crater.

Table 17. Results of concentric zone sampling of three plumes.

Plume/zone	Increments	Zone area (m^2)	Increments/m^2	HMX (mg)	RDX (mg)
Plume 81-5					
<10-m	135	187	0.72	2.6	16
10- to 20-m	70	226	0.31	0.7	5.4
>20-m	58	377	0.15	0.0	1.2
Total	263	790	0.33	3.2	22
Plume 105-3					
<10-m	39	282	0.14	6.5	21
10- to 20-m	59	393	0.15	1.2	3.4
>20-m	43	263	0.16	0.53	0.27
Total	141	938	0.15	8.3	25
Plume 105-7					
<10-m	129	232	0.56	5.3	14.3
10- to 20-m	80	367	0.22	1.1	3.7
>20-m	100	347	0.29	0.12	1.1
Total	309	946	0.33	6.5	19.1
Average (n=3)					
<10-m	100	230	0.44	4.8	17
10- to 20-m	70	330	0.21	1.0	4.2
>20-m	70	320	0.22	0.22	0.86
Total	240	980	0.24	6.0	22

The final factor that will be examined is the effect of plume density or "grayness" on sampling. Only one plume was sampled by gray zones using the LIS method, Plume 105-4. The results are given in Table 18.

Table 18. Comparison of gray-zone results, Plume 105-4.

Method	HMX (mg)	RDX (mg)
3-Zone LIS	2.8	7.6
Weighted DSM	4.5	10
Standard DSM	6.6	15

These data reinforce the conclusion reached in the discrete sampling method section that there is a tendency toward sampling where the plume is darkest, or "where the good stuff is." If we assume that the weighting of the DSM sampling is correct and increases the accuracy of the method, it follows that the LIS results, which sampled the three zones separately and combined them for an overall residues value, are even more accurate in that any bias is minimized by separating out the gray zones within the plume and then sampling those zones in a systematic random manner. There remains a possibility of some bias as the sampler still has some choice as to where the sample is to be collected when a collection point is reached, but the options are limited due to the pattern that must be walked to collect the requisite number of samples.

In summary, the use of multiple-increment sampling for characterizing the residues within a detonation plume appears to be a feasible alternative to the discrete sampling method. The two protocols tested, 40-increment MIS sampling and 100-increment LIS sampling, proved repeatable and comparable to both the DSM samples and each other. Both methods are quick, allow for replicate sampling, and result in fewer samples for analysis. By reducing sampling time, more field QA can be done as well. The MIS sampling protocol has the advantage in speed (fewer increments), whereas the LIS has the advantage in forcing the collector to sample in a broader, more complete fashion, thus lowering the ability to bias the sampling.

III. Implementation Tests

Before making multiple-increment sampling the method of choice for sampling explosives residues on snow, additional work needed to be conducted to ensure the proposed method is adequate and repeatable. The method chosen for testing was the large-increment sampling protocol. The planned detonation of 155-mm HE rounds scheduled for March 2004 was used to test the implementation of this method for residue characterization. Two sets of seven rounds each were detonated, each set on a different day, and the plumes sampled for analysis.

155-mm Comp-B

Samples were collected for the 155-mm Comp-B tests using both the LIS and MIS methods. Subsurface samples, crater samples, and samples outside the plume were collected for QA (Table B-3). Table E-1 in Appendix E contains the data for these tests. Table 19 presents the averages and ranges for the plume data.

Table 19. Analysis of plume samples—155-mm Comp-B rounds.

Sample group	n=	Mean (mg)		Range (mg)		Maximum difference	
		HMX	RDX	HMX	RDX	HMX	RDX
LISs							
Plume 1	2	*2.5*	15	*1.5*	11	*29%*	72%
Plume 2	3	ND	*4.4*	—	*3.7*	—	*52%*
Plume 3	2	*1.8*	*3.6*	—	2.5	—	*36%*
Plume 4	3	*1.7*	28	—	46	—	92%
Plume 5	2	*0.35*	33	—	2.0	—	3%
Plume 6	3	ND	*1.9*	—	*4.0*	—	*87%*
Plume 7	3	*0.60*	24	—	32	—	76%
Mean-LISs	7	*1.0*	*16*	—	*14*	—	*59%*
MISs							
Plume 3	2	ND	8.0	—	1.5	—	9%
Plume 5	2	ND	10	—	0.10	—	1%
Mean-MISs	2	—	9.0	—	0.80	—	5%
All							
Maximum	9	2.5	33	1.5	46	29%	92%
Minimum	9	ND	1.9	—	0.10	—	1%
Mean	9	—	14	—	11	—	47%
Median	9	0.35	10	—	3.7	—	52%

Note: Values in italics contain one or more data point(s) at or below detection limits for the analytical method. No TNT was detected in any of these samples. Where both soot and filtrate values are below detection limits, an ND is entered. (Does not apply to **All**.)

The two MIS duplicate samples agree very closely (<±8%). This is to be expected as they were taken as adjacent pairs. The LISs are not nearly as close. Samples were taken independently, sometimes by different personnel within the same plume. Two RDX values, one each in Plume 4 and Plume 7, account for the majority of the disparity in ranges. The replicates are all within an order of magnitude where explosives were detected. This is good repeatability for residues recovery, given the number of results at or below detection limits (12) and within 50% of the analytical detection limits (10), which makes comparative analyses difficult.

The data for the crater samples demonstrate that their contribution is important but not critical (Table 20). For detonations with significant residues (Plumes 1, 4, and 7), the contribution of the crater is relatively small (<9% on average). When the detonation is higher order (>99.99% of explosives load consumed), the crater can contribute significantly to the overall residues, up to 20%.

When the residues are higher and the data are more critical in terms of range sustainability, the importance of data from the crater decreases as it becomes a less significant contributor to the residue mass within the plume. As it is impossible to determine the efficiency of a detonation in the field, it is prudent to always include the detonation point in the sample. This was done throughout the tests with the LISs.

Table 20. Data for crater samples—155-mm Comp-B rounds.

	Crater area (m^2)	Crater masses (mg)			% of plume values*			
		HMX	RDX	TNT	Area	HMX	RDX	TNT
Plume 1	13.8	0.13	0.21	—	1.1%	5.1%	1.3%	—
Plume 2	13.8	0.16	0.56	–	0.80%	—	12%	—
Plume 4	15.8	0.32	1.7	0.03	0.94%	18%	8.1%	—
Plume 6	13.3	0.12	0.38	—	0.80%	—	20%	—
Plume 7	12.4	0.02	0.22	—	0.80%	3.3%	0.92%	—
Average	13.8	0.15	0.61	0.007	0.86%	5.3%	8.1%	—

* Crater mass/plume mass

Data for six of the seven plumes indicate that no residues were detected within a distance of up to 6 m outside the plume. The one detonation with residues outside the plume, Plume 1, was the result of high foot traffic in a small area adjacent to the plume where sampling took place. It was difficult for the samplers to determine the outline of the plume in that area and the sampling was thus likely erroneous. Wind drift of some residues may also have contributed to the error. In the case of Plume 1, the estimate for HMX outside the plume is almost equal to that inside (90%), and the RDX outside the plume is equal to about 13% of that found inside the plume. Recovered residues dropped by an order of magnitude between the 0- to 3-m and the 3- to 6-m ranges in this case, indicating that the plume demarcation may have originally been satisfactory.

Subsurface samples taken beneath the MIS increments (Fig. 6) were clean, indicating that all the residues were recovered during sampling. Although the sample size is small (n=2), the results are indicative of the improvement to be gained from using the smaller sampling tool for the multiple-increment sampling protocol (see Table 6).

Figure 6. Collecting adjacent MIS samples and subsurface samples from 155-mm plume.

155-mm TNT

The sampling strategy for the TNT-filled rounds was the same as for the Comp-B filled rounds. Wind conditions were near ideal, with speeds below 1 m/s, and there was no drifting snow. All rounds were detonated within a three-second window. Replicate MIS and LIS samples were used to characterize the plume. Table 21 contains the averages and ranges for the samples. The complete data set may be found in Table E-2.

Some differences between the averages and ranges of the Comp-B tests (Table 19) and the TNT tests are apparent. The range between the MIS samples for a given plume is somewhat greater for the TNT rounds, but the agreement between the MIS and LIS samples for Plumes 3 and 5 is good. Ranges for the LISs are consistent with the exception of two values out of the 21 (Plume 2 TNT and Plume 7 RDX). Some of the variability with the averages and ranges is due to the detection limits of the analysis equipment, which cuts off the lower values of the residues, thus skewing the averages lower and the ranges higher. This was also seen with the Comp-B tests and is a factor that will have to be taken into consideration when detonations are high- or near-high-order. In all, the sampling method looks consistent and repeatable for the TNT rounds.

Table 21. Analysis of plume samples—155-mm TNT rounds.							
		Mean (mg)		Range (mg)		Maximum difference	
Sample group	n=	RDX	TNT	RDX	TNT	RDX	TNT
LIS							
Plume 1	3	6.0	8.8	7.6	15	126%	104%
Plume 2	3	6.9	11	1.8	14	9%	69%
Plume 3	2	5.9	4.5	2.1	0.6	13%	7%
Plume 4	3	ND	4.4	—	5.0	—	70%
Plume 5	2	ND	6.4	—	3.3	—	26%
Plume 6	3	4.3	2.3	4.1	1.7	53%	39%
Plume 7	2	12	4.4	19	1.2	59%	14%
Mean-LIS	7	6.6*	6.1	6.9	5.8	52%	47%
MIS							
Plume 3	2	5.9	13	5	8.0	42%	33%
Plume 5	2	ND	5.9	—	0.50	—	4%
Mean-MIS	2	—	9.5	—	4.3	—	19%
All							
Maximum	9	12	13	19	15	126%	104%
Minimum	9	ND	2.3	—	0.50	—	4%
Mean	9	6.5*	6.7	6.6*	5.5	50%	41%
Median	9	5.9	5.9	4.5	3.3	47%	39%

Note: Values in italics contain one or more data point(s) at or below detection limits for the instrumentation. Where both soot and filtrate values are below detection limits, an ND is entered. All HMX values were at or below detection limits.

* Means of the values above detection limits.

Analysis of the OTP and subsurface data are not as consistent. The data (Table 22) indicate that TNT detonation kinetics may differ significantly from Comp-B detonation kinetics (Taylor et al. 2004). Normally, TNT is not found in the soot fraction of the residues. In these tests, TNT was detected in every test in significant quantities compared to the plume surface samples. The indication is that during detonation, particles of unexploded TNT are distributed by the explosion. These particles are pale and would be difficult to see on the surface of the snow, making plume delineation problematic. These particles may also penetrate deeper into the snow than the soot. Only one OTP had any residue other than TNT in it, and neither subsurface sample had anything other than TNT in it. This indicates that the C4 donor charge fully detonated with little unreacted explosives remaining, and that the residues recovered are primarily from the projectile filler.

Table 22. Sampling QA non-plume analysis for TNT-filled 155-mm implementation tests.

	Estimated residues			Percent of demarcated plume		
Sample group	**HMX (mg)**	**RDX (mg)**	**TNT (mg)**	**HMX**	**RDX**	**TNT**
Subsurface samples						
Plume 3	ND	ND	210	0%	0%	4700%
Plume 5	ND	ND	5.4	0%	0%	70%
OTP samples						
Plume 1 (10–20 m R)	ND	ND	1.1	0%	0%	12%
Plume 2 (0–3 m A)	1.5	ND	7.3	—*	0%	66%
Plume 3 (0–3 m A)	ND	ND	4.5	0%	0%	100%
Plume 3 (3–6 m A)	ND	ND	3.0	0%	0%	66%
Plume 4 (0–3 m A)	ND	ND	11	0%	0%	250%
Plume 5 (0–3 m A)	ND	ND	0.82	0%	0%	13%
Plume 5 (3–6 m A)	ND	ND	0.52	0%	0%	8.1%
Plume 6 (0–3 m A)	ND	ND	1.2	0%	0%	52%
Plume 7 (10–20 m R)	ND	ND	1.9	0%	0%	43%
Average OTP	0.21	0.0	4.5	0%	0%	87%

* No HMX detected in plume.

In summary, the multi-increment sampling technique proved very successful in representing the detonation plumes for 155-mm Comp-B-filled fuzed artillery projectiles. Problems were encountered with plume delineation and sampling depth for the 155-mm TNT-filled fuzed projectiles. These problems would also have been encountered with the DSM protocol and point to the need for modifying the sampling protocol to take the kinetics of the TNT projectile into account. More work needs to be done to refine the protocol for TNT-filled projectiles.

5 CONCLUSIONS

Sampling residues on snow is a simple and effective method for characterizing detonation residues. Plumes can be demarcated visually with detonations involving Comp-B filler, although more care is necessary with TNT-filled rounds. The standard protocol for sampling, the discrete sampling method, is prone to bias but compares well with several other sampling protocols tested. Several multiple-increment sampling protocols were designed and tested, and all were demonstrated to be comparable and repeatable within less than an order of magnitude. Either the medium-increment or the large-increment sampling protocol will work effectively in place of the DSM protocol. The large-increment sampling protocol was tested on 155-mm high-explosive projectiles with very good results. The seven tests involving Comp-B-filled rounds were better at capturing residues than those involving the seven TNT-filled rounds, pointing to the need to modify the sampling protocol for those types of rounds. Repeatability of samples within a plume was also good, subject to influence by the detection limits of the analysis equipment. Several quality assurance tests were designed and applied to check the various sampling procedures throughout these tests and should be applied in the future to sampling of all detonation plumes.

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APPENDIX A. MUNITIONS DATA

Tables A-1 and A-2 contain munitions data for all the tests conducted for this report. Table A-3 lists munitions explosives constituents and loading for these munitions. Only constituents with significant quantities (>1 g) are listed except for HMX. Note that HMX is an unmeasured constituent of RDX, the result of the manufacturing process, and may constitute up to 9% of the total RDX load. The number of blasting caps and amount of detonation cord used in each test varied according to the training needs of the troops and the discretion of the UXO technicians assisting with the operation. The majority of the explosives, however, were contributed by the test projectiles or rounds and the donor charge.

Table A-1. Munitions and explosives data—January tests.

NSN	DODIC	Nomenclature	Lot number	Quantity drawn
1315005637067	C256	Cartridge, 81 mm: HE M374 w/PD fuze	MA-84B153-025	7
1315000284857	C445	Cartridge, 105 mm: M1 HE w/o fuze	LS-88D125-007	7
1375014151232	ML47	Cap, blasting, nonelectric, 30-ft shock tube	EBW97K060-008	8
1375014151231	MN03	Cap, blasting, M13	ENB00M002-007	8
1375014151233	MN06	Cap, blasting, Non-electric, M13	SHK98D001-001	15
1375001809356	M456	Cord, detonating, pentaerthyrie tetranitrate	EBG03A002-015	610 m
1375014151235	MN08	Igniter, time blasting fuse with shock, M81	LNO98E001-003	15
1375007247040	M023	Charge, demolition block, Comp C4, M112	MA-97A003-007A	16
1390010809447	N340	Fuze, point detonating, M739	MA-84B007-013	7

Notes: Drawn from Fort Richardson Ammo Supply Point.
Data from DA Form 581—Request for Issue and Turn-in of Ammunition.
Supplemental charge used in all 105-mm rounds.
Some munitions quantities used in subsequent tests not covered in this report.

Table A-2. Munitions and explosives data—March tests.				
NSN	**DODIC**	**Nomenclature**	**Lot number**	**Quantity drawn**
1320012574222	D544	Projectile, 155 mm, M107, HE, w/o fuze	IOP03E100-011	14
1320014605087	D544	Projectile, 155 mm, M107, HE, w/o fuze	IOP02K025-005	7
1390010809447	N340	Fuze, point detonating, M739	MA-84B007-013	21
1375014151232	ML47	Cap, blasting non-electric, 30 Foot, M11	EBW97K060-008	24
1375014151231	MN03	Cap, blasting, non-electric, M13	ENB00M002-007	36
1375014151233	MN06	Cap, blasting, non-electric delay, M14	SHK98D001-001	24
1375001809356	M456	Cord, detonating, pentaerthyrie tetranitrate	EBG03A002-015	305 m
			ENB83H001-027	1830 m
1375014151235	MN08	Igniter, time blasting fuse with shock, M81	LNO98E001-003	25
1375007247040	M023	Charge, demolition block, Comp C4, M112	MA-97A003-007A	30

Notes: Drawn from Fort Richardson Ammo Supply Point.

Data from DA Form 581—Request for Issue and Turn-in of Ammunition.

Supplemental charge used in all rounds.

Some munitions quantities used in subsequent tests not covered in this report.

Table A-3 Explosives loading for munitions used in protocol tests.

DODIC	Nomenclature	Load (g)	Consituent loads (g)			
			TNT	RDX	HMX	NG*
C256	Cartridge, 81 mm, HE, M374, w/fuze PD F	953	371	572		46
C445	Cartridge, 105 mm, M1, HE, w/o fuze	2086	812	1253		
	Supplementary charge (for fuze well: Howitzer rnds)	136	136			
D544	Projectile, 155 mm, M107, HE, w/o fuze	6955	2724	4620		
D544	Projectile, 155 mm, M107, HE, w/o fuze	6622	6622			
N340	Fuze, point detonating, M739 (used w/C445 & D544)			21		
N340	Fuze, point detonating, M567 (supplied w/C256)		<1	27		
ML47	Cap, blasting, M11, non-electric, 30-ft. shock tube	1.175		<1	<1	
MN03	Cap, blasting, M13	5.06				
MN06	Cap, blasting, non-electric delay, M14	11.73				
M456	Cord, detonating, pentaerthyrie tetranitrate (1000 ft)	2900				
MN08	Igniter, M81, time blasting fuse, shock tube capable	0.05				
M023	Charge, demolition, block, COMP C-4, 1.25 lb.	570		520		

*NG is found in the tail assembly of the mortar round and is normally reacted during firing.

APPENDIX B. QUALITY ASSURANCE PROCEDURES FOR PROTOCOL TESTS

The following tables contain a list of the QA procedures and their descriptions for each round or projectile detonated for the protocol and confirmation tests. The variability of the procedures for the tests reflects the development of the QA procedures during the course of the tests. In all, 139 QA tests consisting of over 9850 increments were performed on the 28 rounds used in these tests. A write-up of the recommended QA procedures for use with detonations on snow-covered ice will be presented in a future report.

Table B-1. QA Procedures conducted at each detonation—81-mm tests.

Detonation	Procedure	Description
81-1	Subsurface sampling Duplicate sampling Annular OTP sampling	One at each DSM surface sample Adjacent samples 0- to 3-m annulus
81-2	Duplicate sampling Annular OTP sampling	MIS and LIS 0- to 3-m annulus
81-3	Duplicate sampling Annular OTP sampling	DSMs, adjacents, and LIS 0- to 3-m annulus
81-4	Duplicate sampling Triplicate sampling	Adjacents LIS
81-5	Subsurface sampling Radial sampling Annular OTP sampling Radial OTP sampling	One at each DSM surface sample LIS (0- to 10-/10- to 20-/>20-m R zones) 0- to 3-m annulus 0- to 10-m R OTP from detonation point
81-6	Triplicate sampling	MIS
81-7	Triplicate sampling	LIS

Table B-2. QA Procedures conducted at each detonation—105-mm tests.		
Detonation	**Procedure**	**Description**
105-1	None	
105-2	None	
105-3	Radial sampling Annular OTP sampling	LIS (0- to 10-/10- to 20-/>20-m R zones) 0- to 3-m annulus
105-4	Triplicate sampling Gradient sampling	Laps LIS (gray-scale)
105-5	Duplicate sampling Triplicate sampling Annular OTP sampling	Adjacents MIS and LIS 0- to 3-m annulus
105-6	Subsurface sampling Duplicate sampling	One at each DSM surface sample LIS
105-7	Duplicate sampling Triplicate sampling Radial sampling Annular OTP sampling	Adjacents MIS LIS (0- to 10-/10- to 20-/>20-m R zones) 0- to 3-m annulus

Table B-3. QA Procedures conducted at each detonation—155-mm Comp-B tests.		
Detonation	**Procedure**	**Description**
155-1B	Duplicate sampling Radial OTP sampling	LIS 0- to 10- and 10- to 20-m radii from det point
155-2B	Triplicate sampling Annular OTP sampling	LIS 0- to 3-m annulus
155-3B	Adjacent sampling Subsurface sampling Duplicate sampling Annular OTP sampling	MIS Below each of one of the MIS samples LIS 0- to 3-m and 3- to 6-m annuli
155-4B	Triplicate sampling Annular OTP sampling	LIS 0- to 3-m annulus
155-5B	Adjacent sampling Subsurface sampling Duplicate sampling Annular OTP sampling	MIS Below each of one of the MIS samples LIS 0- to 3-m and 3- to 6-m annuli
155-6B	Triplicate sampling Annular OTP sampling	LIS 0- to 3-m annulus
155-7B	Triplicate sampling Radial OTP sampling	LIS 0- to 10- and 10- to 20-m radii from det point

Table B-4. QA Procedures conducted at each detonation—155-mm TNT tests.

Detonation	Procedure	Description
155-1	Triplicate sampling Gradient sampling Radial OTP sampling	LIS LIS 10- to 20-m radius from detonation point
155-2	Triplicate sampling Annular OTP sampling	LIS 0- to 3-m annulus
155-3	Adjacent sampling Subsurface sampling Duplicate sampling Annular OTP sampling	MIS Below each of one of the MIS samples LIS 0- to 3-m and 3- to 6-m annuli
155-4	Triplicate sampling Annular OTP sampling	LIS 0- to 3-m annulus
155-5	Adjacent sampling Subsurface sampling Duplicate sampling Annular OTP sampling	MIS Below each of one of the MIS samples LIS 0- to 3-m and 3- to 6-m annuli
155-6	Triplicate sampling Annular OTP sampling	LIS 0- to 3-m annulus
155-7	Triplicate sampling Gradient sampling Radial OTP sampling	LIS LIS 10- to 20-m radius from detonation point

Table B-5. List of all QA procedures conducted over the course of testing.			
Test type	**Round tested**	**# of reps.**	**Increments***
Replicate DSMs	81-3	1	1 @ 17 points
Subsurface—DSMs	81-1	1	1 @ 11 points
	81-5	1	1 @ 11 points
	105-6	1	1 @ 15 points
Adjacents—DSMs	81-1	2	11
	81-2	1	12
	81-3	1	17
	81-4	2	11
	81-5	1	11
	81-6	1	11
	81-7	1	11
	105-1	1	15
	105-2	1	15
	105-3	1	17
	105-4	1	15
	105-5	2	18
	105-6	1	15
	105-7	1	18
Replicates—DSM adjacents	81-1	1	1 @ 11 points
	81-4	1	1 @ 11 points
	105-5	1	1 @ 18 points
MIS	81-1	1	38
	81-2	2	36
	81-3	1	40
	81-4	1	34
	81-6	3	40
	81-7	1	40
	105-1	11	40
	105-2	1	40
	105-3	1	40
	105-4	3	41
	105-5	3	41
	105-6	1	32
	105-7	3	35
	155-3 Comp-B	1	40
	155-5 Comp-B	1	40

Table B-5 (cont'd). List of all QA procedures conducted over the course of testing.

Test type	Round tested	# of reps.	Increments*
MIS	155-3 TNT	1	40
	155-5 TNT	1	40
Adjacents—MIS	155-3 Comp-B	1	40
	155-5 Comp-B	1	40
	155-3 TNT	1	40
	155-5 TNT	1	40
LIS	81-1	1	109
	81-2	2	110
	81-3	2	103
	81-4	3	103
	81-6	1	73
	81-7	3	100
	105-1	1	100
	105-2	1	100
	105-5	3	120
	105-6	2	97
	155-1 Comp-B	1	100
	155-2 Comp B	2	100
	155-3 Comp-B	1	100
	155-4 Comp-B	2	105
	155-5 Comp-B	1	105
	155-6 Comp-B	2	111
	155-7 Comp-B	2	115
	155-1 TNT	2	105
	155-2 TNT	2	102
	155-3 TNT	1	100
	155-4 TNT	2	101
	155-5 TNT	1	100
	155-6 TNT	2	143
	155-7 TNT	1	96
LIS—Gray-scale zones	105-4	1	137
	155-1 TNT	1	269
	155-7 TNT	1	180
LIS—Radial zones	81-5	1	263
	105-3	1	141
	105-7	1	309

Table B-5 (cont'd).			
Test type	**Round tested**	**# of reps.**	**Increments***
OTP—0- to 3-m annulus	81-1	1	40
	81-2	1	41
	81-3	1	80
	81-5	1	120
	105-3	1	66
	105-5	1	82
	105-7	1	66
	155-2 Comp B	1	100
	155-3 Comp-B	1	100
	155-4 Comp-B	1	100
	155-5 Comp-B	1	100
	155-6 Comp-B	1	73
	155-2 TNT	1	100
	155-3 TNT	1	100
	155-4 TNT	1	100
	155-5 TNT	1	100
	155-6 TNT	1	100
OTP—3- to 6-m annulus	155-1 Comp-B	1	100
	155-7 Comp-B	1	100
	155-1 TNT	1	100
	155-7 TNT	1	100
OTP—0- to 10-m radius	81-5	1	35
	155-1 Comp-B	1	35
	155-7 Comp-B	1	50
OTP—10- to 20-m radius	155-1 Comp-B	1	73
	155-7 Comp-B	1	87
	155-1 TNT	1	98
	155-7 TNT	1	78

Notes: For 81-mm and 105-mm tests, the DSM protocol was the standard sampling protocol. For the 155-mm tests, the LIS protocol was the standard protocol. QA was performed to verify these protocols.

* The number of increments is the average per rep.

APPENDIX C. DATA FOR THE DISCRETE SAMPLING METHOD (DSM) TESTS

The following tables contain data generated from residues sampling during the DSM baseline test phase of this project. Three tests were conducted concurrently with the DSM tests: discrete sampling, subsurface sampling beneath the discrete sampling area, and sampling outside the demarcated plume using a large-increment composite sampling protocol. Each table is self-explanatory with the notes given at the bottom of the tables in the body of the report. Residue masses are given in milligrams.

Table C-1. Plume sampling statistics for DSM test (excludes crater samples).

Plume #	Plume (OTP) area (m^2)	Sample type	# Samples	Increments	Sampled area (m^2)	% of area sampled
81-1	1506	Discretes (m^2)	11	1 × 11	11	0.73%
		Subsurface (m^2)	11	1 × 11	11	—
	(489)	0- to 3-m OTP	1	40 × 1	1.6	0.33%
81-2	637	Discretes (m^2)	12	1 × 12	12	1.9%
	(354)	0- to 3-m OTP	1	41 × 1	1.6	0.46%
81-3	790	Discretes (m^2)	2 x 17	2 × 17	34	2.2%
	(378)	0- to 3-m OTP	1	80	0.80	0.21%
81-4	695	Discretes (m^2)	11	1 × 11	11	1.6%
81-5	693	Discretes (m^2)	11	1 × 11	11	1.6%
		Subsurface (m^2)	11	1 × 11	11	–
	(312)	0- to 3-m OTP	1	120 × 1	1.2	0.38%
	(129)	0- to 10-m R OTP	1	35 × 1	1.4	1.1%
81-6	741	Discretes (m^2)	11	1 × 11	11	1.5%
81-7	678	Discretes (m^2)	11	1 × 11	11	1.6%
Total			128	439		
Average*	720	Discretes (m^2)	13	13	13	1.6%
		Subsurface (m^2)	11	11	11	1.2%
	(380)	0- to 3-m OTP	1	79	1.6	0.42%
105-1	731	Discretes (m^2)	15	1 × 15	15	2.1%
105-2	443	Discretes (m^2)	15	1 × 15	15	3.4%
105-3	938	Discretes (m^2)	17	1 × 15	17	1.8%
	(402)	0- to 3-m OTP	1	66 × 1	0.66	0.16%
105-4	808	Discretes (m^2)	15	1 × 15	15	1.9%
105-5	872	Discretes (m^2)	18	1 × 18	18	2.1%
	(457)	0- to 3-m OTP	1	82 × 1	0.82	0.18%

Table C-1 (cont'd).						
Plume #	**Plume (OTP) area (m^2)**	**Sample type**	**# Samples**	**Increments**	**Sampled area (m^2)**	**% of area sampled**
105-6	1310	Discretes (m^2)	15	1 × 15	15	1.2%
		Subsurface (m^2)	15	1 × 15	15	—
105-7	946	Discretes (m^2)	18	1 × 18	18	1.9%
	(486)	0- to 3-m OTP	1	66 × 1	0.66	0.14%
Total			131	342		
Average*	864	Discretes (m^2)	16	16	16	1.9%
		Subsurface (m^2)	15	15	15	1.2%
	(450)	0- to 3-m OTP	1	71	0.71	0.16%

Note: Average for plumes where tests were conducted.

Table C-2. Estimated residues masses for DSM protocol tests.

Plume #	Sample type	HMX (mg)	RDX (mg)
81-1	Discretes (m^2)	11	20
	Subsurface (m^2)	1.4	3.7
	OTP 0–3 m	0.18	—
81-2	Discretes (m^2)	5.6	7.2
	OTP 0–3 m	—	—
81-3	Discretes (m^2)-a	1.1	8.1
	Discretes (m^2)-b	1.5	10
	OTP 0–3 m	—	—
81-4	Discretes (m^2)	57	470
81-5	Discretes (m^2)	7.3	31
	Subsurface (m^2)	1.0	4.0
	OTP 0–3 m	—	0.36
	OTP 0- to 10-m radius	—	—
81-6	Discretes (m^2)	65	220
81-7	Discretes (m^2)	31	92
105-1	Discretes (m^2)	2.8	13
105-2	Discretes (m^2)	5.0	18
105-3	Discretes (m^2)	6.9	33
	OTP 0–3 m	0.15	2.2
105-4	Discretes (m^2)	6.6	15
105-5	Discretes (m^2)	11	82
	OTP 0–3 m	0.25	2.4
105-6	Discretes (m^2)	11	25
	Subsurface (m^2)	0.68	1.6
105-7	Discretes (m^2)	5.3	17
	OTP 0–3 m	—	0.43

Table C-3. Detonation proximity and soot density bias data for DSM sampling.

Plume	Test	Zone	Area (m^2)	# Samples	% Samples	% area	Samples/area†
81-1	Concentric	0- to 10 m	184	4	36%	12%	3.0
		10- to 20 m	331	4	36%	22%	1.6
		>20 m	991*	3	27%	66%	0.41
81-2	Concentric	0- to 10 m	169	7	58%	27%	2.1
		10- to 20 m	231	4	33%	36%	0.92
		>20 m	237	1	8%	37%	0.22
81-3(a)	Concentric	0- to 10 m	187	6	35%	24%	1.5
		10- to 20 m	226	5	30%	29%	1.0
		>20 m	377	6	35%	47%	0.74
81-3(b)	Concentric	0- to 10 m	187	9	53%	24%	2.2
		10- to 20 m	226	6	35%	29%	1.2
		>20 m	377*	2	12%	47%	0.26
81-4	Concentric	0- to 10 m	144	4	36%	21%	1.7
		10- to 20 m	194	5	45%	28%	1.6
		>20 m	357*	2	18%	51%	0.35
81-5	Concentric	0- to 10 m	185	5	45%	27%	1.7
		10- to 20 m	272	5	45%	39%	1.2
		>20 m	235	1	9%	34%	0.26
81-6	Concentric	0- to 10 m	194	4	36%	26%	1.4
		10- to 20 m	313	6	55%	42%	1.3
		>20 m	234	1	9%	32%	0.28
81-7	Concentric	0- to 10 m	182	6	55%	27%	2.0
		10- to 20 m	261	5	45%	38%	1.2
		>20 m	236	0	0%	35%	0.00
105-3	Concentric	0- to 10 m	282	7	41%	30%	1.4
		10- to 20 m	393	7	41%	42%	1.0
		>20 m	263	3	18%	28%	0.64
105-4	Gray	Dark	97	4	27%	12%	2.2
		Medium	61	2	13%	8%	1.6
		Light	650	9	60%	80%	0.75
	Concentric	0- to 10 m	288	7	47%	36%	1.3
		10- to 20 m	401	7	47%	50%	0.94
		>20 m	121	1	6.7%	15%	0.45
105-7	Concentric	0- to 10 m	232	7	39%	25%	1.6
		10- to 20 m	357	5	28%	39%	0.72
		>20 m	347	6	33%	36%	0.92

* These plumes all extended beyond 30 m. No samples were taken in that zone.

† Samples/area is the ratio of the % samples to % area.

Table C-4. Detonation proximity bias in DSM residues estimates.

Plume	Test	Condition	HMX (mg)	RDX (mg)
81-1	Concentric	Weighted	5.5	10
		Standard DSM	11	20
		Difference	100%	100%
81-2	Concentric	Weighted	4.1	7.1
		Standard DSM	5.6	7.2
		Difference	37%	1.4%
81-3a	Concentric	Weighted	0.91	6.5
		Standard DSM	1.1	8.1
		Difference	21%	25%
81-3b	Concentric	Weighted	1.1	17
		Standard DSM	1.5	10
		Difference	36%	−42%
81-4	Concentric	Weighted	46	446
		Standard DSM	57	473
		Difference	24%	6.1%
81-5	Concentric	Weighted	4.7	19
		Standard DSM	7.3	31
		Difference	55%	63%
81-6	Concentric	Weighted	43	170
		Standard DSM	55	220
		Difference	28%	29%
81-7	Concentric	Weighted	19	56
		Standard DSM	31	92
		Difference	63%	64%
Average difference: 81-mm			46%	31%
105-3	Concentric	Weighted	5.6	27
		Standard DSM	6.9	33
		Difference	23%	22%
105-4	Concentric	Weighted	5.1	11
		Standard DSM	6.6	15
		Difference	29%	36%
105-7	Concentric	Weighted	4.1	15
		Standard DSM	5.3	17
		Difference	29%	13%
Average difference: 105-mm			27%	24%
Average overall difference			38%	28%

Note: Weighted values take proximity to the detonation point into account, weighting for the area in the concentric band in which the samples fall. There are three concentric bands within the plume: 0- to 10-*m* R., 10- to 20-*m* R., and >20-*m* R. Standard DSM values "composite" all the DSM samples without regard to location within the plume. The difference is the percent difference between the weighted and standard (unweighted) results: ([Standard−Weighted]/Weighted)

APPENDIX D. DATA FOR PROTOCOL TESTS

The following tables contain data generated from residues sampling during the protocol test phase of this project. Three tests were conducted concurrently with the DSM tests: Sampling adjacent to the DSM sample, plume characterization using a medium-increment composite sampling protocol, and plume characterization using a large-increment composite sampling protocol. Each table contains the number of samples taken, the increments per sample, the total area for each sample, and the percent of the demarcated plume sampled. Residues masses are given in milligrams.

Table D-1. Data for adjacent samples.

Plume #	# Samples	Increments	Sampled area (m^2)	% of plume sampled	HMX mass (mg)	RDX mass (mg)
81-1	2	11 each	0.44 each	0.03%	15	23
					6.8	14
81-2	1	12	0.48	0.08%	9.4	4.9
81-3	2	17 each	0.68	0.09%	1.6	7.8
					2.2	16
81-4	2	11 each	0.44 each	0.06%	20	640
					97	720
81-5	1	11	0.44	0.06%	10	45
81-6	1	11	0.44	0.06%	67	280
81-7	1	11	0.44	0.06%	45	130
105-1	1	15	0.60	0.08%	6.1	11
105-2	1	15	0.60	0.14%	6.6	14
105-3	1	17	0.68	0.07%	9.6	17
105-4	1	15	0.60	0.07%	8.4	18
105-5	2	18	0.72 each	0.08%	29	180
					24	54
105-6	1	15	0.60	0.05%	17	32
105-7	2	18	0.72 each	0.08%	10	23
					8.5	12

Table D-2. Data for medium-increment (MIS) samples.						
Plume #	# Samples	Increments	Sampled area (m^2)	% of plume sampled	HMX mass (mg)	RDX mass (mg)
81-1	1	38	1.5	0.10%	2.9	3.6
81-2	2	35	1.4	0.22%	2.2	3.2
		37	1.5	0.24%	2.9	6.0
81-3	1	40	1.6	0.20%	1.1	8.4
81-4	1	34	1.5	0.22%	58	480
81-6	3	40	1.6	0.22%	75	335
		40	1.6	0.22%	55	290
		40	1.6	0.22%	65	220
81-7	1	40	1.6	0.24%	16	43
105-1	1	40	1.6	0.22%	2.3	11
105-2	1	40	1.6	0.36%	4.6	16
105-3	1	40	1.6	0.17%	6.3	20
105-4	3	40	1.6	0.20%	5.1	10
		40	1.6	0.20%	8.9	23
		42	1.7	0.21%	4.9	11
105-5	3	42	1.7	0.19%	9.9	104
		40	1.6	0.18%	9.2	74
		40	1.6	0.18%	9.6	540
105-6	1	32	1.3	0.10%	8.6	17
105-7	3	31	1.2	0.13%	7.7	14
		36	1.4	0.15%	7.6	19
		39	1.6	0.17%	5.1	12

Table D-3. Data for large-increment (LIS) samples.						
Plume #	# Samples	Increments	Sampled area (m^2)	% of plume sampled	HMX mass (mg)	RDX mass (mg)
81-1	1	109	1.1	0.07%	6.3	12
81-2	2	106	1.1	0.17%	2.7	2.9
		113	1.1	0.17%	7.2	7.2
81-3	2	104	1.0	0.13%	0.92	11
		102	1.0	0.13%	2.3	29
81-4	3	100	1.0	0.14%	66	520
		100	1.0	0.14%	65	540
		110	1.1	0.16%	40	370
81-5	1	263	2.6	0.38%	3.2	22
81-6	1	73	0.73	0.10%	56	270
81-7	3	100	1.0	0.15%	17	42
		100	1.0	0.15%	15	44
		100	1.0	0.15%	12	33
105-1	1	100	1.0	0.14%	4.4	6.9
105-2	1	100	1.0	0.23%	2.8	14
105-3	1	141	1.4	0.15%	8.3	25
105-4	1	137	1.4	0.17%	2.8	7.6
105-5	3	105	1.1	0.13%	22	150
		111	1.1	0.13%	28	200
		144	1.4	0.17%	28	190
105-6	2	97	0.97	0.07%	15	30
		97	0.97	0.07%	10	18
105-7	1	309	3.1	0.33%	6.5	19

APPENDIX E. DATA FOR IMPLEMENTATION TESTS

The following two tables contain data derived from the implementation tests. In these tables, M_R is the mass of the residues collected in the sample (both filtrate and soot fractions), C_S is the surface concentration calculated for the area sampled, and M_T is the total mass calculated for either the crater, the plume, or the area outside the plume sampled. Italics indicates extract concentrations of the analytes below or near (+50%) the detection limit (30 µg/L) of the analytical method. Residues are given in micrograms.

Table E-1. Estimated total residues masses for detonation tests—155-mm Comp-B rounds.

Sample type	Areas (m^2) Plume/sample	HMX M_R (µg)	HMX C_S (µg/m^2)	HMX M_T (mg)	RDX M_R (µg)	RDX C_S (µg/m^2)	RDX M_T (mg)	TNT M_R (µg)	TNT C_S (µg/m^2)	TNT M_T (mg)
Plume 1	**1275**									
Crater	0.90	8.4	9.4	0.13	13	15	0.21	ND	—	—
LIS	0.94	*1.3*	*1.4*	*1.8*	7.3	7.7	9.8	ND	—	—
LIS	1.00	*2.6*	*2.6*	*3.3*	17	17	21	ND	—	—
OTP-10R	0.35	*0.67*	*1.9*	*2.4*	23	66	3.7	ND	—	—
OTP-20R	0.73	ND	—	—	0.71	0.97	0.37	ND	—	—
Plume 2	**1731**									
Crater	0.30	3.6	12	.16	12	41	0.56	ND	—	—
LIS	1.00	ND	—	—	3.1	3.1	5.4	ND	—	—
LIS	1.00	ND	—	—	*1.2*	*1.2*	*2.1*	ND	—	—
LIS	1.00	ND	—	—	3.3	3.3	5.8	ND	—	—
OTP-3A	2.25	ND	—	—	ND	—	—	ND	—	—
Plume 3	**1835**									
MIS	0.90	ND	—	—	1.9	4.8	8.8	ND	—	—
MIS	0.90	ND	—	—	3.6	4.0	7.3	ND	—	—
Sub-surface	0.40	ND	—	—	ND	—	—	ND	—	—
LIS	1.00	ND	—	—	1.3	1.3	2.3	ND	—	—
LIS	1.00	2.0	2.0	3.7	2.6	2.6	4.8	ND	—	—
OTP-3A	1.00	ND	—	—	ND	—	—	ND	—	—
OTP-6A	1.00	ND	—	—	ND	—	—	ND	—	—

Table E-1 (cont'd).

Sample type	Areas (m^2)	HMX			RDX			TNT		
	Plume/ sample	M_R (µg)	C_S (µg/m^2)	M_T (mg)	M_R (µg)	C_S (µg/m^2)	M_T (mg)	M_R (µg)	C_S (µg/m^2)	M_T (mg)
Plume 4	**1654**									
Crater	0.66	14	20	0.32	73	110	1.7	1.4	2.1	0.03
LIS	1.04	*3.3*	*3.1*	*5.2*	33	32	53	ND	—	—
LIS	1.06	ND	—	—	15	14	23	ND	—	—
LIS	1.05	ND	—	—	4.3	4.1	6.7	ND	—	—
OTP-3A	2.25	ND	—	—	ND	—	—	ND	—	—
Plume 5	**1638**									
MIS	0.90	ND	—	—	5.4	6.0	9.9	ND	—	—
MIS	0.90	ND	—	—	5.5	6.1	10	ND	—	—
Sub-surface	0.40	ND	—	—	ND	—	—	ND	—	—
LIS	1.00	ND	—	—	21	21	34	ND	—	—
LIS	1.10	*0.48*	*0.44*	*0.71*	22	20	32	ND	—	—
OTP-3A	1.00	ND	—	—	ND	—	—	ND	—	—
OTP-6A	1.00	ND	—	—	ND	—	—	ND	—	—
Plume 6	**1656**									
Crater	0.30	2.7	8.8	0.12	8.7	29	0.38	ND	—	—
LIS	1.10	ND	—	—	*0.39*	*0.35*	*0.59*	ND	—	—
LIS	1.05	ND	—	—	*2.9*	*2.8*	*4.6*	ND	—	—
LIS	1.28	ND	—	—	*0.44*	*0.34*	*0.57*	ND	—	—
OTP-3A	0.73	ND	—	—	ND	—	—	ND	—	—
Plume 7	**1556**									
Crater	0.50	0.75	1.5	0.02	8.9	18	0.22	ND	—	—
LIS	1.09	ND	—	—	3.5	3.2	5.0	ND	—	—
LIS	1.30	*1.5*	*1.2*	*1.8*	31	24	37	ND	—	—
LIS	1.00	ND	—	—	19	19	29	ND	—	—
OTP-10R	0.87	ND	—	—	ND	—	—	ND	—	—
OTP-20R	0.50	ND	—	—	ND	—	—	ND	—	—

Table E-2. Estimated total residues masses for detonation tests—155-mm TNT rounds.										
	Areas (m^2)	HMX			RDX			TNT		
Sample type	Plume/ sample	M_R (µg)	C_S (µg/m^2)	M_T (mg)	M_R (µg)	C_S (µg/m^2)	M_T (mg)	M_R (µg)	C_S (µg/m^2)	M_T (mg)
Plume 1	**1234**									
LIS-1	1.1	1.8	1.6	2.0	9.5	8.7	11	2.9	2.6	3.3
LIS-2	0.91	ND	—	—	2.6	2.8	3.4	4.2	4.2	5.2
LIS-3	0.52	2.4	2.4	2.9	3.0	3.0	3.7	15	15	18
LIS-Lt	1.26	ND	0	0	ND	—	—	16.2	13	11
LIS-Med	0.91	6.2	6.8	2.2	12	13	4.2	3.6	4.0	1.3
LIS-Dk	0.52	ND	—	—	ND	—	—	2.3	4.3	0.30
OTP-20R	0.98	ND	—	—	ND	—	—	2.92	3.0	1.1
Plume 2	**1600**									
Crater	0.20	1.4	7	1.5	4.5	22	0.27	6	30	0.36
LIS-1	1.0	1.8	1.8	2.9	4.7	4.7	7.5	6.5	6.5	10
LIS-2	1.0	1.5	1.4	2.3	3.7	3.6	5.7	3	3.0	4.7
LIS-3	1.0	2.1	2.1	3.3	4.7	4.7	7.5	12	12	19
OTP-3A	1.0	2.0	2.0	1.5	ND	—	—	10	10	7.3
Plume 3	**1311**									
MIS	0.90	1.7	1.9	2.5	5.8	6.4	8.4	5.8	6.5	8.5
MIS	0.90	ND	—	—	2.4	2.6	3.4	12	13	17
Sub-surface	0.40	ND	—	—	ND	—	—	63	160	210
LIS-1	1.0	ND	—	—	5.4	5.43	7.1	3.2	3.2	4.2
LIS-2	1.0	1.5	1.5	1.9	7.0	7.0	9.2	3.7	3.7	4.8
OTP-3A	1.0	ND	—	—	ND	—	—	7.8	7.8	4.5
OTP-6A	1.0	ND	—	—	ND	—	—	4.8	4.8	3.0
Plume 4	**1201**									
LIS-1	1.0	ND	—	—	ND	—	—	15	10	1.3
LIS-2	1.0	ND	—	—	ND	—	—	4.7	4.7	5.6
LIS-3	1.4	ND	—	—	ND	—	—	5.3	5.2	6.3
OTP-3A	1.0	ND	—	—	ND	—	—	23	23	11
Plume 5	**1108**									
MIS	0.9	ND	—	—	ND	—	—	5	5.5	6.1
MIS	0.90	ND	—	—	ND	—	—	4.6	5.1	5.6
Sub-surface	0.40	ND	—	—	ND	—	—	2	4.9	5.4
LIS-1	1.0	ND	—	—	ND	—	—	7.4	7.2	8.0
LIS-2	1.0	ND	—	—	ND	—	—	4.3	4.3	4.7

Table E-2 (cont'd).										
	Areas (m^2)	HMX			RDX			TNT		
Sample type	Plume/ sample	M_R (µg)	C_S (µg/m^2)	M_T (mg)	M_R (µg)	C_S (µg/m^2)	M_T (mg)	M_R (µg)	C_S (µg/m^2)	M_T (mg)
OTP-3A	1.0	ND	—	—	ND	—	—	1.6	1.6	0.82
OTP-6A	1.1	ND	—	—	ND	—	—	1	0.94	0.52
Plume 6	**1375**									
Crater	0.20	ND	—	—	ND	—	—	12	61	0.89
LIS-1	1.3	ND	—	—	4.0	3.4	4.7	1.3	1.1	1.5
LIS-2	1.2	ND	—	—	1.9	1.5	2.0	2	1.6	2.2
LIS-3	1.6	5.1	3.2	4.4	7.1	4.5	6.1	3.8	2.3	3.2
OTP-3A	1.0	ND	—	—	2.2	2.2		2.3	2.3	1.2
Plume 7	**1180**									
LIS-1	1.1	ND	—	—	4.0	4.2	4.9	4.1	4.2	5.0
LIS-2	1.0	2.6	2.5	3.0	17	16	19	3.4	3.2	3.8
LIS-Lt	0.83	ND	—	—	ND	—	—	4.2	5.1	3.9
LIS-Med	0.52	2.3	4.4	1.4	2.8	5.4	1.7	5.1	9.8	3.1
LIS-Dk	0.45	3.2	7.1	0.69	94	94	9.1	6.7	15	1.4
OTP-20R	0.78	ND	—	—	ND	—	—	4.1	5.2	1.9

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14. ABSTRACT

Range contamination and sustainability are major issues for the United States military. Training is a critical factor in force readiness, and the availability of ranges is crucial to this need. To determine the impact of training on ranges, data are required on the deposition of explosives residues from live-fire and blow-in-place detonation of munitions. A method of sampling on snow-covered ranges, the discrete sampling method, was developed by the Army's Cold Regions Research and Engineering Laboratory to determine residues from the detonation of munitions. Although very effective, it requires the collection of many large samples, resulting in labor-intensive field operations and much processing and analysis work in the laboratory. By examining sampled locations within detonation plumes, it appears that collection bias may be affecting the results. There was also no methodology for quality assurance in the collection of the samples. We have examined the process currently in use and carried out a series of experiments to determine whether bias and sample quality issues are present in the sampling technique. Alternative methods of sample collection that afford a greater opportunity for quality control were examined and compared to the discrete sampling method. The recommended alternative sampling protocol is to collect multi-increment samples, and experimental results using this method are presented.

15. SUBJECT TERMS

Bias	Howitzer	Quality assurance	Snow
Blow-in-place	Mortar	Residues	
Explosives constituents	Munitions	Sampling	

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Posted on Fri, Dec. 09, 2005

U.S. Army wants to dig up soil in arsenal burn zone

Plan, backed by EPA, to be subject of public hearing on Dec. 20

By Bob Downing
Beacon Journal staff writer

The U.S. Army would like to excavate and clean up a contaminated site at the now-closed Ravenna Army Ammunition Plant.

Digging up the 200-acre site, known as the Winklepeck Burning Ground, would probably cost $1.2 million, said Army spokesman Irv Venger.

The Army is considering two options: digging up the contaminated soil or doing nothing with the site.

Digging up the soil, a plan developed in consultation with the Ohio Environmental Protection Agency, is the Army's preferred choice, Venger said.

The 14-page plan was developed for the Army by Tennessee-based Science Applications International Corp., a consulting firm.

A public hearing on the two options will be held Dec. 20 at the Newton Falls Community Center auditorium, 52 E. Quarry St., Newton Falls. There will be an open house at 5 p.m. and a public meeting at 6 p.m.

The report is available for review at the Reed Memorial Library, 167 E. Main St., Ravenna, and at the Newton Falls Public Library, 204 S. Canal St., Newton Falls. It is also available at www.rvaap.org.

The public comment period ends Jan. 8. Comments may be sent to Irv Venger, Acting RAAP Facility Manager, Building 1037, RVAAP, 8451 State Route 5, Ravenna, OH 44266.

``Our goal over the next month is to listen to what the public has to say about the alternatives that are presented in this proposed plan," Venger said. ``Public input is important for the final remedy selection and full consideration will be given to all public comments."

The Winklepeck Burning Ground is where the Army burned bulk explosives along with trash from the arsenal, where munitions were produced for World War II and the Korean and Vietnam wars.

The site -- about 200 yards wide and one mile long near the center of the facility -- would be excavated up to 4 feet deep for explosives, heavy metals and volatile organic compounds, Venger said.

The Army intends to remove the contaminated soil and to characterize the samples. Then the soil, depending on contamination levels, would be used on-site as backfill or be shipped off-site for disposal at an approved facility.

The burning ground is one of 53 contaminated sites at the 21,419-acre facility in eastern Portage and western Trumbull counties.

The Army intends to spend an estimated $70 million to clean up the facility, which has largely been turned over to the Ohio National Guard for training.

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