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February 2006

ERDC/CRREL TR-06-2

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## ABSTRACT

Field sampling experiments were conducted at an Air Force live-fire bombing range. The main objective was to assess the effectiveness of using a systematic-random, multi-increment sampling strategy for the collection of representative surface soil samples in areas where bombing practice is conducted with bombs containing high explosives. Replicate surface soil samples were collected within several craters and in different sized grids (1 m × 1 m, 10 m × 10 m, and 100 m × 100 m). One area sampled had been impacted by a low-order 2000-lb bomb detonation and several hundred small chunks of tritonal were present on the surface. Another area sampled had many fewer recognizable chunks of tritonal on the surface. An arroyo, located downslope of the heaviest impacted area of this live-fire range, where runoff from the area would be captured, was also sampled at several locations. TNT was the major energetic compound present within the live-fire bombing area. Short-range heterogeneity in TNT concentrations was very large and the ability to estimate mean concentration using discrete samples, even for an area as small as 1 m<sup>2</sup>, was poor. Much more reproducible estimates of mean concentrations for areas as large as 100 m × 100 m were achieved using multi-increment samples collected with a stratified systematic-random sampling design compared with that achieved using discrete samples. Results from soil profile samples and samples from the arroyo draining this area indicate that the energetic compounds present at the bombing range are not migrating from the site.

Another area sampled was a small demolition range where C4 explosive is used to ensure that practice bombs contain no residual explosive prior to removing scrap metal from the range. RDX and HMX were the energetic compounds detected at the highest concentration in surface soil at the demolition range. These compounds originated from the demolition explosive.

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## PREFACE

This report was prepared by Dr. Thomas F. Jenkins and Alan D. Hewitt, Environmental Sciences Branch, U.S. Army Engineer Research and Development Center (ERDC), Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire; Charles A. Ramsey, EnviroStat, Inc., Fort Collins, Colorado; Kevin L. Bjella, Applied and Military Engineering Branch, ERDC-CRREL; Susan R. Bigl, Geophysical Sciences Branch, ERDC-CRREL; and Dennis J. Lambert, Engineering Resources Branch, ERDC-CRREL.

The authors acknowledge the financial support for this work by the Strategic Environmental Research and Development Program (SERDP), Mr. Bradley Smith, Executive Director, Dr. Jeffrey Marqusee, Technical Director, and Dr. Robert Holst and Dr. Andrea Leeson, project monitors. Dr. Judith C. Pennington, ERDC Environmental Laboratory (EL), Vicksburg, Mississippi, was Principal Investigator for SERDP Project CP1155. Additional funding was received from U.S. Army Corps of Engineers program "Characterization, Evaluation, and Remediation of Distributed Source Compounds (UXO-C) on Army Ranges." The Technical Director and Program Manager for this work is Dr. John M. Cullinane, ERDC-EL.

This report was reviewed by Dr. C.L. Grant, Professor Emeritus, University of New Hampshire, and Marianne E. Walsh, Environmental Engineer, ERDC-CRREL.

Nancy Perron of CRREL is acknowledged for her assistance in laboratory analysis of soil samples from this range.

Finally, the authors acknowledge the assistance of Larry Hoppes, Range Manager at Holloman Air Force Base, New Mexico, for his logistical support of this work, which could not have been conducted without his generous assistance and support. Technical Sergeant Scott Barclay is also acknowledged for providing explosive ordnance disposal (EOD) support for this study and for collecting soil samples from the demolition range after the mid-May detonation.

This report was prepared under the general supervision of Rae A. Melloh, Acting Chief, Environmental Sciences Branch, CRREL; Dr. Lance D. Hansen, Deputy Director, CRREL; and James L. Wuebben, Acting Director, CRREL.

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

# Sampling Studies at an Air Force Live-Fire Bombing Range Impact Area

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## 1 INTRODUCTION

Air Force ranges are very large, generally hundreds of square kilometers in size, but the areas used for training with high-explosives-containing bombs is much smaller, generally only tens of hectares. Very little research has been conducted at live-fire bombing ranges to assess the levels of residue accumulation, to investigate the best approach for collecting representative soil samples, or to study methods used to process and subsample these soil samples in the laboratory.

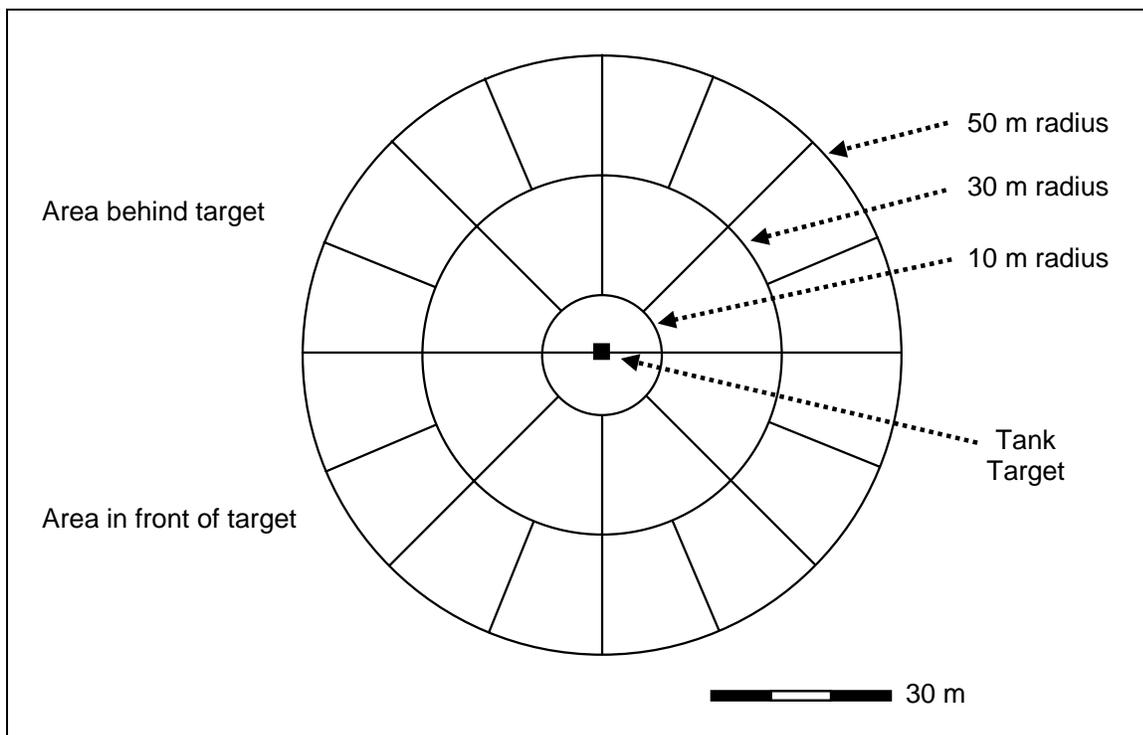
Because bombs contain a much larger mass of explosive than artillery rounds, craters from bomb detonations are very large. The Air Force conducts regularly scheduled range maintenance activities during which craters are often filled, duds are detonated using C4 explosive, and larger-than-golf-ball-size chunks of high explosive observed on the surface are gathered and destroyed by detonating with C4.

The high explosive present in U.S. and Canadian Air Force bombs is usually either tritonal (TNT, aluminum powder) or H-6 (TNT, RDX, aluminum powder). Some older bombs contained TNT only. Although experiments have not been conducted to document the residue deposited when a bomb detonates as designed, experimental results for large artillery rounds indicate that large-mass high-explosive detonations are very efficient, dispersing only microgram-to-milligram quantities of residue when they explode with a high-order detonation (Hewitt et al. 2003, M.R. Walsh et al. 2005).

One Canadian Air Force installation where research has been conducted is Cold Lake Air Weapons Range (CLAWR) in Alberta, Canada. At CLAWR, Ampleman et al. (2003, 2004) collected soil samples at the Shaver River Range, the only live-fire bombing range on the installation. Here 250-, 500-, and 1000-lb

high-explosive-containing bombs are dropped regularly at a stationary tank target and the surface of the range is tilled to reduce vegetation, thus alleviating the risk of forest fires in this remote area.

To study the distribution of energetic residues around the target at this range, Ampleman used a segmented halo sampling design. Three rings at radii of 10, 30, and 50 m around the single target were established and subdivided into 26 sections as shown in Figure 1. Two of these sections were 157 m<sup>2</sup> and the others were 314 m<sup>2</sup> in area. Thirty-increment surface soil samples (0- to 5-cm depth) were collected in each of the 26 sections (Fig. 2). Several field replicate samples also were collected. A 10-m × 10-m grid also was established about 15 m from the target. Four replicate 30-increment samples were collected in this grid using a totally random sampling design. This grid also was subdivided into 100 1-m × 1-m cells and a single discrete sample was collected in each. Also, a set of seven discrete surface soil samples was collected using a wheel sampling pattern (Jenkins et al. 1997) to further investigate the short-range heterogeneity in residue concentration near the target.



**Figure 1. Sampling design used by Ampleman et al. (2003) at Cold Lake Air Weapons Range live-fire bombing range, resulting in 26 sections around a target.**



**Figure 2. Establishing circular sampling pattern surrounding a tank target at the live-fire bombing range impact area at Cold Lake Air Weapons Range.**

TNT was found to be the residue present at the highest concentration at the Shaver River Range. RDX concentrations generally were below detection in these samples with only an occasional detection at low concentration. Because of the lack of RDX in these samples, we believe that the residue observed at CLAWR is from tritonal-containing bombs that underwent low-order detonations. Communication with personnel at CLAWR indicates that several low-order bomb detonations are observed each year at the Shaver River Range.\* Several small pieces of tritonal were observed on the surface of this range during sampling.

TNT concentrations among the 26 multi-increment section samples around the target ranged from 2.2 to 408 mg/kg for samples collected in 2002 (Ampleman et al. 2003) and from 1.3 to 165 mg/kg for samples collected in 2003

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\* Personal communication, Jeffrey Lewis, DRDC–Valcartier, 2005.

(Ampleman et al. 2004). Duplicate soil samples for nine different sections were collected over these two years, and the TNT concentrations for the duplicates were always within a factor of three, except for one sample collected in 2003, where they differed by a factor of 7.2. The mean concentration for four replicate samples for the 10-m  $\times$  10-m grid was 10.7 mg/kg, with a relative standard deviation (RSD) of 5.5%. The TNT concentrations in the 100 discrete samples varied from 0.38 to 290 mg/kg within this same 10-m  $\times$  10-m area. The range in TNT concentration for the seven discrete samples varied from 6.6 to 62 mg/kg, even though these samples were collected within a 1.5-m-diameter circle.

The level of agreement among replicate multi-increment samples at this range was excellent; this may be the result of a management practice in which the surface of the soil is periodically tilled. Doing so tends to mix the soil and homogenize the residue concentrations better than if tilling were not done. Even so, the variability among discrete samples collected in close proximity indicates that short-range heterogeneity remains very large.

## 2 OBJECTIVES

This research project was conducted to assess the reproducibility of a sampling strategy employing a systematic-random design and multi-increment samples for the collection of representative soil samples for grids as large as 10,000 m<sup>2</sup> (one hectare) where high-explosives-containing bomb detonations occur. Samples also were obtained from a number of large craters, and from an arroyo downhill of the live-fire bombing ranges. The arroyo samples were used to assess whether surface runoff could transport residues of energetic compounds off range. An additional set of samples was obtained to estimate the level of accumulation of residues at a demolition range where practice bombs are detonated with C4 to ensure that they contain no energetic compounds prior to recycling of the metal casings.

### 3 METHODS

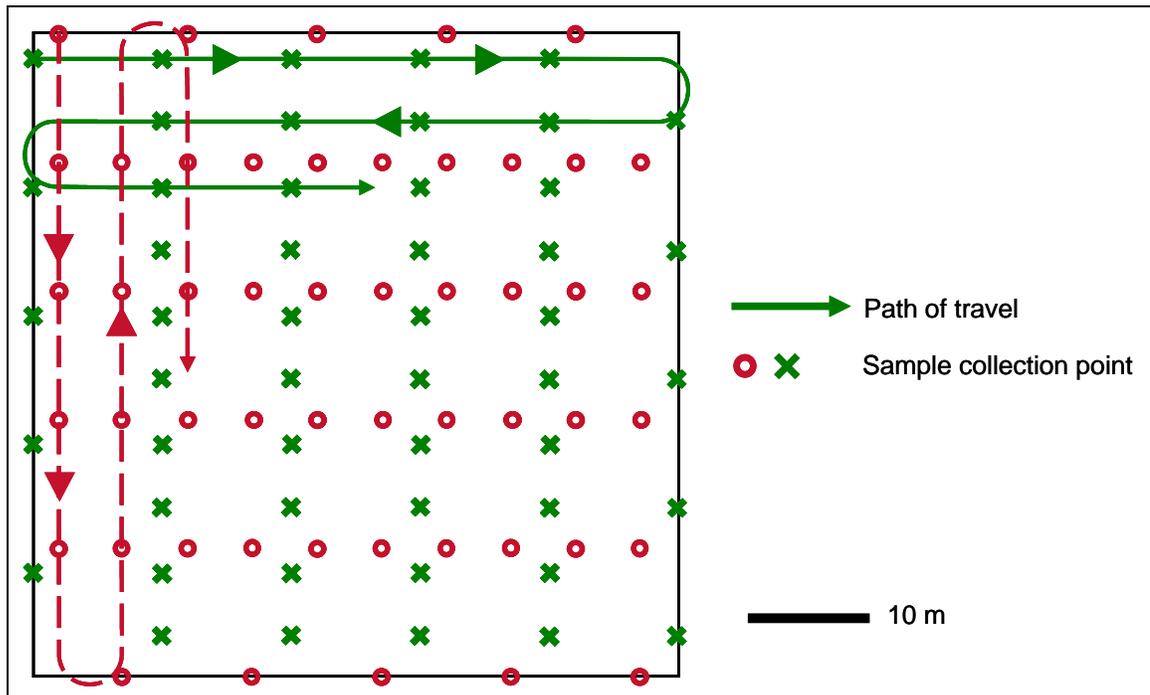
#### Soil sampling

Soil sampling was conducted at the live-fire bombing range impact area and the demolition range at Holloman AFB, Alamogordo, New Mexico, from 3 to 5 May 2005. GPS positions for all sampling locations were obtained using a Trimble ProXR global positioning system with a TSC1 data collector, which has about 1-m accuracy. Several samples also were collected at the demolition range. A second set of samples from the demolition range was collected on 11 May 2005.

Soil samples were collected from several-sized grids (1 m × 1 m, 10 m × 10 m, 100 m × 100 m) within craters and along an arroyo that was downslope of the impact area on a live-fire bombing range. Several additional samples were collected at a small demolition range. All soil samples were collected using metal scoops (AMS). The scoops were cleaned with deionized water, wiped with a disposable paper towel, and rinsed with acetone between samples.

Within craters, arroyos, square grids, and at the demolition range, soil samples were collected by combining multiple increments from the surface to a 2.5-cm depth and placed in clean polyethylene bags (KNF Clean Room Products Corporation). The number of increments for a given sample varied from 30 to 100 depending on the size of the area being sampled. Sample masses varied from 1 to 5 kg.

Individual increments within a grid were collected using a systematic sampling pattern with a random starting point (Hewitt et al. 2005). This sampling design is referred to as a systematic-random design. This was accomplished by walking from one corner of the grid systematically back and forth across the entire grid area, collecting an increment of soil every so many paces, depending on the grid size and number of increments to be collected (Fig. 3). Within arroyos, samples were collected linearly along the bottom in areas where deposition of particles from runoff was expected, and where standing water would tend to deposit solutes as the water evaporated. Individual increments were collected from a 10-m length, 5 m on either side of the designated location. Craters were sampled by starting at a random location at the top edge and proceeding in a spiral pattern from the top to bottom, collecting individual increments from the side walls and bottom.

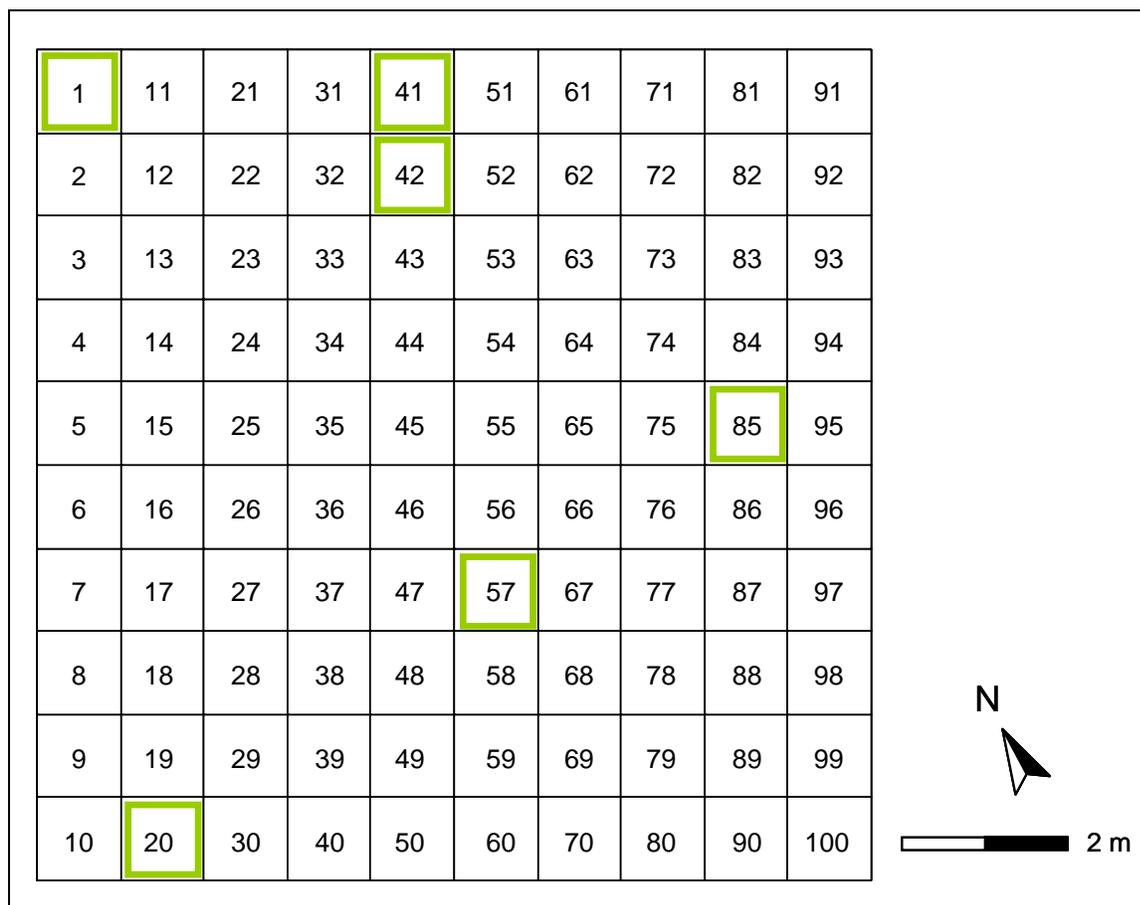


**Figure 3. Systematic-random 50-increment sampling pattern used for collecting samples in grid areas.**

One 10-m  $\times$  10-m grid was divided into 100 cells (1 m  $\times$  1 m) and we collected a discrete soil sample from an area near the center of each cell (Fig. 4). Within six of these 1-m  $\times$  1-m cells, we also collected nine separate discrete surface soil samples. We divided a second 10-m  $\times$  10-m grid into 25 2-m  $\times$  2-m cells and collected a discrete sample near the center of each cell. All discrete soil samples were collected from the top 2.5 cm of soil and placed in Ziploc plastic bags. Several profile samples also were collected within these cells and in the lowest lying location within the arroyo at depths as deep as 40 cm below surface. These samples were collected using stainless steel scoops. After each depth increment was collected, the soil was carefully swept away from the hole to minimize any deposition from above as deeper soil was collected.

### Sample processing and subsampling

All soil samples were returned to CRREL by overnight carrier. Multi-increment soil samples were spread out on trays and allowed to air dry. Discrete samples were placed in 4-oz glass jars and air dried. Each sample was then passed through a 10-mesh (2-mm) sieve in its entirety to remove oversized material.



**Figure 4. Layout of the 100 cells (1-m x 1-m) within Grid A near 2000-lb bomb crater at Holloman Air Force Base. Green-outlined cells were selected for collection of nine discrete samples within each 1-m cell.**

For the multi-increment samples, the entire <2-mm fraction was ground on a Lab TechEssa LM2 (LabTech Essa Pty. Ltd., Bassendean, WA, Australia) puck-mill grinder for 90 seconds, thereby reducing the particle size of the material to a flour (<75  $\mu\text{m}$ ). After grinding, samples were mixed thoroughly and spread to form a 1-cm-thick layer, and subsamples were obtained by collecting 30 increments randomly through the entire thickness of the layer of ground material. Each subsample (about 10 g) was placed in a 2-oz jar and extracted on a shaker table for 18 hours using 20 mL of acetonitrile (AcN).

For the discrete samples, the entire <2-mm portion was weighed in a 4-oz jar and a volume of AcN (in mL) about twice the mass of soil (in g) was added. These samples were also extracted on the shaker table for 18 hours. All extracts were filtered by passing each through a Millex-FH PTFE 0.45- $\mu\text{m}$  syringe filter

(Millipore Corp.). For GC-ECD analysis, this extract was injected without further dilution. For RP-HPLC-UV analysis, this extract was diluted 1 to 4 with deionized water to match the solvent strength of the HPLC eluent.

### Extract analysis

All sample extracts were analyzed using RP-HPLC-UV according to the general procedures outlined in EPA SW846 Method 8330 (USEPA 1994). Analysis was conducted on a modular RP-HPLC system from Thermo Finnigan composed of a SpectraSYSTEM Model P1000 isocratic pump, a SpectraSYSTEM UV2000 dual wavelength UV/VS absorbance detector set at 210 and 254 nm (cell path 1 cm), and a SpectraSYSTEM AS300 autosampler. Samples were introduced by overfilling a 100- $\mu$ L sampling loop. Separations were made on a 15-cm  $\times$  3.9-mm (4- $\mu$ m) NovaPak C-8 column (Waters Chromatography Division, Milford, Massachusetts) maintained at 28°C and eluted with 15:85 isopropanol/water (v/v) at 1.4-mL/min. Concentrations were estimated from peak heights compared to commercial multi-analyte standards (Restek). Reporting limits for RP-HPLC-UV analyses on a soil weight basis were 0.01 mg/kg for all target analytes. The target analytes for RP-HPLC-UV analyses were the 14 energetic compounds of Method 8330 with the addition of nitroglycerin and PETN.

Selected sample extracts were analyzed by GC-ECD according to EPA SW846 Method 8095 (USEPA 1999). These were either extracts where analyte concentrations were near or below the detection limits for the RP-HPLC-UV method, or where additional analyte confirmation was desired.

The GC-ECD analyses were conducted on an HP 6890 Gas Chromatograph equipped with a micro ECD detector. Direct injection of 1  $\mu$ L of soil extract was made into a purged packed inlet port (250°C) equipped with a deactivated Restek Uniliner. Primary separation was conducted on a 6-m-  $\times$  0.53-mm-ID fused-silica column, with a 0.5- $\mu$ m film thickness of 5% diphenyl-95% dimethyl polysilicate (Rtx-5, Restek, Bellefonte, Pennsylvania). The GC oven was temperature-programmed as follows: 100°C for 2 min, 10°C/min ramp to 280°C. The carrier gas was hydrogen at 10 mL/min (linear velocity approximately 90 cm/sec). The ECD detector temperature was 310°C and the makeup gas was nitrogen flowing at 45 mL/min. All GC-ECD samples were reanalyzed on a confirmation column, 6-m  $\times$  0.53-mm ID, having a 1.5- $\mu$ m film thickness of a proprietary polymer (Rtx-TNT-2 from Restek). The GC oven was temperature-programmed as follows: 130°C for 1 min, 10°C/min ramp to 160°C, followed by a 30°C/min ramp to 270°C, which is then held for 2.33 min. The carrier gas was hydrogen at 15 mL/min (linear velocity approximately 147 cm/sec) and the

nitrogen makeup gas was flowing at 60 mL/min. Inlet and detector temperature were the same as above. Multi-analyte standards were purchased from Restek and the instrument was calibrated over five concentrations. The reporting limits for GC-ECD analysis on a soil concentration basis were 0.01 mg/kg.

#### **QA/QC**

Replicate soil samples from eight sampling areas were obtained at the same time, often by different individuals. Triplicate multi-increment samples were collected for seven areas, but only duplicates were collected for one area. These samples provide an assessment of total characterization error because they include components from sampling, sample processing, subsampling, extraction, and determination. Triplicate laboratory subsamples were analyzed from 12 different multi-increment samples to assess the error associated with sample processing, subsampling, extraction, and determination.

Six blank soil samples were processed and analyzed along with those from the range to assess the potential carryover between samples. Three matrix spiked samples were prepared and analyzed to estimate analyte recovery for a soil sample from this site.

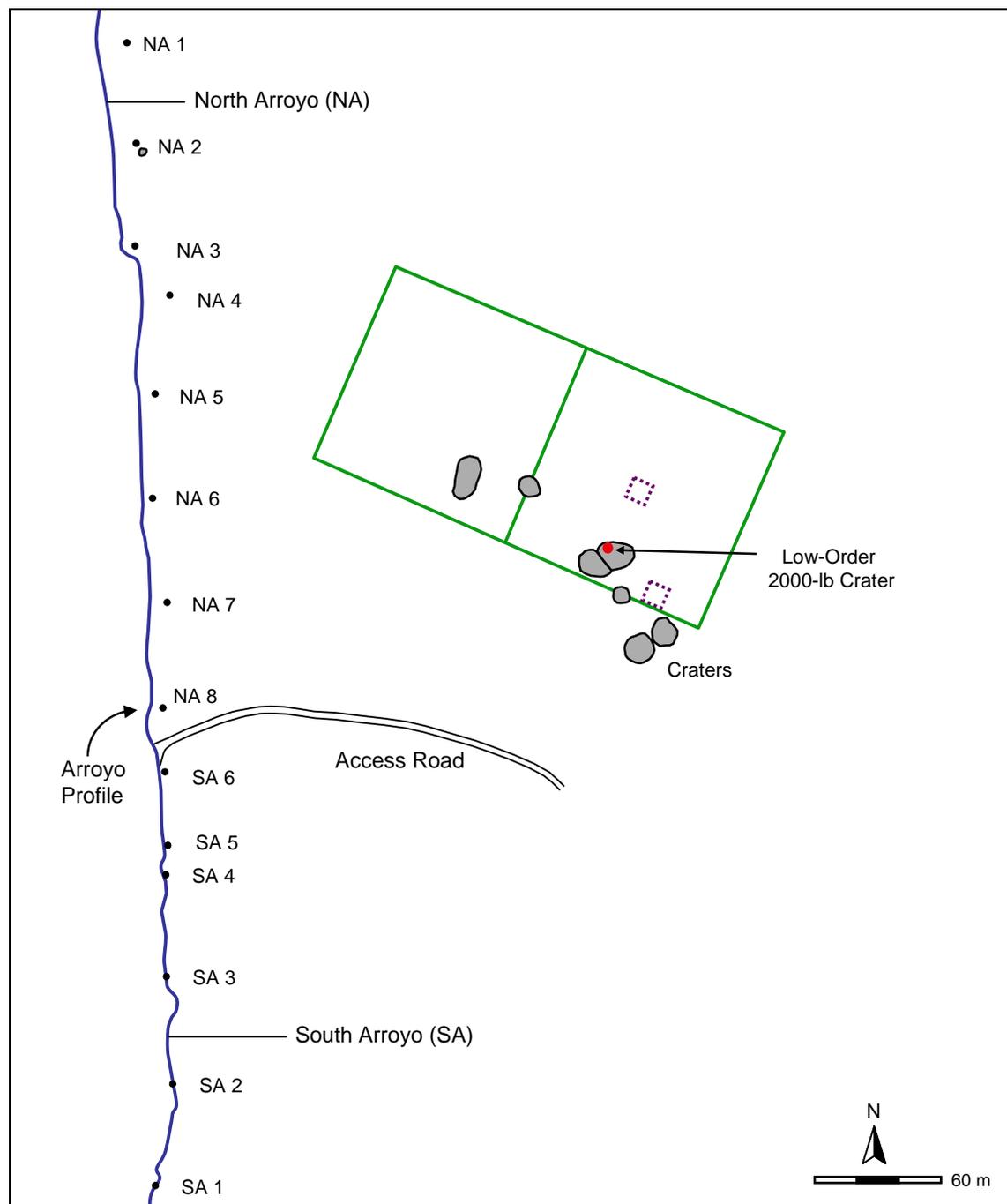
## 4 RESULTS AND DISCUSSION

### Crater from a low-order 2000-lb bomb

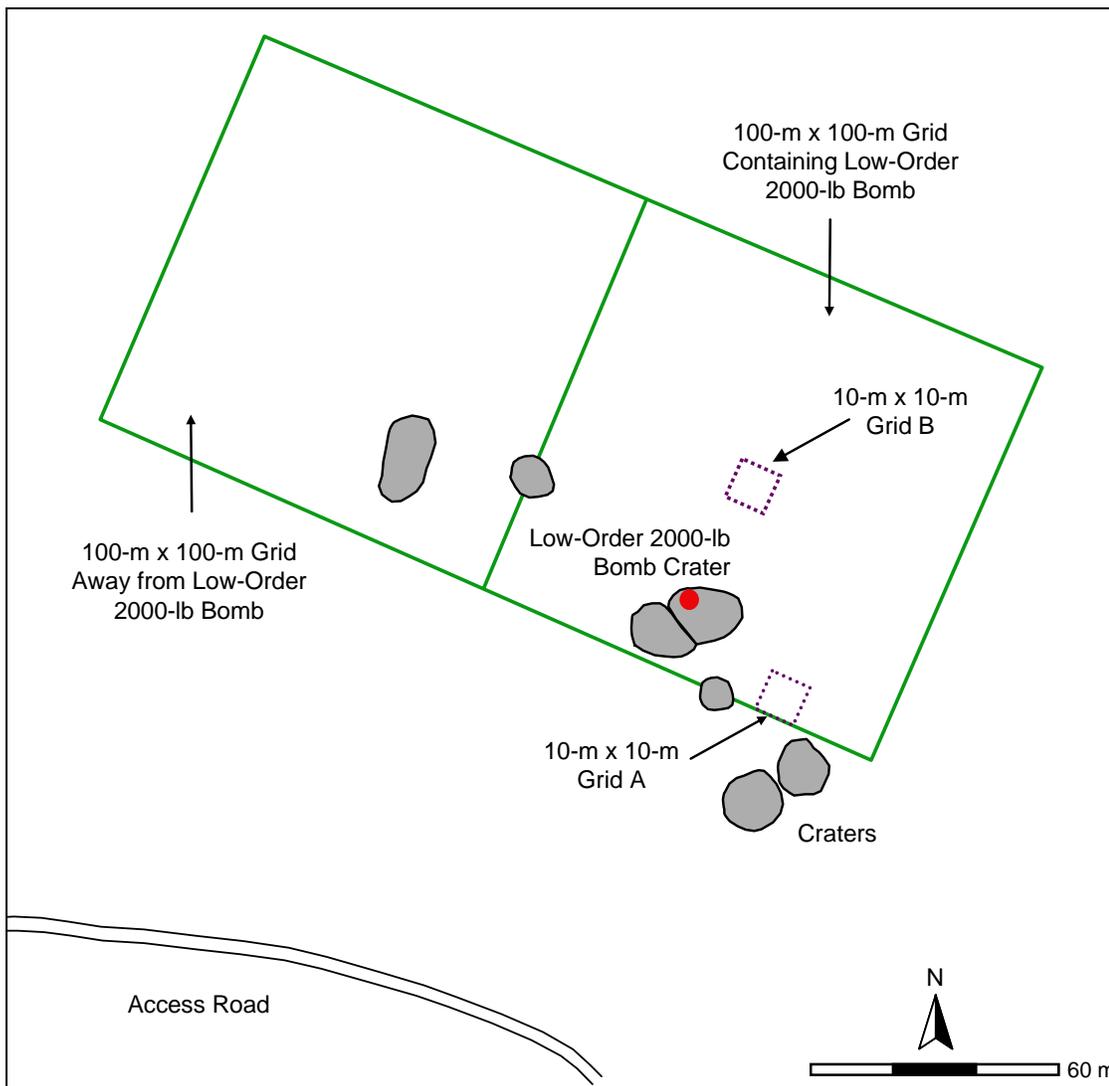
During an initial survey of the live-fire bombing range at Holloman Air Force Base (HAFB), we observed an area with a very large number of pieces of explosive lying on the surface. Further investigation of the area revealed a 2000-lb bomb with most of its casing intact near the bottom of a large crater. Our explosive ordnance disposal (EOD) team believed that the bomb originally had been a dud. Then a nearby explosion of a second bomb ruptured this bomb's side wall, resulting in a low-order detonation, and dispersing chunks of explosive in a direction uphill (southeast) from the crater (Fig. 5). Inspection of the surface southeast of this crater revealed hundreds of small pieces of explosive fill scattered over the soil surface for tens of meters in that direction. However, no pieces were observed on the surface within the crater. Subsequent analysis indicated that these chunks of explosive were tritonal (TNT and aluminum). Diagrams of the area that we investigated at HAFB are shown in Figures 6 and 7. The largest craters delineated in these diagrams were the ones we sampled; hundreds of other craters were present in the area depicted by these two figures.



**Figure 5. Sampling in front of low-order 2000-lb bomb in crater at Holloman Air Force Base.**



**Figure 6. Area sampled for energetic residues near a 2000-lb bomb crater (solid red circle) at Holloman Air Force Base. Samples were collected in the arroyo (solid black circles). Multiple sampling strategies were employed within the green- and purple-outlined grids.**



**Figure 7. Grid areas sampled at Holloman Air Force Base. Multi-increment samples were collected in the 100-m x 100-m green-outlined grids, located near to and away from a 2000-lb bomb that exploded low order and in Grids A and B. One hundred discrete samples were collected from 1-m x 1-m cells in Grid A, near the low-order crater. Twenty-five discrete samples were collected from 2-m x 2-m cells in Grid B, away from the low-order bomb. Multi-increment samples also were collected within the grey-shaded craters.**

Triplicate 50-increment surface soil samples were collected within the crater from the sidewalls and crater bottom using a systematic-random sampling design described previously (Table 1). Analysis of these three samples yielded TNT concentrations that varied from 42.8 to 89.8 mg/kg with a mean concentration of 60.0 mg/kg and a relative standard deviation (RSD) of 43.2%. Other energetic compounds detected in these crater samples included 1,3,5-trinitrobenzene (TNB), 2,4-dinitrotoluene (2,4-DNT), 3,5-dinitroaniline (DNA), 1,3-dinitrobenzene (DNB), 2-amino-4,6-dinitrotoluene (2ADNT), and 4-amino-2,6-dinitrotoluene (4-ADNT). Concentrations of these other energetic compounds were always less than 1 mg/kg in these crater samples. These compounds are either impurities in the manufacture of TNT or environmental transformation products of TNT. Even at these low concentrations, however, the RSD for these compounds, when a detectable concentration was found for all three replicates, ranged from 38 to 68%. Overall, the 50-increment samples appear to provide adequate characterization for the TNT present in the soil size fraction (<2 mm) in this crater.

#### **Discrete samples for 10-m × 10-m grid near low-order 2000-lb bomb crater**

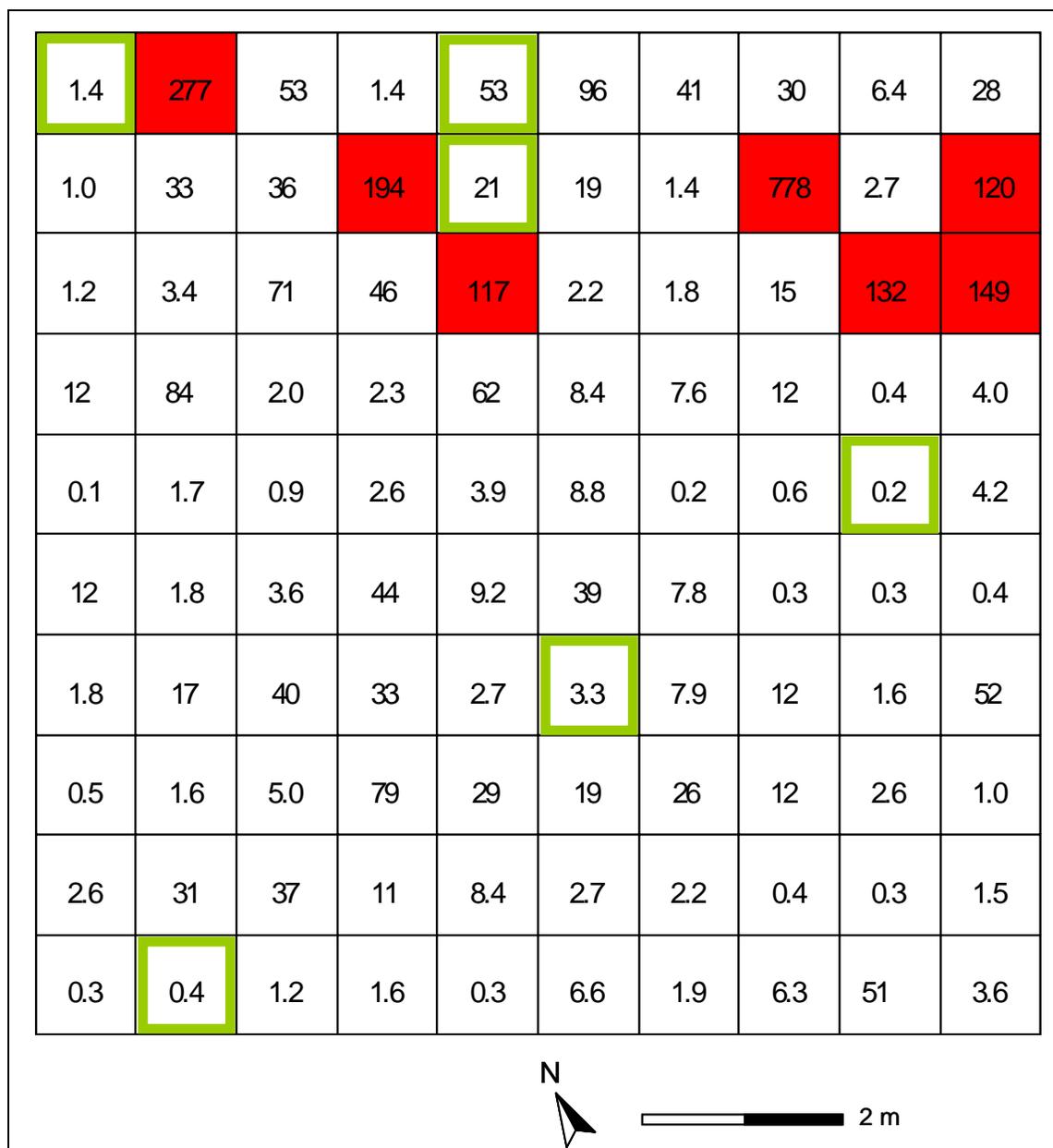
We established a 10-m × 10-m grid about 20 m uphill (southeast) from the 2000-lb low-order bomb crater in the direction of the residue fallout (Fig. 7, Grid A). This grid was subdivided into 100 1-m × 1-m cells, and a discrete soil sample was collected from an area near the center of each cell.

The same suite of energetic compounds detected in the crater was also detected in these samples, and likewise, TNT was present at the highest concentration, varying from a minimum of 0.12 mg/kg to a maximum of 778 mg/kg (Fig. 8) with a mean value of 31.8 mg/kg and an RSD of 274% (Table 2). Clearly these data are not normally distributed and thus, while we can compute a mean, a standard deviation, and a relative standard deviation, these statistics are not valid descriptors of this data. Seventy-five of the 100 TNT values are below the mean, thus the distribution is skewed right. Estimating the mean for this 10-m × 10-m grid from a single discrete sample would be extremely unreliable and would underestimate the mean about 75 percent of the time.

The maximum-to-minimum ratio for TNT concentrations for this set of 100 discrete samples is about 6480. The maximum-to-minimum TNT ratio from an identical study conducted at the Cold Lake Air Weapons Range (CLAWR) was 932 (Ampleman et al. 2004). The lower ratio obtained at CLAWR is probably due to the practice of tilling the soil to reduce vegetation in an area prone to forest fires.

<b>Table 1. Concentrations of energetic compounds in multi-increment soil samples from a crater containing a low-order 2000-lb bomb.</b>									
<b>Sample</b>	<b>Number of increments</b>	<b>Concentration (mg/kg)</b>							
		<b>TNT</b>	<b>TNB</b>	<b>2,4-DNT</b>	<b>2-ADNT</b>	<b>4-ADNT</b>	<b>DNB</b>	<b>DNA</b>	<b>RDX</b>
<b>Low-order crater</b>									
Replicate 1	50	89.8	0.95	0.35	0.29	0.29	0.03	0.05	<d
Replicate 2	50	42.8	0.28	0.08	0.15	0.15	<d	<d	<d
Replicate 3	50	47.4	0.83	0.18	0.16	0.17	<d	0.04	<d
Mean		60.0	0.69	0.20	0.20	0.20	0.01*	0.03*	<d
Std dev		25.9	0.36	0.14	0.077	0.076			
% RSD		43.2	52	68	39	38			

\* When one or two replicate values were <d, a value equal to half the detection limit of 0.01 mg/kg was used to compute the mean.



**Figure 8. TNT concentrations (mg/kg) in 100 discrete samples collected within 10-m x 10-m Grid A (Fig. 7) at Holloman Air Force Base near a low-order 2000-lb bomb. Red cells have concentrations greater than 100 mg/kg. Green-outlined cells were sampled with nine increments.**

**Table 2. Concentrations of energetic compounds in 100 discrete samples collected in 1-m x 1-m cells within 10-m x 10-m Grid A near low-order 2000-lb bomb crater.**

Parameter	Concentration (mg/kg)						
	TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA
Maximum	778	0.22	0.43	3.49	2.78	<d	0.23
Minimum	0.12	<d	<d	0.02	0.03	<d	0.01
Mean <sup>a</sup>	31.8 <sup>b</sup>	0.04	0.08	0.62	0.59		0.03
Std dev	87.0	0.04	0.07	0.46	0.38		0.04
Median	6.36	0.03	0.06	0.52	0.53		0.02
RSD (%)	274	104	81.5	73.6	64.4		116
n (values >0.01)	100	67	93	100	100		57
Values <mean	75	66	62	64	61		60
Value >100	7						

<sup>a</sup> Mean values for sets of data containing <d values were obtained using one-half the detection limit for these values.

<sup>b</sup> Because the distribution of TNT values is non-Gaussian, the mean is not a valid estimate of central tendency.

The maximum-to-minimum ratio for TNT concentrations for this set of 100 discrete samples is about 6480. The maximum-to-minimum TNT ratio from an identical study conducted at the Cold Lake Air Weapons Range (CLAWR) was 932 (Ampleman et al. 2004). The lower ratio obtained at CLAWR probably is due to the practice of tilling the soil to reduce vegetation in an area prone to forest fires.

We also collected and weighed the chunks of tritonal in cells 1 through 42 (Fig. 4) and the mass was greater than 459 g. For several chunks of tritonal, the masses were greater than the 150-g upper limit on our scale, so this total mass should be considered a minimum. The mean concentration measured for the soil samples from cells 1 to 42 was 29 mg/kg. If we estimate the mass of TNT in the soil size fraction (<2 mm) in the surface 2.5 cm of soil (bulk density = 1.7 g/cm<sup>3</sup>) in these 42 cells, the mass is 123 g. Thus we estimate that about 80% of the mass of TNT in these 42 cells is still present as chunks of tritonal, and about 20% is present in the soil-size material. The ratio of the mass of energetic residues present as chunks relative to that present in the less-than-2-mm-size fraction at this site is similar to what was measured at 29 Palms, California, in an area where pieces of H6 from a bomb detonation were present on the surface (Hewitt et al. 2005). Both of these training facilities are located in arid regions.

#### **Variability of discrete samples within the 1-m × 1-m cells**

Within the 10-m × 10-m grid described above, a set of nine equally spaced (systematic) replicate discrete soil samples was also collected within six 1-m × 1-m cells (cells 1, 20, 41, 42, 57, and 85). Cells 41, 42, and 57 were selected because they had many pieces of tritonal on the surface and cells 1, 20, and 85 were selected because they did not. Even so, the highest concentration for any discrete sample was found in cell 85 (Fig. 9). Therefore, the presence of visible pieces of energetic residues on the surface doesn't necessarily correlate to the highest soil concentrations, although the trend is clearly evident. The mean TNT concentrations for these groups of nine discrete samples ranged from 1.04 to 52.4 mg/kg (Table 3). The maximum-to-minimum ratios for individual discrete samples within these six cells ranged from 17.7 to 2871. These ratios are somewhat higher than the ratio of 9.3 for discrete samples collected from within a similar-sized area at CLAWR (Ampleman et al. 2004). Thus, even within an area as small as 1 m × 1 m, a single discrete sample does not provide a reliable estimate of the mean concentration. The maximum-to-minimum ratio for the 100 discrete samples from the entire 10-m × 10-m grid was 5190. The short-range variability within 1-m × 1-m cells was generally one to two orders of magnitude less than within the 10-m × 10-m grid, except for cell 85, where it was similar in magnitude. One reason for the lower variability within most of the 1-m × 1-m

areas is due to the lower number of observations in the 1-m × 1-m areas relative to that for the 10-m × 10-m areas (9 vs. 100).

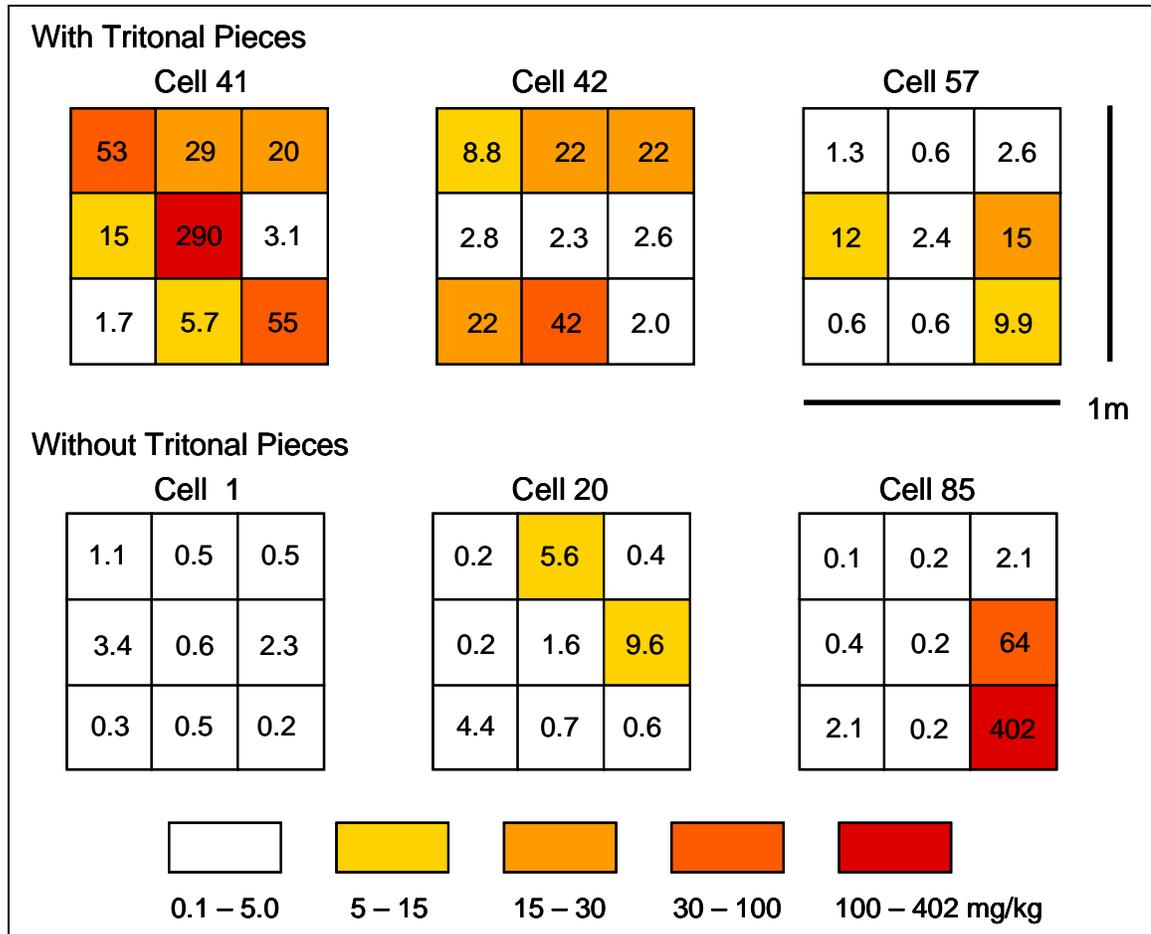


Figure 9. TNT concentrations (mg/kg) in nine discrete samples collected at six 1-m x 1-m cells within 10-m x 10-m Grid A (Fig. 7) near a low-order 2000-lb bomb. Upper three cells were collected where tritonal pieces were visible at the surface; lower three cells had no visible tritonal.

**Table 3. Concentrations of energetic compounds in replicate samples from individual cells within 10-m x 10-m Grid A near low-order bomb crater.**

Cell #	Replicate	Concentration (mg/kg)								
		TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX	NG
1	1	1.13	0.02	0.05	0.37	0.50	<d	0.04	<d	<d
	2	3.37	0.03	0.05	0.47	0.68	<d	0.04	<d	<d
	3	0.27	<d	0.35	0.18	0.28	<d	<d	<d	<d
	4	0.46	<d	0.05	0.30	0.34	<d	0.03	<d	<d
	5	0.62	<d	0.06	0.48	0.70	<d	0.04	<d	<d
	6	0.54	0.02	0.08	0.35	0.50	<d	0.05	<d	<d
	7	0.46	<d	0.16	0.52	0.76	<d	0.06	<d	<d
	8	2.28	<d	0.09	0.31	0.46	<d	<d	<d	<d
	9	0.19	<d	0.07	0.39	0.46	<d	0.05	<d	<d
	Maximum	3.37	0.03	0.35	0.52	0.76	<d	0.06	<d	<d
	Minimum	0.19	0.02	0.05	0.18	0.28	<d	<d	<d	<d
Mean*	1.04	0.02	0.11	0.37	0.52	<d	0.04	<d	<d	
20	1	0.23	<d	<d	0.13	0.21	<d	<d	<d	<d
	2	0.21	<d	<d	0.05	0.14	<d	<d	<d	<d
	3	4.42	<d	0.10	0.45	0.67	<d	<d	<d	<d
	4	5.61	<d	0.19	0.63	0.95	<d	<d	<d	<d
	5	1.57	<d	0.05	0.28	0.36	<d	<d	<d	<d
	6	0.70	<d	<d	0.03	0.07	<d	<d	<d	<d
	7	0.36	<d	<d	0.04	0.21	<d	<d	<d	<d
	8	9.61	<d	0.05	0.15	0.29	<d	<d	<d	<d
	9	0.55	<d	<d	0.02	0.05	<d	<d	<d	<d
	Maximum	9.61	<d	0.19	0.63	0.95	<d	<d	<d	<d
	Minimum	0.21	<d	<d	0.02	0.05	<d	<d	<d	<d
Mean	2.59	<d	0.10	0.20	0.33	<d	<d	<d	<d	

\* Mean values for sets of data containing <d values were obtained using one-half the detection limit for these values.

Table 3 (cont'd).										
Cell #	Replicate	Concentration (mg/kg)								
		TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX	NG
41	1	52.9	0.06	0.16	0.66	0.59	<d	0.03	<d	<d
	2	14.8	0.09	0.09	1.06	0.91	<d	0.05	<d	<d
	3	1.75	0.03	0.11	0.66	0.61	<d	0.05	<d	<d
	4	28.7	0.08	0.10	1.34	1.16	<d	0.07	<d	<d
	5	290	1.16	0.31	1.09	0.91	0.019	<d	<d	<d
	6	5.67	0.04	0.05	0.67	0.62	<d	0.04	<d	<d
	7	20.4	0.14	0.15	0.97	0.89	<d	0.05	<d	<d
	8	3.12	0.04	0.05	0.61	0.52	<d	0.03	<d	<d
	9	54.6	0.07	0.09	1.00	0.84	<d	0.05	<d	<d
	Maximum	290	1.16	0.31	1.34	1.16	0.02	0.07	<d	<d
	Minimum	1.75	0.03	0.05	0.61	0.52	<d	<d	<d	<d
Mean	52.4	0.19	0.12	0.90	0.78	0.02	0.05	<d	<d	
42	1	8.75	0.04	0.04	0.60	0.62	<d	0.06	<d	<d
	2	2.78	0.05	0.06	0.73	0.65	<d	0.04	<d	<d
	3	22.4	0.07	0.09	1.12	1.02	<d	0.06	<d	<d
	4	21.8	0.05	0.05	0.71	0.60	<d	0.06	<d	<d
	5	2.26	0.06	0.20	0.20	0.17	<d	<d	<d	<d
	6	42.2	0.11	0.23	0.99	0.90	0.02	<d	<d	0.05
	7	21.5	0.09	0.12	0.89	0.79	<d	0.07	<d	<d
	8	2.62	0.04	0.07	0.70	0.58	<d	0.03	<d	<d
	9	1.99	0.02	0.07	0.82	0.69	<d	0.02	<d	<d
	Maximum	42.2	0.11	0.23	1.12	1.02	0.02	0.07	<d	0.05
	Minimum	1.99	0.02	0.04	0.20	0.17	<d	<d	<d	<d
Mean	14.0	0.06	0.10	0.75	0.67	0.02	0.05	<d	0.05	

**Table 3 (cont'd). Concentrations of energetic compounds in replicate samples from individual cells within 10-m x 10-m Grid A near low-order bomb crater.**

Cell #	Replicate	Concentration (mg/kg)								
		TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX	NG
57	1	1.28	<d	0.08	0.53	0.56	<d	<d	<d	<d
	2	12.1	<d	0.04	0.69	0.81	<d	<d	<d	<d
	3	0.62	<d	0.06	0.12	0.11	<d	<d	<d	<d
	4	0.56	<d	0.04	0.22	0.19	<d	<d	<d	<d
	5	2.43	0.05	0.14	0.36	0.36	<d	<d	<d	<d
	6	0.64	0.02	<d	0.25	0.36	<d	<d	<d	3.99
	7	2.63	<d	0.03	0.17	0.15	<d	<d	<d	<d
	8	15.5	0.02	0.13	0.69	0.63	<d	<d	<d	0.05
	9	9.90	0.05	0.05	0.49	0.40	<d	0.04	<d	<d
	Maximum	15.5	0.05	0.14	0.69	0.81	<d	0.04	<d	3.99
	Minimum	0.56	0.02	0.03	0.12	0.11	<d	<d	<d	<d
	Mean	5.07	0.04	0.07	0.39	0.40	<d	0.04	<d	2.02
85	1	0.14	<d	<d	0.06	0.07	<d	<d	<d	<d
	2	0.37	<d	<d	0.22	0.24	<d	<d	<d	<d
	3	2.12	<d	0.04	0.65	0.56	<d	0.02	<d	<d
	4	0.20	<d	<d	0.22	0.20	<d	<d	<d	<d
	5	0.23	<d	<d	0.18	0.16	<d	<d	<d	<d
	6	0.16	<d	<d	0.25	0.25	<d	<d	<d	<d
	7	2.11	<d	0.03	0.52	0.52	<d	<d	<d	<d
	8	63.7	0.04	0.07	0.91	1.03	<d	<d	<d	<d
	9	402	0.37	0.28	1.58	1.63	<d	0.14	<d	0.08
	Maximum	402	0.37	0.28	1.58	1.63	<d	0.14	<d	0.08
	Minimum	0.14	0.04	0.03	0.06	0.07	<d	<d	<d	<d
	Mean	52.4	0.21	0.11	0.51	0.52	<d	0.08	<d	0.08

**Table 4. Concentrations of energetic compounds in multi-increment soil samples for a 10-m x 10-m area (Grid A) impacted by low-order 2000-lb bomb.**

Sample	Number of increments	Concentration (mg/kg)							
		TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX
<b>10-m x 10-m grid</b>									
Replicate 1	33	13.5	0.06	0.09	0.55	0.54	<d	0.04	<d
Replicate 2	33	12.5	0.05	0.08	0.62	0.59	<d	0.04	<d
Replicate 3	34	17.2	0.05	0.08	0.64	0.61	<d	0.04	<d
Mean		14.4	0.05	0.08	0.61	0.58		0.04	
Std dev		2.45	0.005	0.002	0.047	0.035		0.001	
% RSD		17.0	11	2.6	7.7	6.1		2.8	
<b>10-m x 10-m grid</b>	100	21.2	0.05	0.09	0.63	0.66	<d	0.04	<d

### **Multi-increment samples for 10-m × 10-m Grid A near low-order 2000-lb bomb crater**

Four multi-increment samples were also collected within the 10-m × 10-m grid using a systematic-random design (Table 4, Fig. 3). One sample was built from 100 increments, one increment from the back left corner of each cell. The other three multi-increment samples were built from 33 (or 34) increments and were built using the systematic-random sampling design by collecting an increment in every third cell from the back right corner of every third cell.

The mean value for TNT from the three 33- (or 34-) increment replicate samples of this 10-m × 10-m grid was 14.4 mg/kg with an RSD of 17%. The mean values for the other detectable energetic compounds were all less than 1 mg/kg with RSDs of 11% or less. Thus the 33-increment systematic-random sampling strategy employed provided much more reproducible results for this 10-m × 10-m area than the discrete samples that ranged from 0.12 to 778 mg/kg. This improvement in reproducibility using multi-increment samples is consistent with results reported by Ampleman et al. (2004) for a bombing range impact area at CLAWR. The mean TNT concentration for the 100-increment sample from this same area was 21.2 mg/kg, which was about 50% more than the mean of the 33- (34-) increment samples. The concentrations for the other energetic compounds in the 100-increment samples, however, were nearly identical to those for the mean of the 33-increment samples. The higher TNT concentration in the 100-increment sample was probably due to the inclusion of a small piece of tritonal in this sample.

### **Profile samples within 10-m × 10-m Grid A**

Sets of depth profile samples were also collected within the 10-m × 10-m grid, in the same six 1-m × 1-m cells where multiple discrete samples were collected. Samples were collected up to 40 cm deep. Concentration versus depth profiles from several cells are presented in Figure 10. In most cases, the TNT concentrations decline rapidly below the 5- to 7-cm depth, with concentrations less than 0.5 mg/kg at greater depth (Table 5). For the profile in cell #1, however, samples from the 16- to 19-cm- and 19- to 23-cm-depth intervals had TNT concentrations of 2.27 and 3.34 mg/kg, respectively. It is likely that these concentrations represent small pieces of energetic residue that had been buried by the large number of individual detonations that have occurred in this area over time, rather than leaching from the surface due to the arid nature of this site.

In general, though, it does not appear that TNT is leaching downward to any extent into the soil profile, even in this area where large numbers of chunks of tritonal are present on the surface.

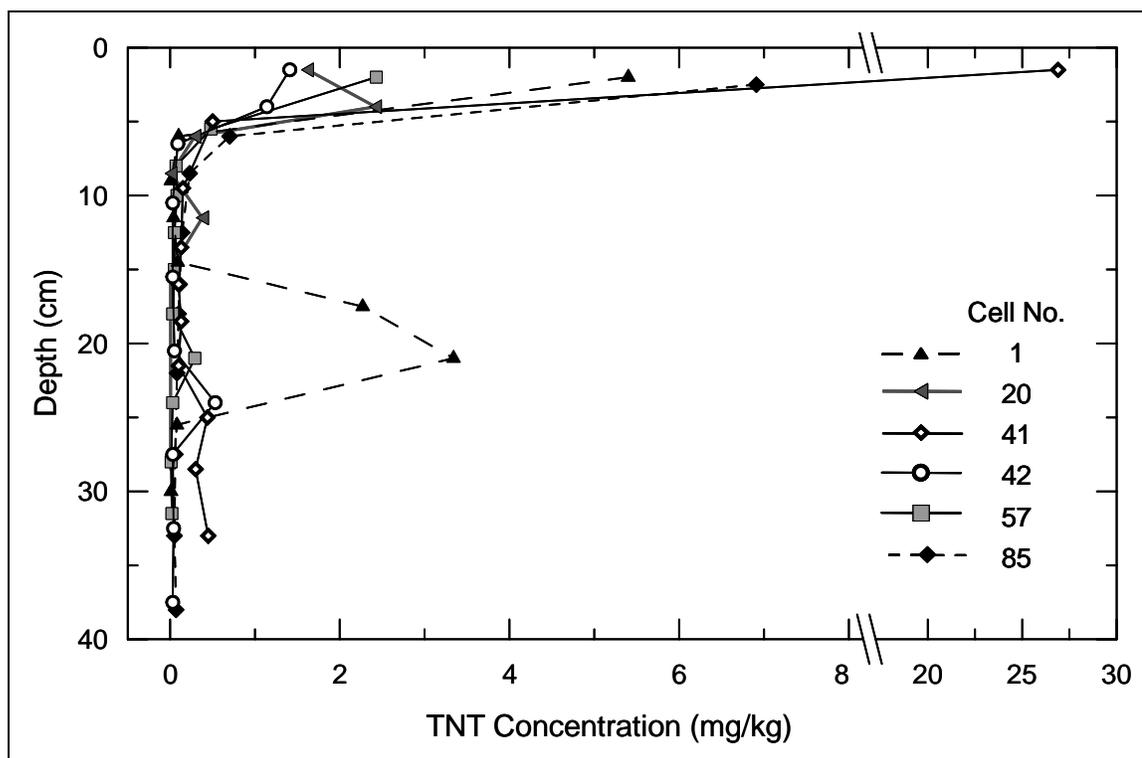


Figure 10. Depth profile of TNT concentrations (mg/kg) in selected cells within 10-m  $\times$  10-m Grid A near a low-order 2000-lb bomb.

#### 10-m $\times$ 10-m Grid B not heavily impacted by low-order 2000-lb bomb crater

A second 10-m  $\times$  10-m grid that also was located about 20 m from the low-order bomb crater also was sampled (Fig. 7, Grid B). This grid, however, was located north-northeast of the crater containing the low-order 2000-lb bomb and inspection of the surface indicated that only a few small chunks of tritonal were present compared with many hundred in the grid located southeast of the crater.

This grid was divided into 25 2-m  $\times$  2-m cells and a discrete sample was collected from each. TNT was also the energetic compound present at the highest concentration here, but the concentrations ranged from only 0.01 to 0.70 mg/kg (Table 6). The distribution of TNT concentrations was non-Gaussian, as seen by an RSD of 135% and a median value that was only about half of the mean.







**Table 6. Results for 25 discrete samples collected in 2-m × 2-m cells within 10-m × 10-m Grid B about 20 m north–northeast from low-order 2000-lb bomb crater.**

Cell #	Concentration mg/kg							
	TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX
1	0.05	0.01	<d	0.01	0.01	<d	<d	<d
2	0.20	0.02	<d	0.03	0.02	<d	<d	<d
3	0.01	<d	<d	<d	<d	<d	<d	<d
4	0.06	<d	<d	0.01	<d	<d	<d	<d
5	0.05	<d	<d	0.01	0.01	<d	<d	<d
6	0.05	<d	<d	0.01	0.01	<d	<d	<d
7	0.06	0.01	<d	0.03	0.02	<d	<d	<d
8	0.06	<d	<d	0.02	0.01	<d	<d	<d
9	0.13	<d	<d	0.02	0.02	<d	<d	<d
10	0.04	<d	<d	0.01	0.01	<d	<d	0.01
11	0.09	0.01	<d	0.02	0.02	<d	<d	<d
12	0.09	<d	0.01	0.02	0.02	<d	<d	<d
13	0.06	0.01	<d	0.02	0.02	<d	<d	<d
14	0.05	<d	<d	0.01	0.01	<d	<d	<d
15	0.48	0.02	<d	0.04	0.04	<d	<d	<d
16	0.09	<d	<d	0.03	0.03	<d	<d	<d
17	0.04	0.01	<d	0.01	0.01	<d	<d	<d
18	0.07	0.01	0.01	0.04	0.04	<d	<d	<d
19	0.05	<d	0.01	0.02	0.01	<d	<d	<d
20	0.05	<d	<d	<d	0.01	<d	<d	<d

**Table 6 (cont'd). Results for 25 discrete samples collected in 2-m x 2-m cells within 10-m x 10-m Grid B about 20 m north-northeast from low-order 2000-lb bomb crater.**

Cell #	Concentration mg/kg							
	TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX
21	0.06	<d	<d	0.03	0.01	<d	<d	<d
22	0.03	<d	<d	0.01	0.01	<d	<d	<d
23	0.70	0.03	0.01	0.13	0.24	<d	<d	<d
24	0.15	0.01	<d	0.10	0.06	<d	<d	<d
25	0.09	0.01	<d	0.06	0.02	<d	<d	<d
Maximum	0.70	0.03	0.01	0.13	0.24	<d	<d	0.01
Minimum	0.01	<d	<d	<d	<d	<d	<d	<d
Mean*	0.11			0.03	0.03			
Std dev	0.15			0.03	0.05			
% RSD	135			100	170			
Median	0.06							

\* Mean values for sets of data containing values <d were obtained by replacement with a value of half the detection limit.

**Table 7. Concentrations of energetic compounds in multi-increment samples from 10-m × 10-m Grid B about 20 m north–northeast from low-order bomb crater.**

Sample	Number of increments	Concentration mg/kg							
		TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX
<b>10-m × 10-m grid</b>									
Replicate 1	30	0.15	0.01	<d	0.03	0.04	<d	<d	<d
Replicate 2	30	0.54	0.01	<d	0.02	0.02	<d	<d	<d
Replicate 3	30	2.02	<d	<d	0.02	0.04	<d	<d	<d
Mean*		0.90	0.01		0.02	0.03			
Std dev		0.99			0.01	0.01			
% RSD		109			25	33			

\* Mean values for sets of data containing values <d were obtained by replacement with a value of half the detection limit.

Three replicate 30-increment samples were also collected from this grid (Table 7). The TNT estimates for these samples ranged from 0.15 to 2.02 mg/kg with an RSD of 109%. All three values exceeded the mean of the 25 discrete samples; the reason for this is uncertain, but probably due to presence of random nuggets that were not captured with the 25 discrete samples. When concentrations are low, the presence of a few random nuggets can have a much greater influence than when concentrations are more elevated. More mass and/or more increments would be required to provide a more reliable estimate of the mean concentration in this area.

### **100-m × 100-m grids**

Three replicate 100-increment samples were collected from two 100-m × 100-m grids (Fig. 7). The first grid encompassed both the low-order 2000-lb crater and the two 10-m × 10-m grids discussed above. TNT was the energetic compound detected at the highest concentration with values ranging from 2.60 to 12.5 mg/kg (Table 8). The mean TNT concentration was 5.94 mg/kg with an RSD of 95%. The mean concentrations of the other energetic compounds associated with TNT were all either 0.10 mg/kg or less. In this grid, RDX was detectable, but the mean concentration was only 0.10 mg/kg. The presence of RDX may be due to the practice of blowing-in-place dud bombs using C4 when EOD personnel conduct their semi-annual range maintenance.

Three replicate 100-increment samples were also collected from the second 100-m × 100-m grid located west-northwest of the first 100-m × 100-m grid. No low-order debris was observed in this area, and it was located in the opposite direction from the low-order detonation observed above. The TNT concentrations for these samples ranged from 0.08 to 0.58 mg/kg with a mean value of 0.28 mg/kg and an RSD of 93% (Table 8). The mean concentrations for the other energetic compounds were less than 0.06 mg/kg.

The maximum-to-minimum ratios for TNT concentration estimates from the 100-increment samples from these two 100-m × 100-m grids are 4.8 and 7.25, respectively, for the grids with and without low-order debris. While these ratios are larger than we might like, we must keep in mind that they are for only three replicates and yet they are many times smaller than found for discrete samples, even within an area as small as 1 m × 1 m. Thus, concentration estimates based on multi-increment samples provide a higher level of confidence in the estimate of the mean than can be achieved with mean estimates based on one or several discrete samples.

Table 8. Concentrations of energetic compounds in 100-increment soil samples from two 100-m x 100-m grids.

Sample	Number of increments	Concentration (mg/kg)								
		TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX	HMX
<b>100-m x 100-m grid encompassing the low-order 2000-lb bomb</b>										
Replicate 1	100	12.5	0.03	<d	0.11	0.11	<d	<d	0.25	<d
Replicate 2	100	2.60	0.03	0.08	0.12	0.09	<d	0.03	0.04	<d
Replicate 3	100	2.76	0.01	<d	0.12	0.11	<d	<d	0.01	<d
Mean*		5.94	0.02	0.03	0.12	0.10	<d	0.01	0.10	<d
Std dev		5.65	0.01		0.01	0.01			0.13	
% RSD		95.1	62		4.5	11			135	
<b>100-m x 100-m grid with no visible low-order debris</b>										
Replicate 1	100	0.58	0.08	<d	<d	<d	<d	<d	<d	<d
Replicate 2	100	0.19	0.03	<d	0.06	<d	<d	<d	<d	<d
Replicate 3	100	0.08	0.02	0.01	0.11	0.03	<d	0.01	0.02	0.01
Mean		0.28	0.04	<d	0.06	0.01	<d	<d	0.01	<d
Std dev		0.26	0.03							
% RSD		93	71							

\* Mean values for sets of data containing values <d were obtained by replacement with a value of half the detection limit.

**Table 9. Concentrations of energetic compounds in multi-increment soil samples from other craters.**

Sample	Number of increments	Soil concentration (mg/kg)								
		TNT	1,3,5-TNB	2,4-DNT	2-ADNT	4-ADNT	1,3-DNB	3,5-DNA	RDX	NG
Old 500-lb bomb crater <sup>a</sup>	30	0.14	0.01	0.01	0.03	0.04	<d	<d	<d	<d
Old 2000-lb bomb crater 1 <sup>b</sup>	56	3.55	0.17	0.02	0.14	0.15	<d	0.03	<d	<d
Old 2000-lb bomb crater 2	26	0.09	0.03	<d	0.19	0.02	<d	0.01	<d	<d
Old 2000-lb bomb crater 3	45	0.05	<d	<d	<d	<d	<d	<d	<d	0.01
Old 2000-lb bomb crater 4	31	0.04	<d	<d	<d	<d	<d	<d	<d	<d
Old 2000-lb bomb crater 5	45	<d	<d	0.02	<d	<d	<d	<d	<d	0.18

<sup>a</sup> Mean of lab triplicates  
<sup>b</sup> Mean of field duplicates

### **Other crater samples**

Multi-increment surface soil samples were collected from six other craters within the live-fire bombing area at Holloman AFB (Table 9). The concentrations of individual energetic compounds for these samples were less than 0.2 mg/kg in all but one case; one of the 2000-lb bomb craters had a TNT concentration of 3.55 mg/kg. Thus it appears that these craters were formed by bombs that probably detonated high-order and deposited only microgram-to-milligram quantities of energetic residues (Hewitt et al. 2003, M.R. Walsh et al. 2005).

### **Arroyo downslope of the live-fire bombing range**

An arroyo is located downslope of the live-fire bombing range at Holloman AFB (Fig. 6). If energetic residue in either particulate form or dissolved in precipitation is running off the range, the runoff would be captured within the channel of this arroyo. A set of 14 multi-increment sediment samples was collected beginning beyond the north edge of the live-fire range and continuing south along the channel for about 550 m. Each sample was built from 30 increments taken from the surface 0–2.5 cm. A set of profile samples was also collected from the lowest-lying location within the arroyo.

Analytical results for these samples are presented in Table 10. The concentration of energetic compounds in the surface sediment samples from the arroyo was always less than 0.2 mg/kg, except for one sample collected 210 m north of the access road, where the TNT concentration was 2.28 mg/kg. This high concentration area appears to be isolated either from up- or downstream samples and may be due to a small piece of tritonal from a low-order detonation landing within or near the arroyo.

A set of depth profile samples up to 35 cm deep was also collected within the arroyo, 15 m north of the access road (Table 10). Energetic compounds within these samples were generally below the detection limit of 0.01 mg/kg. In three samples, TNT in one, 2,4-DNT in another, and 4-ADNT in the third, the concentration was detected at 0.01 mg/kg. In the third sample, 2-ADNT was also detected at 0.02 mg/kg.

It does not appear that TNT or any other energetic compound is running off the range to an off-site location, or is penetrating downward within the channel of the arroyo.





### Demolition range

Two sets of multi-increment samples were collected within a 20-m radius of a demolition area where C4 was used to ensure that practice bombs contained no residual explosive prior to removal of metal scrap from the range. The first set of samples was collected on 5 May 2005 during our visit to the site; the second set of multi-increment samples was collected by EOD personnel on 11 May 2005 after a demolition event in which C4 was used to blow holes in several types of practice bombs containing no high explosive.

Results for triplicate 30-increment samples before and after the demolition event are presented in Table 11. In all cases, the major energetic compounds present are RDX and HMX. RDX concentrations ranged from 2.04 to 27.8 mg/kg prior to the demolition event and from 4.07 to 12.5 mg/kg afterward. Similarly, HMX concentrations ranged from 0.59 to 3.98 mg/kg prior to demolition and from 1.02 to 2.43 mg/kg after demolition. TNT concentrations for five of these six samples ranged from <0.01 to 0.15 mg/kg, but one sample had a TNT concentration of 2.05 mg/kg.

The reproducibility of RDX and HMX concentrations for five of these six 30-increment samples was quite good, ranging from 2.04 to 12.5 mg/kg and 0.59 to 2.44 mg/kg, respectively. The concentrations in the sixth sample are about two times higher than in any other sample and this is likely due to the incorporation of a small piece of C4 within an increment used to build that sample. Overall though, the ability to provide reproducible estimates of the mean concentration for this area is quite good.

The source of the RDX and HMX at this site is certainly the C4 used as the demolition charge. C4 is composed of 91% military-grade RDX, of which HMX is present as impurity in excess of 10%. The fact that the RDX and HMX concentrations did not appear to increase after the most recent demolition event indicates that the major residues are not deposited every time an event occurs, but rather when an individual detonation does not proceed properly. The reason for the presence of TNT in one sample, however, is unclear. TNT is not a component of C4 and should not be present in these practice bombs. It is possible it could have been tracked into this area from EOD personnel who had been conducting clearance activities within the live-fire bombing area where pieces of tritonal were widely dispersed. The demolition area is the only area at Holloman AFB where we detected significant residues of RDX and HMX.

Table 11. Concentrations of energetic compounds in multi-increment samples from a demolition range.

Sample	Number of increments	Concentration mg/kg									
		RDX	HMX	TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	NG
<b>Demolition range before mid-May 2005 blow-in-place</b>											
Replicate 1	30	2.04	0.59	0.02	<d	0.04	0.02	0.04	<d	<d	<d
Replicate 2	30	27.8	3.98	0.15	<d	0.06	0.05	0.05	<d	<d	<d
Replicate 3	30	4.39	0.96	<d	<d	0.02	0.02	0.02	<d	<d	0.70
	Mean	11.4	1.8	0.06*		0.04	0.03	0.04			0.24
	Std dev	14.2	1.9			0.02	0.02	0.02			
	% RSD	125	101			50	67	50			
<b>Demolition range after mid-May 2005 blow-in-place</b>											
Replicate 1	30	12.0	2.16	0.05	0.02	<d	<d	<d	<d	0.04	<d
Replicate 2	30	12.5	2.43	2.05	0.02	<d	<d	<d	<d	0.01	<d
Replicate 3	30	4.07	1.02	0.12	<d	<d	<d	<d	<d	0.06	<d
	Mean	9.50	1.87	0.74	0.01	<d	<d	<d	<d	0.04	<d
	Std dev	4.71	0.75	1.14						0.02	
	% RSD	50	40	153						61	

\* Mean values for datasets containing values <d were obtained by replacement with a value of half the detection limit.

### **Estimation of sample processing and determination error for these samples**

Triplicate subsamples from 12 soil samples from Holloman AFB were used to assess the contribution of a combination of laboratory processing, subsampling, and determination to the total characterization error (Table 12). The mean concentration of TNT in the sample from the low-order crater was 89.8 mg/kg with an RSD of 0.67%. Clearly, for this high-concentration sample, the contribution from the laboratory error (including subsampling) was insignificant compared with total error estimate of RSD = 43.2% (Table 1). Thus, the major portion of the total error was sampling error, even when sampling was conducted using multi-increment samples. This was also the case for the other energetic compounds in these subsamples, even though the concentrations of these analytes were at least two orders of magnitude less than TNT.

Triplicate laboratory subsamples from the only sample collected in a second crater with much lower TNT concentrations are also presented in Table 12. The mean TNT concentration for these subsamples was 0.14 mg/kg with an RSD of 25%. Even though the absolute standard deviation is much lower for this set of three laboratory subsamples compared with those from a sample collected from the crater with the low-order-bomb, the much lower mean concentration inflates the RSD estimate. This is typically found as concentrations decline toward detection limits because in this concentration range the absolute standard deviation often becomes constant. Also, when the contaminant is present as discrete particles, the number of these particles becomes quite small and difficult to reproduce as you approach the detection limit. Overall, however, the error due to sample processing, subsampling, and analysis in this study is minor compared to sampling error as shown for the other eleven sets of lab replicates in Table 12.



<b>Table 12 (cont'd). Results for energetic compounds in replicate laboratory subsamples.</b>										
<b>Sample</b>	<b>Concentration (mg/kg)</b>									
	<b>TNT</b>	<b>TNB</b>	<b>2,4-DNT</b>	<b>2-ADNT</b>	<b>4-ADNT</b>	<b>DNB</b>	<b>DNA</b>	<b>RDX</b>	<b>NG</b>	<b>HMX</b>
<b>South Arroyo #4</b>										
Replicate 1	<d	<d	0.01	0.01	<d	<d	<d	<d	<d	<d
Replicate 2	<d	<d	<d	0.01	<d	<d	<d	<d	<d	<d
Replicate 3	<d	<d	<d	0.01	<d	<d	<d	<d	<d	<d
Mean				0.01						
Std dev				0.001						
% RSD				13						
<b>North Arroyo #5</b>										
Replicate 1	0.07	<d	<d	<d	0.01	0.01	<d	<d	<d	<d
Replicate 2	0.05	<d	<d	<d	<d	<d	<d	<d	<d	<d
Replicate 3	0.03	<d	<d	<d	<d	<d	<d	<d	<d	<d
Mean	0.05									
Std dev	0.02									
% RSD	38									
<b>10-m x 10-m grid near 2000-lb bomb</b>										
Replicate 1	17.2	0.05	0.08	0.63	0.61	<d	0.04	<d	<d	<d
Replicate 2	17.1	0.05	0.08	0.64	0.61	<d	0.04	<d	<d	<d
Replicate 3	17.3	0.05	0.08	0.64	0.60	<d	0.04	<d	<d	<d
Mean	17.2	0.05	0.08	0.64	0.61		0.04			
Std dev	0.09	0.00	0.00	0.01	0.01		0.00			
% RSD	0.5	0.00	1.4	0.8	0.8		3.1			

Table 12 (cont'd).										
Sample	Concentration (mg/kg)									
	TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX	NG	HMX
<b>10-m x 10-m grid away from 2000-lb bomb</b>										
Replicate 1	0.50	0.01	<d	0.02	0.02	<d	<d	<d	<d	<d
Replicate 2	0.54	0.01	<d	0.02	0.02	<d	<d	<d	<d	<d
Replicate 3	0.58	0.01	<d	0.01	0.02	<d	<d	<d	<d	<d
Mean	0.54	0.01		0.02	0.02					
Std dev	0.04	0.001		0.004	0.002					
% RSD	6.7	14		19	8.9					
<b>100-m x 100-m grid near 2000-lb bomb</b>										
Replicate 1	2.76	0.02	<d	0.12	0.11	<d	<d	<d	<d	<d
Replicate 2	2.74	<d	<d	0.12	0.11	<d	<d	<d	<d	<d
Replicate 3	2.78	<d	<d	0.11	0.10	<d	<d	0.02	<d	<d
Mean	2.76			0.12	0.11					
Std dev	0.02			0.01	0.01					
% RSD	0.7			5.5	4.6					
<b>100-m x 100-m grid away from 2000-lb bomb</b>										
Replicate 1	<d	0.02	0.01	0.12	0.04	<d	0.01	0.02	0.09	0.01
Replicate 2	<d	0.02	0.01	0.10	0.03	<d	0.01	0.02	0.01	0.01
Replicate 3	<d	0.02	0.00	0.09	0.03	<d	0.01	0.02	0.01	0.01
Mean		0.02	0.01	0.11	0.03		0.01	0.02	0.04	0.01
Std dev		0.00	0.00	0.02	0.01		0.001	0.004	0.05	0.001
% RSD		14	14	16	18		11	20	126	11

<b>Table 12 (cont'd). Results for energetic compounds in replicate laboratory subsamples.</b>										
<b>Sample</b>	<b>Concentration (mg/kg)</b>									
	<b>TNT</b>	<b>TNB</b>	<b>2,4-DNT</b>	<b>2-ADNT</b>	<b>4-ADNT</b>	<b>DNB</b>	<b>DNA</b>	<b>RDX</b>	<b>NG</b>	<b>HMX</b>
<b>Demolition area #1</b>										
Replicate 1	<d	<d	0.01	0.01	0.02	<d	<d	4.80	0.60	1.12
Replicate 2	<d	<d	0.02	0.01	0.02	<d	<d	4.48	0.39	0.95
Replicate 3	<d	<d	0.03	0.02	0.02	<d	<d	3.88	1.12	0.81
Mean			0.02	0.01	0.02			4.39	0.70	0.96
Std dev			0.011	0.001	0.002			0.47	0.37	0.16
% RSD			55	8.2	7.8			11	53	17
<b>Demolition area #2</b>										
Replicate 1	0.06	0.02	<d	<d	<d	<d	0.06	12.70	<d	2.3
Replicate 2	0.05	<d	<d	<d	<d	<d	0.03	10.98	<d	1.93
Replicate 3	0.04	0.02	<d	<d	<d	<d	0.04	12.24	<d	2.24
Mean	0.05	0.02					0.04	11.97		2.16
Std dev	0.01	0.01					0.02	0.89		0.20
% RSD	12	61.3					37	7.4		9.2
<b>Demolition area #3</b>										
Replicate 1	2.02	0.02	<d	<d	<d	<d	0.03	13.32	<d	2.32
Replicate 2	2.10	<d	<d	<d	<d	<d	<d	15.42	<d	2.56
Replicate 3	2.04	0.02	<d	<d	<d	<d	<d	8.66	<d	2.42
Mean	2.05	0.02						12.47		2.43
Std dev	0.04	0.01						3.46		0.12
% RSD	2.0	57.7						28		5.0

Table 12 (cont'd).										
Sample	Concentration (mg/kg)									
	TNT	TNB	2,4-DNT	2-ADNT	4-ADNT	DNB	DNA	RDX	NG	HMX
<b>Demolition area #4</b>										
Replicate 1	0.13	<d	<d	<d	<d	<d	<d	4.42	<d	1.10
Replicate 2	0.12	<d	<d	<d	<d	<d	0.04	4.04	<d	1.01
Replicate 3	0.12	<d	<d	<d	<d	<d	0.14	3.76	<d	0.93
Mean	0.12						0.06	4.07		1.02
Std dev	0.01						0.07	0.33		0.08
% RSD	5.7						116	8.1		8.3
<b>Summary Statistics</b>										
<i>n</i>	9*	6	5	8	7	0	5	5	2	5
Mean % RSD	10.2	25.8	15.2	18.5	10.5		34.0	14.7	89.6	10.1
Minimum % RSD	0.52	0.00	1.40	0.40	0.40		2.44	7.44	53.2	4.95
Maximum % RSD	38.3	61.3	54.5	84.9	33.2		116	27.8	126	16.6
Mean concentration	12.5	0.02	0.09	0.15	0.16		0.04	6.58	0.37	1.31
* One additional sample analyzed by GC-ECD had a mean TNT concentration of 0.08 mg/kg, with an RSD of 17%.										

## 5 CONCLUSIONS

Results from this study indicate that discrete surface soil samples collected from a live-fire bombing range are not normally distributed. The variability among discrete samples is very large even for areas as small as  $1\text{ m} \times 1\text{ m}$ . This is consistent with results from a variety of other types of military firing range impact areas (Ampleman et al. 2003; Jenkins et al. 1999, 2001, 2004a, b; Hewitt et al. 2005; M.E. Walsh et al. 2004, 2005). Using a sampling strategy based on a few discrete samples or a multi-increment sample using only a few increments would provide estimates of the mean concentration with a very large uncertainty. Much more reliable estimates of the mean concentrations were achieved using a stratified systematic random sampling design with collection of multi-increment samples with at least 30 increments.

TNT was the energetic compound detected at the highest concentration in surface and shallow subsurface samples from the live-fire range. RDX was generally below analytical detection limits in these samples, although it was occasionally detected at trace levels, perhaps due to the use of C4 demolition explosive to destroy duds during semi-annual range maintenance activities. No evidence of off-site migration of residues was found in either depth profile samples or samples collected along an arroyo that drains the entire live-fire range.

RDX and HMX were the two energetic compounds detected at the highest concentration at a small demolition range used to ensure that practice bombs do not contain energetic compounds prior to removal for metal recycling. C4 demolition explosive is the most likely source of these residues.

Sample processing and subsampling protocols employed in this work were adequate to maintain the laboratory contribution to total characterization uncertainty at acceptable levels. These protocols used a 2-mm sieve to remove oversized material, machine grinding for material that passed the 2-mm sieve, a subsampling procedure that combined 30-increments from the ground soil, and a subsample mass of 10 grams.

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# REPORT DOCUMENTATION PAGE

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<b>1. REPORT DATE (DD-MM-YY)</b> February 2006		<b>2. REPORT TYPE</b> Technical Report		<b>3. DATES COVERED (From - To)</b>	
<b>4. TITLE AND SUBTITLE</b>  Sampling Studies at an Air Force Live-Fire Bombing Range Impact Area				<b>5a. CONTRACT NUMBER</b>	
				<b>5b. GRANT NUMBER</b>	
				<b>5c. PROGRAM ELEMENT NUMBER</b>	
<b>6. AUTHOR(S)</b>  Thomas F. Jenkins, Alan D. Hewitt, Charles A. Ramsey, Kevin L. Bjella, Susan R. Bigl, and Dennis J. Lambert				<b>5d. PROJECT NUMBER</b>	
				<b>5e. TASK NUMBER</b>	
				<b>5f. WORK UNIT NUMBER</b>	
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b>  U.S. Army Engineer Research and Development Center Cold Regions Research and Engineering Laboratory 72 Lyme Road Hanover, New Hampshire 03755				<b>8. PERFORMING ORGANIZATION REPORT</b>  ERDC/CRREL TR-06-2	
<b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b>  U.S. Army Corps of Engineers Washington, DC 20314-1000				<b>10. SPONSOR / MONITOR'S ACRONYM(S)</b>	
				<b>11. SPONSOR / MONITOR'S REPORT NUMBER(S)</b>	
<b>12. DISTRIBUTION / AVAILABILITY STATEMENT</b>  Approved for public release; distribution is unlimited.  Available from NTIS, Springfield, Virginia 22161.					
<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b> <p>Field sampling experiments were conducted at an Air Force live-fire bombing range. The main objective was to assess the effectiveness of using a systematic-random, multi-increment sampling strategy for the collection of representative surface soil samples in areas where bombing practice is conducted with bombs containing high explosives. Replicate surface soil samples were collected within several craters and in different sized grids (1 m × 1 m, 10 m × 10 m, and 100 m × 100 m). One area sampled had been impacted by a low-order 2000-lb bomb detonation and several hundred small chunks of tritonal were present on the surface. Another area sampled had many fewer recognizable chunks of tritonal on the surface. An arroyo, located downslope of the heaviest impacted area of this live-fire range, where runoff from the area would be captured, was also sampled at several locations. TNT was the major energetic compound present within the live-fire bombing area. Short-range heterogeneity in TNT concentrations was very large and the ability to estimate mean concentration using discrete samples, even for an area as small as 1 m<sup>2</sup>, was poor. Much more reproducible estimates of mean concentrations for areas as large as 100 m × 100 m were achieved using multi-increment samples collected with a stratified systematic-random sampling design compared with that achieved using discrete samples. Results from soil profile samples and samples from the arroyo draining this area indicate that the energetic compounds present at the bombing range are not migrating from the site.</p> <p>Another area sampled was a small demolition range where C4 explosive is used to ensure that practice bombs contain no residual explosive prior to removing scrap metal from the range. RDX and HMX were the energetic compounds detected at the highest concentration in surface soil at the demolition range. These compounds originated from the demolition explosive.</p>					
<b>15. SUBJECT TERMS</b>					
Bombing ranges		Energetic compounds		Impact areas	
C4		Explosives		Live-fire	
Demolition ranges		HMX		Multi-increment sampling	
				RDX	
				TNT	
				Training ranges	
<b>16. SECURITY CLASSIFICATION OF:</b>				<b>17. LIMITATION OF OF ABSTRACT</b>	<b>18. NUMBER OF PAGES</b>
<b>a. REPORT</b>	<b>b. ABSTRACT</b>	<b>c. THIS PAGE</b>			
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