ERDC/CRREL TR-09-6

Cold Regions Research
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US Army Corps
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Environmental Security Technology Certification Program – Project ER-0628

Validation of Sampling Protocol and the Promulgation of Method Modifications for the Characterization of Energetic Residues on Military Testing and Training Ranges

Alan D. Hewitt, Dr. Thomas F. Jenkins, Marianne E. Walsh,
Susan R. Bigl, and Dr. Sylvie Brochu

June 2009

Cover photo: Field sampling team with soil samples collected at a demolition range at Fort Richardson, Alaska.

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Final report

Prepared for Environmental Security Technology Certification Program
Arlington VA 22203

Under ER-0628

Abstract: The Military maintains sustainability and environmental stewardship responsibilities for training ranges. One of the environmental challenges is to monitor the surface loading of energetic residues. Method 8330B, which was developed to assist in this task, recommends the use of a *MULTI INCREMENT*®[1] sampling strategy and total sample processing prior to subsampling and analysis. This demonstration/validation project evaluated scientifically defensible sampling and sample-processing protocols used for the characterization of energetic residues on military training ranges. This project also specifically assessed the scientific uncertainty involved with the now-common practice of splitting a sample in the field and subsampling it prior to processing the entire laboratory sample. To address the cost aspect, this study then evaluated the cost benefits of coupling *MULTI INCREMENT* sampling methodology with recently developed sample handling and processing protocols designed to address the unique attributes of energetic residues. Consequently, the authors recommend that *MULTI INCREMENT* sampling and the U.S. Environmental Protection Agency (USEPA) Method 8330B be used for future characterization activities on military training ranges.

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[1] *MULTI INCREMENT* is a registered trademark of EnviroStat, Inc. of Fort Collins, CO, for a comprehensive sampling methodology. More information is available at **www.envirostat.org**

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Preface

This report was prepared by the U.S. Army Engineer Research and Development Center (ERDC), Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire, in partnership with the Defence Research and Development Canada-Valcartier (DRDC-Val), Quebec, Canada. Funding for this work was provided under project ER-0628 by the Environmental Security Technology Certification Program (ESTCP), with Dr. Jeffrey Marqusee, Director, and Dr. Andrea Leeson, Cleanup Program Manager.

The principal investigator was Alan D. Hewitt, Biogeochemical Science Branch (BSB), CRREL. Co-principal investigators were Dr. Thomas F. Jenkins and Marianne Walsh, CRREL, and Dr. Sylvie Brochu, DRDC-Val.

The authors gratefully acknowledge the assistance and technical reviews provided by Dr. Clarence Grant, ERDC-CRREL, and Mr. Charles Ramsey, Envirostat, Inc.

The report was prepared under the general supervision of Dr. Terrence Sobecki, Chief, Biogeochemical Sciences Branch. At the time of publication, Dr. Lance Hansen was Deputy Director, and Dr. Robert E. Davis was Director, CRREL.

The Commander of ERDC was COL Gary E. Johnston; the Director was Dr. James R. Houston.

Executive Summary

The objectives of this demonstration/validation project were to promote scientifically defensible sampling and sample-processing protocols for the characterization of energetic residues on military training ranges. As a consequence of this effort, *MULTI INCREMENT*®[2] sampling and the U.S. Environmental Protection Agency (USEPA) Method 8330B should be used for future characterization activities on military training ranges.

The present protocols used for investigations addressing the presence or mass loading of energetic residues, or both, are inadequate. For example, it is common to collect a few discrete or 5- to 7-increment samples, split the samples in the field, and remove only small portions of the laboratory sample for further processing and analysis. The inadequacy of collecting only a few samples to establish the mean concentration of energetic residues was previously demonstrated by Jenkins et al. (2005a, 2006).

The study described in this report specifically assessed the uncertainty involved with the practice of splitting a sample in the field and subsampling it prior to processing the entire laboratory sample. This study also evaluated the cost benefits of coupling *MULTI INCREMENT* sampling methodology with recently developed sample handling and processing protocols designed to address the unique attributes of energetic residues.

During the first year of the project, the Office of Solid Waste (OSW) of the USEPA released Method 8330B to the general public (USEPA 2006a). This revision of Method 8330 recommends collecting *MULTI INCREMENT* samples that weigh 1 kg or more for establishing the concentration of energetic residues in areas of concern and the subsequent laboratory processing of the entire sample. Laboratory protocols include air-drying, sieving, mechanical pulverization, and collecting ≈30 increments to build 10-g subsamples for extraction and analysis. This study demonstrated that when field samples of this size are processed following the guidelines in Appendix A of Method 8330B, the 10-g subsample taken for analysis is representative of the mean concentration the entire sample. That is, the precision among subsample replicates is typically less than 10% relative

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percent difference (RSD) and, the comparison of the subsample concentration to that of the total sample resulted in a relative percent difference (RPD) of less than 10%.

By contrast, the evaluation of uncertainty associated with the practice of splitting a sample in the field after mixing in a bowl and removing only a small portion for further laboratory processing and analysis, showed median RSD values among replicates greater than 40%. In addition, there were large biases and occurrences of false positives and false negatives when comparing values obtained for sample splits and subsamples to that of total field sample or total laboratory sample, respectively. As a consequence, the values reported for these split samples only represent the subsample that was analyzed. Moreover, because of these sources of indeterminate error, only assumptions (professional judgment) can then be applied to qualify the relationship between the concentrations obtained for subsamples to that of the area sampled.

Sampling strategies and design were evaluated by comparing means and 95% upper confidence limits for means generated with the computer program ProUCL (USEPA 2007). This analysis showed the futility of using 95% upper confidence level (UCL) intervals derived from small data sets (e.g., n = 5) of discrete samples. However, often a single or a small number of *MULTI INCREMENT* samples provided much more reasonable (closer to the grand mean) and reliable (less variable) means or 95% UCL intervals of the means, as compared to those based on 30 discrete samples.

A cost analysis (see Section 5) that compared 1–5 *MULTI INCREMENT* samples with 30 discrete samples indicated a potential 50–80% savings for sample handling, processing, and analysis costs (assuming the logistics and sampling activities would be comparable). This comparative study and others have demonstrated that *MULTI INCREMENT* sample collection is more cost-effective and practical for achieving a reasonable estimate of the mean than the collection of discrete samples.

Since this project was proposed and funded, several workshops and seminars have been given and over 20 presentations have been made on the benefits of *MULTI INCREMENT* sampling and Method 8330B. In addition, we have provided solicited information and guidance to Department of Defense (DoD) facilities, U.S. Army Corps of Engineer (USACE) Districts, state regulatory agencies, and two USEPA Regions that have been involved with site characterization activities on military training and testing ranges. Currently several Military Munitions Response Program (MMRP) Formerly Used Defense Sites (FUDS) have already gone through the site investigation stage using the *MULTI INCREMENT* sampling strategy and Method 8330B, and more than 200 additional sites are anticipated to follow this initiative in the next few years.

1 Introduction

1.1 Background

The Environmental Security Technology Certification Program (ESTCP) Environmental Restoration (ER) project (ER-0628)[3], recognizes that the use of different sampling and sample processing protocols impacts the data quality of samples used to estimate the mass loading of energetic residues on Department of Defense (DoD) training and testing ranges. These concerns resulted from the findings of several sampling and sample-processing studies performed during the Strategic Environmental Research and Development Program (SERDP) Environmental Restoration (ER) projects, ER-1155[4] and ER-1481[5]. Briefly stated, these programs showed that using a *MULTI INCREMENT* sampling strategy and a systematic-random sampling design routinely produces data sets with much less uncertainty. They also showed that this combination of sampling strategy and design often provides a better estimate of the mean concentrations of energetic residues within an area of concern, when compared to other commonly practiced procedures (Jenkins et al. 2005a, 2006, 2008; Hewitt et al. 2007a). In addition, whole sample processing in the laboratory by pulverization (particle size reduction) was necessary to attain the degree of homogenization necessary for the sample to be representatively subsampled (Walsh et al. 2002).

To promote a unified sampling and sample-processing protocol for the characterization of energetic residues on DoD training ranges, the ESTCP ER-0628 program revised the USEPA Method 8330 by working with the agency's Office of Solid Waste (OSW) organic methods working group. A consequence of this effort was USEPA Method 8330B[6], which became publicly available in November 2006. The demonstration phase of this program documented the attributes of Method 8330B and compared its

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[3] ER-0628: Validation of Sampling Protocol and Promulgation of Method modifications for the Characterization of Energetic residues on Military Training Ranges (also the title of this Technical Report).

[4] ER-1155: Distribution and Fate of Energetics on DoD Test and Training Ranges, Completed FY06. (Formerly known as Compliance Project 1155)

[5] ER-1481: Characterization and Fate of Gun and Rocket Propellant Residues on Testing and Training Ranges, Completed FY 08

[6] Subsequently referred to only as Method 8330B.

results to the large uncertainty associated with the collection of discrete samples, field-splitting of large samples, and subsampling of an inadequately processed field sample in the laboratory. Establishing reliable and defensible environmental data is paramount to site characterization programs that may lead to remedial activities in support of range sustainability and stewardship responsibilities.

1.2 Objectives of the demonstration

The principal objective of this demonstration was to inform potential users of the sampling and sample processing protocols developed under SERDP ER-1155 and ER-1481, and also recommended in USEPA Method 8330B. During the demonstration portion of the ESTCP ER-0628 program, two field studies and several workshops were held to facilitate transfer of this innovative technology to the DoD, regulatory organizations, commercial enterprises, and other end-users. Field demonstrations were performed at two military training facilities — Fort Richardson, Alaska and Canadian Forces Base (CFB) Petawawa, Ontario. The field studies at Fort Richardson were performed at a firing point, demolition range, and central impact range; field studies at CFB Petawawa included a firing point and hand grenade range. In total, seven workshops were held at the following locations: (1) Massachusetts Military Reservation (MMR) in Bourne, Massachusetts (twice); (2) Engineering Research and Development Center–Cold Regions Research and Engineering Laboratory (ERDC–CRREL) in Hanover, New Hampshire (twice); (3) U.S. Army Environmental Command (USAEC) in Aberdeen, Maryland; (4) USEPA laboratory at Silverdale, Washington; and (5) the annual SERDP–ESTCP meeting in Washington, D.C. In total, over 150 participants attended these workshops.

The *MULTI INCREMENT* sampling strategy and whole-sample processing protocol recommended in Appendix A of Method 8330B has several advantages. The area of concern is treated as a single decision unit (alternately termed a population, exposure unit, etc.) or, it can be divided to determine presence of gradients or boundaries. Samples are composed of increments of appropriate mass obtained at evenly spaced locations throughout the chosen decision unit; this attempts to address both distributional and compositional heterogeneity that is common in environmental contamination. The number of increments and the final mass of the *MULTI INCREMENT* sample is a function of the data needed to satisfy the project data quality objectives (DQOs). Replicate field samples must be collected to estimate the total measurement error. In contrast, variation among data obtained using discrete and other common sampling designs (e.g., the wheel or box design, see Section 3.5.6) is often much greater than with *MULTI INCREMENT* sampling, and the values are seldom normally distributed. More importantly, if the sample is split in the field or not adequately processed in the laboratory prior to subsampling, the constituents of interest will not be well represented, contributing additional uncertainty to the final value. Method 8330B recommends the entire sample be shipped to the laboratory and that it receive thorough processing prior to subsampling and analysis to minimize these two sources of error.

1.3 Regulatory drivers

The DoD and USEPA both have programs tasked with determining if military training and testing facilities present a risk to human health and the environment. The DoD has established the Military Munitions Response Program (MMRP) and Operational Range Assessment Program (ORAP, DoD Directive 4715.11[7]) requiring site investigations. The USEPA has become involved in the characterization of energetic residues on military training ranges and the potential for off-site migration through ongoing investigations of the MMR (USEPA 2000). As a consequence, presentations covering the development of Method 8330B and application of the *MULTI INCREMENT* sampling strategy for the characterization of energetic residues on military training ranges have been given at the EPA's National Association of Remedial Projects Managers (NARPM) meeting for the past couple of years.

1.4 1.4 Stakeholders/end-user issues

The initial product of this demonstration—posting of Method 8330B on the Web (USEPA 2006a)—helped address many concerns initially expressed by potential stakeholders and end-users. Once posted, we offered laboratory services to several groups to rapidly facilitate implementation of Method 8330B. This was necessary because, initially, commercial laboratories were not prepared to perform the whole sample pulverization procedure. Today, at least four commercial laboratories offer

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[7] Department of Defense Directive 4715.11: Environmental and Explosives Safety management on Operational Ranges Within the United States. Adopted 10 May 2004 and certified current as of 24 April 2007. Available at (accessed June 2009):

the full sample-preparation protocol recommended in Method 8330B. In addition, several workshops have been held to promote the attributes of *MULTI INCREMENT* sampling and to highlight the laboratory modifications required for representative subsampling and analysis. These workshops have facilitated adoption of these new protocols and the acquisition of pulverization equipment by commercial laboratories wishing to support the MMRP. We've also demonstrated the *MULTI INCREMENT* sampling strategy at three MMRP FUDS, and assisted the USACE - South Pacific Division, USEPA Region 6, the USACE Environmental and Munitions Center of Expertise (EM CX), and the states of Texas, Utah, New Mexico, and Arizona in the development of *MULTI INCREMENT* sampling strategies for several different types of ranges (see Appendix F). As a consequence of this commercial availability, several site investigations at MMRP FUDS have implemented Method 8330B.

2 Technology Description

2.1 Technology development and application

Energetic residues are deposited on DoD training ranges as particles from military explosives and also as irregular fibers or pieces from propellants and rocket fuels. These energetic residues accumulate on the surface near firing points and around targets where rounds have partially detonated (low-ordered) or ruptured. In addition, they are found where unexploded ordnance (UXOs) are found or placed and then are blown-in-place as part of a range clearance or training activity (Jenkins et al. 2001, 2004a, 2004b, 2006, 2008; Pennington et al. 2001, 2002, 2003, 2004, 2005, 2006). Figure 1 shows two examples of unconsumed energetic residues: (1) 2,4,6-trinitrotoluene (TNT) particles resulting from the blow-in-place of a 155-mm howitzer round with a block of C4 and (2) propellant fibers deposited on the snow in front of a 105-mm howitzer gun position.

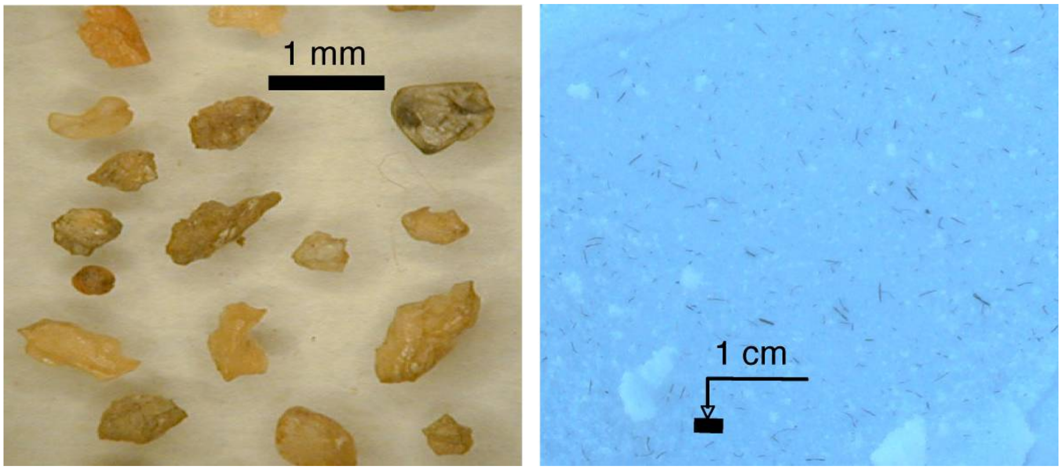

Figure 1. Examples of energetic material particles: TNT particles (<1 mm, fraction) from a blow-in-place detonation (left) and 105-mm howitzer propellant fibers on snow (right).

To representatively (and reproducibly) sample an area where particles have been dispersed, the sampling strategy must address the compositional and distributional heterogeneity of the constituents of concern. Compositional heterogeneity results from the fact that individual particles within a population often have different concentrations of target analytes. This heterogeneity is at a maximum when some of the target analytes are present as discrete particles. Error due to compositional heterogeneity is called the fundamental error and is inversely related to the sample mass. Distributional heterogeneity is due to uneven scattering of contaminant particles across the site, sometimes with a systematic component as well as a short-range random component. Error resulting from distributional heterogeneity is inversely related to the number of increments used to build the sample. This error is at a maximum when a single discrete sample is used to estimate the mean for a large decision unit (e.g., exposure unit).

To reduce the influence of these error sources when estimating the mean concentration of an analyte within a decision unit, Method 8330B recommends collecting 30 or more evenly spaced increments to build a sample with a total sample mass >1 kg (Jenkins et al. 2004a, 2004b, 2005a, 2006, 2008; Walsh, M.E. et al. 2005; Hewitt et al. 2005, 2007a). The objective of this sampling technique is to obtain a representative amount of every particle size, composition (e.g. Tritonal, Composition B, H6, etc.), and configuration (e.g., crystalline spheres or elongated fibers), and to not over-sample or miss any portion of the decision unit. The size of the decision unit is a function of the data quality objectives (DQOs) and can be as small or as large as necessary to satisfy the DQOs. The ability to meet the objective of low uncertainty depends on the type of range and training activity being performed. To estimate total uncertainty for estimating mean concentrations of energetic residues, replicate *MULTI INCREMENT* samples must be collected for each type of activity under investigation. If this step is not included in a sampling plan, the total characterization error cannot be determined.

Because most energetic residues tend to settle on surfaces, the existing vegetative cover can trap these particles. Figure 2 shows two examples of surface vegetation present at a firing point and in and around a crater where an 81-mm mortar projectile had low-ordered on an impact range. If vegetation is removed or avoided during sampling, energetic residue particles trapped within this surface cover will not be included in the sample and the result will be an underestimation of the amount of energetic residues at that location (Walsh et al. 2005, 2007). In cases where surface vegetation is dense, use of specially designed tools will aid in the collection of surface samples with minimal surface disturbance and human effort, and most importantly, will help to avoid inadequate sampling (i.e., sampling only the exposed soil surfaces) (Walsh 2004).

Figure 2. Examples of surface vegetation at a firing point (inset) and near the crater of an 81-mm mortar projectile low-order detonation on an artillery/mortar impact range.

To obtain representative subsamples from a field sample, the processing protocol must also address the compositional and distributional heterogeneity. Method 8330B recommends the entire field sample be air dried and then passed through a #10 (2-mm) sieve. The sieved fraction of the sample is then mechanically pulverized to reduce the particle size of both the matrix and constituents of concern to < 0.07 mm. This step is necessary since within the < 2 mm soil size class, energetic residue particles exist as a variety of sizes, shapes and compositions. The #10 sieve size was selected to encompass those particles that can be readily dissolved and to be consistent with the classification of soil used in risk models for human exposure (Pennington et al. 2004; Hewitt and Bigl 2005). Particle size reduction was deemed necessary because, even after air-drying and sieving, the compositional heterogeneity is too great within the <2 mm fraction to ensure that subsamples or sample splits would retain representative portions of energetic residues (Walsh et al. 2002; Hewitt et al. 2007b). This step also provides a safety feature, since energetic materials greater than 2 mm are excluded from grinding. Furthermore, use of a mortar and pestle, as previously cited in Methods 8330 and 8095, breaks up clumps of soil but does not change the size of discrete particles (Walsh et al. 2002).

To further reduce the uncertainty among subsamples, Method 8330B recommends a 10-g subsample size be obtained by combining many (≥ 30) smaller increments. To lower the detection limits and minimize the consumption of solvent, the 10-g subsample of soil should be extracted with 20 mL of acetonitrile (a 1:2 ratio), instead of the 1:5 ratio cited in the earlier Method 8330 and Method 8095 (Walsh et al. 2005). Lastly, extracting energetic residues from soils can be performed using either an ultrasonic bath or a platform shaker table (Walsh and Lambert 2006). To establish the subsampling uncertainty for estimating mean concentrations of energetic residues in a sample, replicate subsamples should be obtained from an appropriate number of samples. If this step is not included in the laboratory analysis plan, there can be no assessment of the subsampling error.

2.2 Previous testing of the technology

Our current sampling strategy, design, processing protocols, and analysis methods have evolved during studies performed at over 20 military training and testing facilities over the past decade. Results of these studies have also been instrumental in directing research focused on the development of remediation technologies and have helped guide fate and transport modeling. Our findings have been presented at numerous conferences and have been published in government reports and refereed journals (e.g., publications by Jenkins et al., Walsh et al., Hewitt et al., and Pennington et al.). We have also helped to produce guidance manuals for training range characterization, both as a CRREL Technical Report (Thiboutot et. al. 2002) and through two interagency technology Web pages. The Web link entitled “FATE: Field Analytic Technology Encyclopedia: Explosives Module,” was developed in 2003[8] and supported by both USACE and USEPA. The second Web link, a seminar entitled “Field-Based Analytical Methods for Explosive Compounds[9],” covers field-based analytical methods for explosives residues in soil and water matrices, physical and chemical properties of secondary explosives, review of accepted fixed-laboratory-based methods, sampling considerations, and emerging technologies. This site was developed in 2002, and also was supported by USACE and the USEPA Technology Innovation Office.

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[8] www.clu-in.org/char/technologies/exp.cfm

[9] http://clu-in.org/conf/tio/explosives/

2.3 Factors affecting cost and performance

Several end-users and stakeholders have expressed concerns with the increased costs associated with the collection, shipment, and processing of large samples. However, the current protocol of collecting smaller samples from which only a small portion is removed for processing produces data that are not reproducible or representative, and therefore fail to meet any desirable level of data quality (USEPA 2006b). Therefore, the decision to use the procedures in Method 8330B is based primarily on performance and data quality, and not merely on cost. Significantly improved data quality will lead to sound decisions and a reduction in the need to perform additional field sampling activities, resulting in cost saving over the life cycle of a given project.

2.4 Advantages and limitations of the technology

A guidance manual for sampling and analysis plans based on scientifically sound (defensible) studies will help expedite future range characterization programs that address the burden of energetic residues on military training ranges. Once characterization studies have been performed, the proper remediation/management programs can be initiated when needed. Rapid implementation of corrective measures will help control remediation costs, maximize the usability of military training ranges, and help prevent off-site migration of hazardous munitions constituents from source areas. If energetic residues migrate off military ranges, it is anticipated that the life cycle operational and maintenance costs of a pump and treat system would run into the millions of dollars.

This program focused on a two-dimensional area of concern, that is, the mass loading of energetic residues on the surface of military training and testing ranges. However, large amounts of energetic residues on some ranges can also be present in the subsurface. For example, because the surfaces of demolition ranges are often re-graded, residues can become buried from 1 m–2 m below the surface. Development of sampling designs focusing on practical implementation of the *MULTI INCREMENT* sampling strategy for the characterization of a three dimensional area of concern is a research topic that should receive attention in the future.

3 Demonstration Design

3.1 Performance objectives

Performance objectives targeted by the ESTCP ER-0628 program for sample collection, handling, and processing are listed in Table 1. All protocols evaluated in this demonstration (including those recommended in Method 8330B for sampling, handling, and processing) were compared to these performance objectives.

Table 1. Target performance objectives.

Performance Objective	Primary Performance Criteria	Expected Performance (Metric)	Actual Performance (%RSD; n = 3)
Quantitative	Subsampling error	Representative and precise subsamples, split samples, and reproducible field samples	< 10%
	Sample splitting error		< 10%
	Sampling error and collection bias		< 30%*

* Total measurement error.

3.2 Selected test sites

The primary criterion used to select training ranges for these field demonstrations was the known presence of energetic residue concentrations at levels readily detectable by Method 8330B. Fort Richardson, Alaska and Canadian Force Base (CFB) Petawawa, Ontario, Canada were both considered to be ideal candidates since the facilities were in the process of developing environmental impact statements (EIS) and together offered a variety of training range types.

3.3 Test site descriptions

3.3.1 Fort Richardson

Located near the city of Anchorage, Alaska, Fort Richardson has been in operation since 1940. It now occupies approximately 56,000 acres (Figure 3) bounded to the north by Knik Arm, to the west by Elmendorf Air Force Base, and to the south and east by the Municipality of Anchorage.

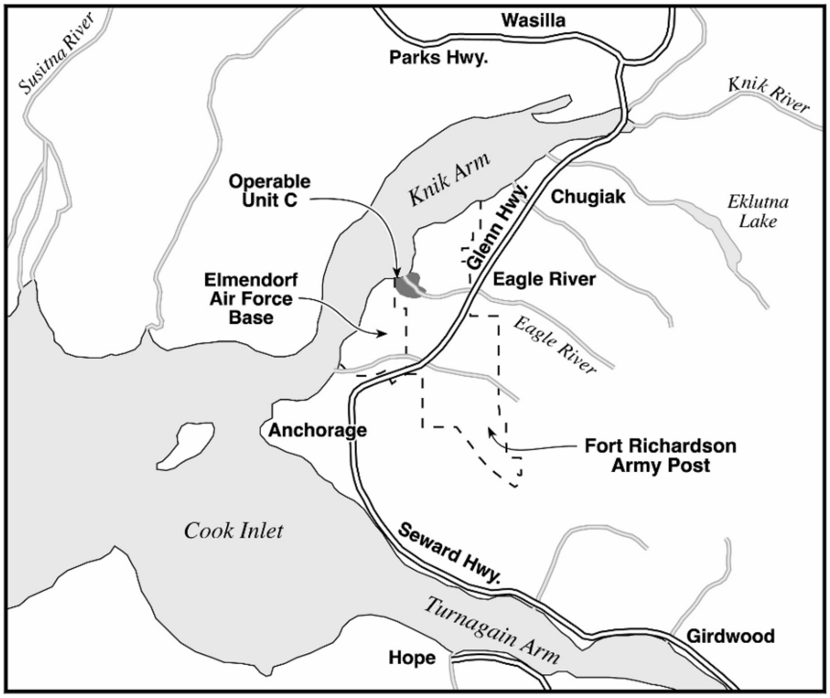

Figure 3. Fort Richardson and surrounding areas.

The current Fort Richardson mission is to support the rapid deployment of Army forces from Alaska to the Pacific Theater and worldwide. ERDC-CRREL has a long-standing relationship working in the arena of range characterization with the U.S. Army Garrison, Alaska, and the U.S. Army, Alaska. The long-running Eagle River Flats project is a study of white phosphorus contamination and remediation at a tidal flat which is utilized as a central impact range. The effort is a prime example throughout the military of the excellent cooperation attainable between the active military and federal agencies. This level of cooperation and trust has enabled ERDC-CRREL to leverage the white phosphorus work at Eagle River Flats with funding from several other projects dealing with munitions residues, thus expanding our knowledge of the impacts of training on ranges.

Fort Richardson is currently obtaining information for an EIS, a required document that informs Army decisionmakers and the public of the potential environmental effects associated with changes in military training activities. In this case, the change is a return to year-round firing of munitions within the Eagle River Flats Impact Area (Figure 3). Both the

National Environmental Policy Act (NEPA)[10] and 32 CFR § 651[11] require the Army to consider the environmental impacts of their actions and alternatives, and to solicit the views of the public before they make a final decision to proceed. Therefore, the work performed under this project naturally follows work currently underway both at Fort Richardson and at other training ranges throughout Alaska.

The advantages of conducting the study at Fort Richardson also included: (a) the historically close working relationship between Range Control and ERDC-CRREL ensured good communications and expediency of field operations; (b) familiarity with the key personnel and procedures necessary that allowed us to conduct the required sampling very quickly during May and June 2007, and (c) prior sampling at several ranges, in conjunction with other studies in August 2006, that allowed us to determine in advance that Fort Richardson sites were suitable for this sampling demonstration study.

3.3.2 CFB Petawawa

CFB Petawawa is located approximately 160 km northwest of Ottawa, in the province of Ontario, Canada (Figure 4). Military activities at Petawawa began in 1905 on a 90-km^2 area located adjacent to the Ottawa River. The base since has grown in size and importance since the mid 1990s with the closure of many Canadian Army bases. CFB Petawawa has become one of the four principal Canadian bases—along with Edmonton, Gagetown, Valcartier, and Wainwright. Although still relatively small (now 400 km^2), CFB Petawawa has been extensively used over a long period of time. As a consequence of such heavy and prolonged training, a very wet climate, and well-drained sandy soils and very shallow groundwater, munitions constituents of concern (HMX and RDX) have been recently detected in the ground water at several locations.

The same advantages listed for Fort Richardson also apply to CFB Petawawa. That is, there is a close working relationship between Range Control and DRDC Valcartier, ensuring good communications during field

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[10] 42 U.S.C. § 4321 et seq., signed into law Jan. 1, 1970. Available at (accessed June 2009): **http://www.epa.gov/oecaerth/basics/nepa.html**

[11] 32 CFR § 651: Environmental Analysis of Army Actions (also covered by AR 200-2). Available at (accessed June 2009): **http://law.justia.com/us/cfr/title32/32cfr651_main_02.html**

operations. In addition, several ranges at CFB Petawawa have already been sampled in conjunction with studies initiated in 2005, providing the information to establish their suitability. The DRDC-Val was tasked by the Canadian Army's (CA) Director – Land Environment (DLE) to perform characterization studies of the main training ranges in Canada, to better understand the nature and extent of munitions constituents.

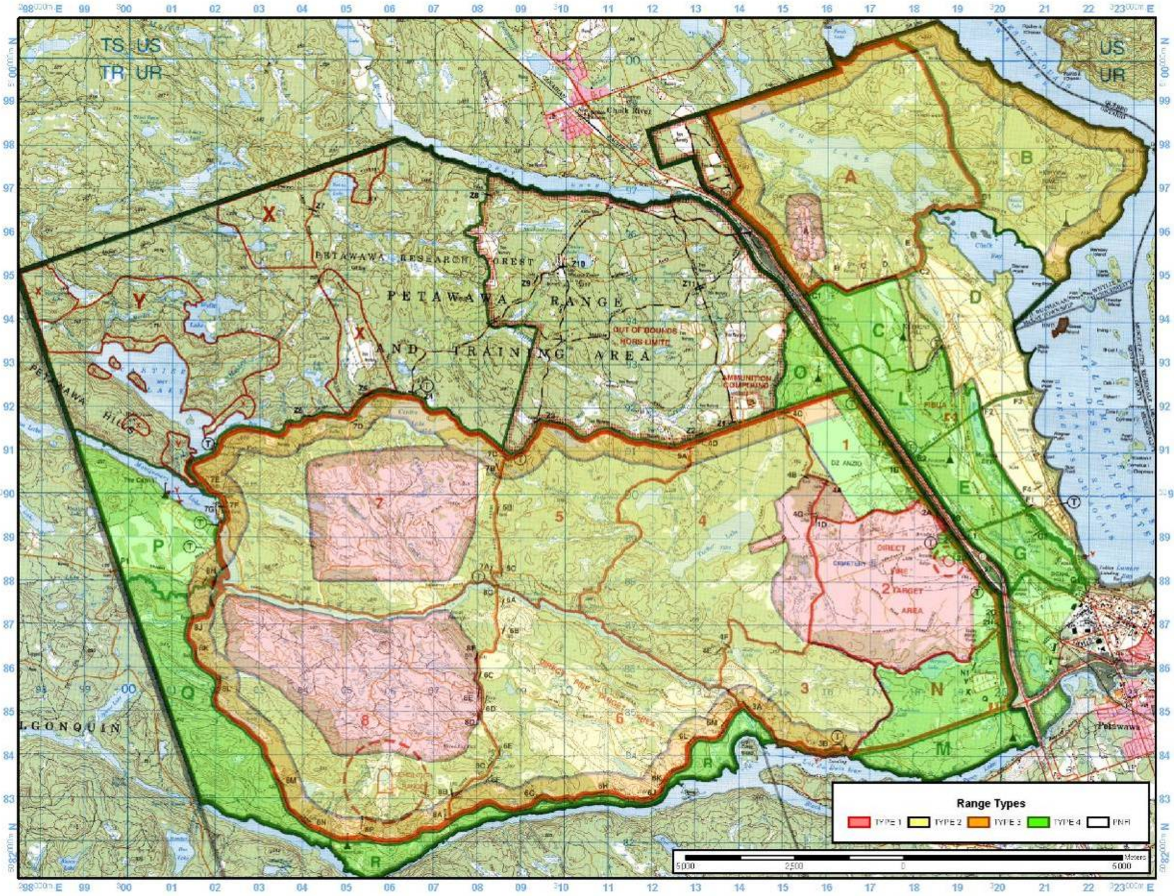

Figure 4. Layout of training ranges at Canadian Forces Base (CFB) Petawawa.

3.4 Pre-demonstration testing and analysis

Pre-demonstration sampling at Fort Richardson performed in August 2006 at small arms ranges, artillery and mortar firing areas, the central impact range, and a demolition range indicated that all sites were good candidates for this sampling demonstration study. The final ranges selected for the demonstration were Firing Point (FP) Fox, Demolition Range #3, and a location within the central impact range where chunks of energetic residues were found near a low-order 120-mm round. The 2006 sampling activities established that surface soil at FP Fox had NG concentrations between 1 and 30 mg/kg (Walsh et al. 2007). At the demolition range, concentrations of HMX, RDX, TNT, 2,4-DNT were between 1 and 200 mg/kg, and at the low-order location within the central impact range, concentrations of HMX, RDX, and TNT varied from below detection to greater than 2,000 mg/kg.[12]

Pre-demonstration sampling at CFB Petawawa was conducted in October of 2005 and 2006. Small arms ranges, mounted artillery firing areas, small caliber guns pads, mortar firing areas, a hand grenade range, an anti-armor firing point, areas of interest in the central impact range, a demolition range, and several groundwater monitoring wells were sampled. For this demonstration, the ranges selected for sampling were FP Juliet Tower and a hand grenade range. At FP Juliet Tower, 2,4-DNT and NG had been determined to be present between 0.5 and 2 mg/kg; at the hand grenade range, concentrations of RDX and TNT ranged from below detection to 5 mg/kg (Pennington et al. 2006, Chapter 3).

It should be recognized that concentrations on these active ranges at any given time depend on usage and practices. For example, a single low-order detonation or the burning of unused propellant bags or rings could heavily influence the concentration of energetic residues.

3.5 Testing and evaluation plan

3.5.1 Demonstration set-up and start-up

Scheduling range access and making provisions for escort by military Explosives Ordinance Disposal (EOD) personnel or a contracted UXO technician were initiated several months prior to each demonstration. In addition, Health and Safety Plans (HASP) were developed (see Appendix D). These coordinating responsibilities were performed by Michael Walsh for Fort Richardson and by Dr. Sylvie Brochu for CFB Petawawa. Once range access had been scheduled, notices were sent through the ESTCP executive office, inviting members of government agencies to attend and participate.

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[12] Marianne Walsh, personal communication with author Alan Hewitt.

3.5.2 Period of operation

The field demonstrations took place at Fort Richardson on 30 May through 4 June 2007 and at CFB Petawawa on 2 and 3 October 2007.

3.5.3 Amount / treatment rate of material to be treated

N/A

3.5.4 Residuals handling

No residual material requiring disposal resulted from this demonstration. All remaining portions of soil samples are archived at ERDC-CRREL. Section 3.5.7 describes sample handling protocols.

3.5.5 Operating parameters for the technology

N/A

3.5.6 Experimental design

Table 2 lists the number of samples collected at the five sampling areas. Five box and wheel samples were collected within each sampling area. For the box sampling design, five increments were combined to form a sample. The increments were collected at the center and at 5-m distances from the center, moving in the four cardinal directions (Figure 5a). In the wheel sampling design, seven increments were combined to form a sample, collected at the center and at six equally spaced locations on the perimeter of a circle with a 0.6-m radius (Figure 5b). There were 10 *MULTI INCREMENT* samples collected, each composed of 100 increments collected at evenly spaced intervals within the decision unit (DU) (Figure 6). This number of increments was chosen for ease of implementation and to ensure that more than 1 kg of sample would be obtained. Ten *MULTI INCREMENT* samples were collected to allow for an analysis of the distribution of concentrations.

Table 2. Samples collected.

Sampling Area	Discrete	Box Design	Wheel Design	*MULTI INCREMENT*
Firing Point Fox	100	5	5	15
Demolition Range	100	5	5	10
Impact Range LO#3	200	5	5	10

Sampling Area	Discrete	Box Design	Wheel Design	*MULTI INCREMENT*
Hand Grenade Range	100	5	5	10
Firing Point Juliet Tower	100	5	5	12

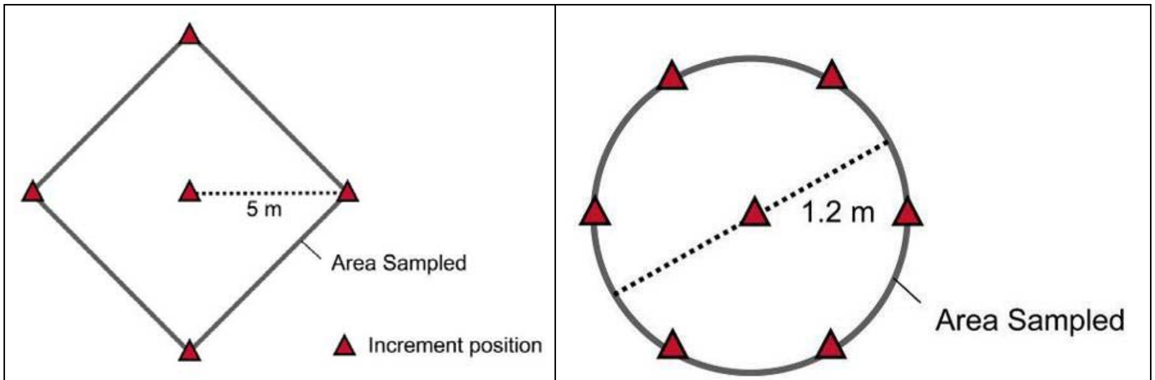

Figure 5a. Five-increment 50-m^2 box.

Seven-increment 1.2-m diameter wheel.

Figure 5. The two conventional sampling designs used.

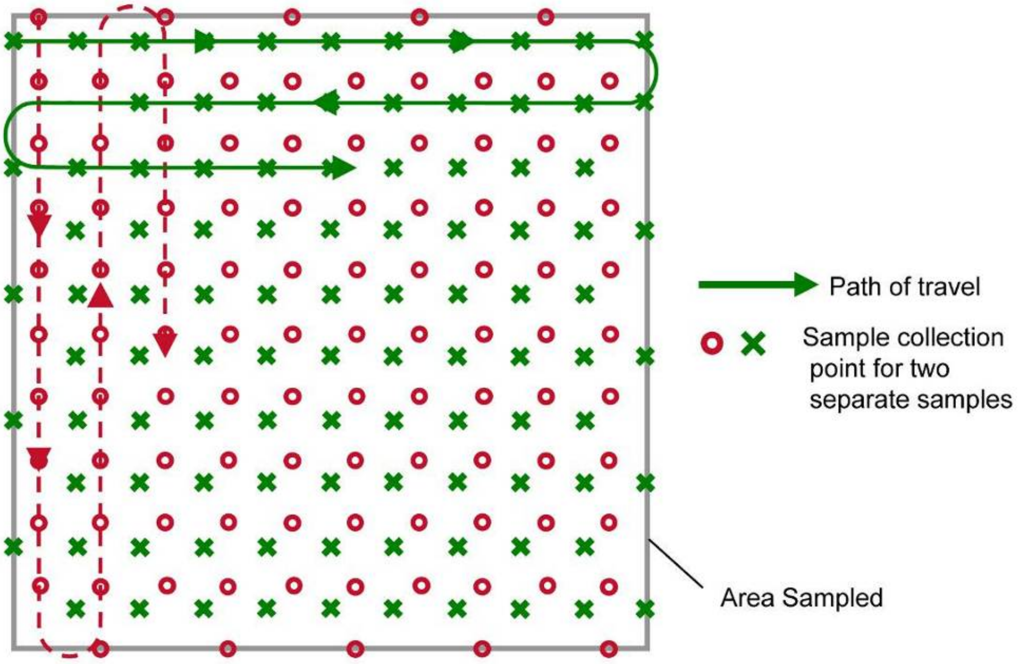

Figure 6. Illustration of *MULTI INCREMENT* sampling designs for collecting two separate samples.

After establishing the decision unit layout, either dice or a random-number algorithm was used to select locations for the box, wheel, and discrete samples. One constraint was that the central positions of the box and wheel designs were required to be such that all increments would be within the area where the *MULTI INCREMENT* samples were collected.

A constant sampling depth of 2.5 cm was used in all sampling activities. The targeted amount for discrete samples was one-half of an 8-oz (250-ml) jar, which was approximately 150 to 200 g of field-moist soil. For the 5- and 7-increment (box and wheel) sampling strategies, 5 and 7 times this amount was collected (i.e., each increment was 150 to 200 g), respectively. In the field, the 5- and 7-increment samples were thoroughly mixed in a stainless steel bowl and one or more splits were transferred to 8-oz (250-ml) jars with a large spoon. At each sampled area, one of the 5- and 7-increment samples was completely divided in the field into 5 or 7 equal splits, respectively (Figure 7). The remaining four samples of each type only had a single split sample removed.

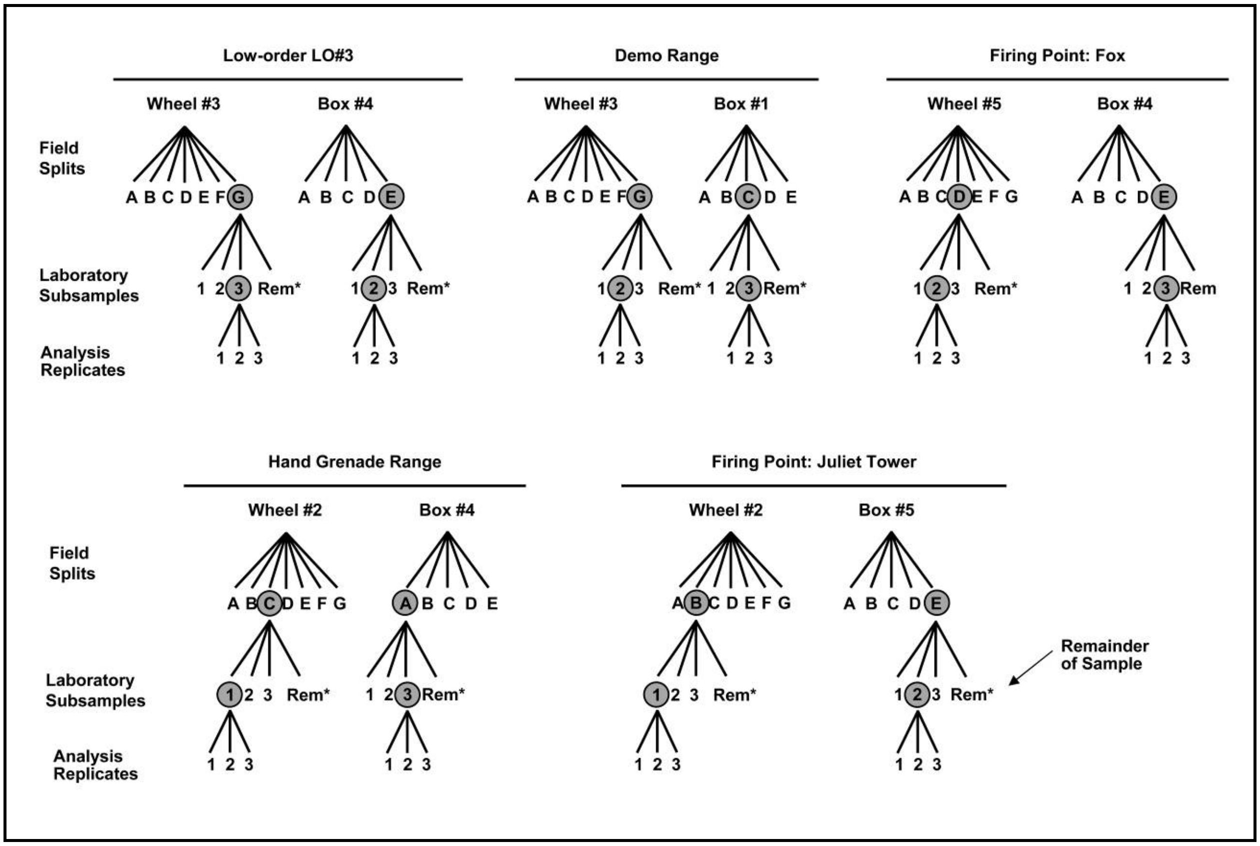

Figure 7. Illustration of sampling tree, including field splits, laboratory subsampling, and analysis replicates.

Note: The size of the split sample and the method for obtaining a field-split sample was based on what we've observed during collaborative efforts with other governmental agencies and private contractors.

Another variable evaluated in this study was whether removing the surface vegetation could potentially bias low the energetic residue concentrations. At both firing points, additional 100-increment samples were obtained following the recommended sampling design, but with an extra step. In the extra step, either the surface vegetation was removed prior to collecting an increment or each increment was divided, separating vegetated layer from subsurface layer, prior to insertion in the sample bag.

3.5.7 Sampling plan

This demonstration compares the level of uncertainty in data obtained when applying a *MULTI INCREMENT* sampling strategy and whole sample processing protocol, as described in Method 8330B, with the uncertainty resulting from discrete sampling and the two common sample collection and handling procedures currently used for characterization of energetic residues on military training ranges. In addition, the cost associated with the collection and analysis of a statistically appropriate number of discrete and *MULTI INCREMENT* samples will be compared by calculation of the upper confidence limit of the mean using the computer program ProUCL (USEPA 2007).

Developing performance or acceptance criteria desired for a project and selecting a resource-effective sampling and analysis plan is addressed in Steps 6 and 7 of the EPA QA/G-4 process (USEPA 2006b). The objective of this demonstration is to show that *MULTI INCREMENT* sampling strategy and whole sample processing is the most cost-effective way to meet the acceptance criteria necessary for estimating the mean concentration of energetic residues on military training ranges.

The compounds analyzed in this study included those energetic compounds commonly found at military training ranges. All the demonstration sites had been previously investigated and energetic residues had been both qualitatively and quantitatively estimated using Method 8330; in some cases Method 8095 was also used to analyze sample extracts. This analysis was limited to HMX, RDX, TNT 2,4-DNT, 2,6-DNT, 2-AmDNT, 4-AmDNT, and NG, the analytes that had been previously found at these sites.

Sample collection and handling protocols followed the guidelines provided in Method 8330B. The other quality assurance methods used during the preparation and analysis of the samples for this demonstration are listed in the Quality Assurance Project Plan (QAPP) in Appendix C.

3.5.8 Sample collection

After initial reconnaissance and selection of a square sampling area, tape measures and range finder binoculars were used to position pin flags at the four corners and along at least two opposite sides. All samples were collected using either stainless steel corers (Walsh et al. 2004) or stainless steel scoops. A 3-cm diameter-coring tool was used for all *MULTI INCREMENT* samples. The box, wheel, and discrete samples were collected with short nose scoops, with one exception. The vegetative cover at FP Juliet Tower made it more practical to use the coring tool for collecting the discrete samples. For consistency, a 2.5-cm sampling depth was used in all sampling activities during this demonstration.

There were five box and five wheel samples collected at each sampling area. Prior to collection, one box and one wheel sampling location were randomly selected to be evenly split into five or seven portions, respectively. At the remaining four box and wheel sampling locations, only a single split was taken for analysis. Mixing the sample in the field was performed with a stainless steel spoon at every area except for the Fort Richardson impact range located on the tidal flat. The water-saturated silts at this site could not be easily stirred; instead, they were thoroughly kneaded with gloved hands prior to splitting.

Fort Richardson, Firing Point Fox. The sampling unit at this firing point was a 40 × 40-m area that covered nearly half of the open terrain at this firing point. It was approximately twice the size of an area that Walsh et al. (2007) have been investigating for the last two years (Figure 8). The surface of this range was moderately vegetated with shrubs, grasses, and mosses, and in a few places the surface was mostly covered with sand or sand and gravel.

Pin flags were placed at the four corners of the 40 × 40-m area and at 4-m intervals along the east and west sides. Dice were used to randomly select the unique positions for the five box and five wheel samples. A combination of dice and computer-generated random numbers was used to select unique locations for the 100 discrete samples. Sampling locations were based on two whole numbers, each between 0 and 40 (1-m resolution). The position was located by moving northward from the southeast corner by using the pin flags and then westward by taking walking strides judged to be equivalent to one meter. These randomly located discrete sampling points within the 40 × 40-m area are listed in Table E-1 in Appendix E. Within the 40 × 40-m area, we collected 15 100-increment samples, comprised of one randomly positioned increment from each 4 × 4-m sub-grid. The last 5 of the 100-increment samples were collected after the surface vegetation (moss and short grasses) had been removed.

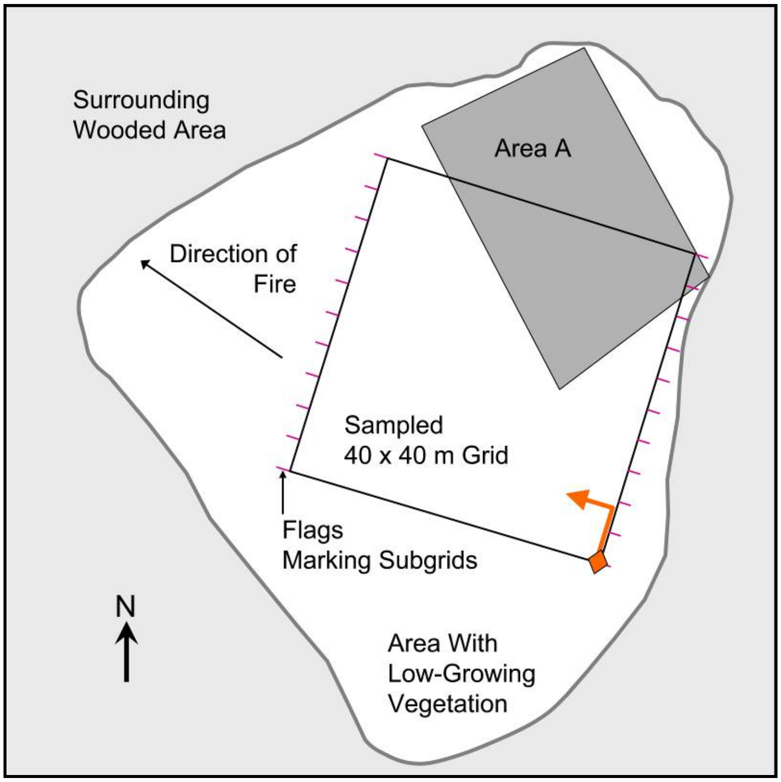

Figure 8. Sampling layout at Firing Point Fox, showing the 40-m x 40-m grid and Area A that was investigated by Walsh et al. (2007). The diamond symbol and bent arrow mark the corner and directions used to determine sample locations.

Fort Richardson, Demolition Range #3. The 30 × 30-m area chosen for the sampling at this demolition range encompassed a 10 × 10-m area that had been sampled the previous year (Figure 9). The area had been heavily used, as evident by the lack of vegetation and also the visible pieces of C4 (91% RDX) on the surface. After setting flags at the four corners and at 3-m intervals around the edges, the surface was visually inspected to delineate areas for avoidance (no sampling) with ribbon flagging. The six locations marked for avoidance had small visible pieces of C4 on the surface that could not be removed easily. The methods that had been used at FP Fox for the selection of sampling locations for the discrete, box, wheel, and *MULTI INCREMENT* sampling designs and strategies were also used for this site. In this case, sampling location designations were based on two whole numbers between 0 and 30 (providing 1-m resolution). Positions were located by moving west-southwest from the north-northeast corner then south-southeast. Likewise, there were 10 of the 100-increment samples collected by combining increments from a randomly chosen position in each of the 100 3 × 3-m sub-grids.

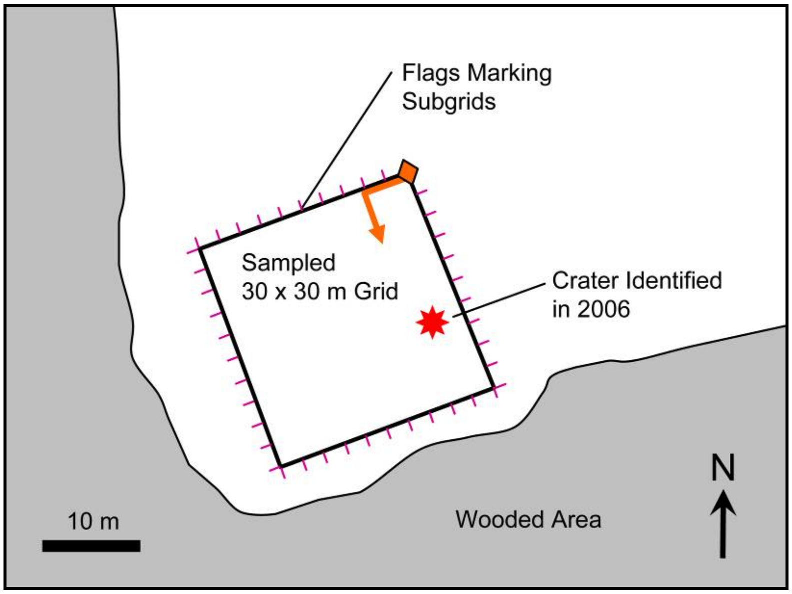

Figure 9. Sampling layout at Demolition Range #3 showing the 30 x 30-m grid orientation. Diamond symbol and bent arrow mark the corner and directions used to determine sample locations.

Fort Richardson, Central Impact Range. At this site, a 20 × 20-m area was chosen that encompassed the location where we found 133 pieces of Composition B [Comp B (60% RDX, 40% TNT)] and a small crater containing a large piece of the tail assembly from a low-ordered 120-mm mortar round (Figure 10). This tidal flat location is flooded with water several times a year. During the sampling activity, the surface was composed of water-saturated silts (mud) with very little vegetation. During the current sampling event, flags were set at the four corners, at 2-m intervals around the exterior and around the remaining pieces of Comp B (marked for avoidance, e.g., no sampling). Dice were used to randomly select locations for the 5-increment box and 7-increment wheel sampling locations within the 20 × 20-m area. They were also used to randomly select 12 unique locations to be sampled within each 2 × 2-m sub-grid. Ten of these locations were used to collect the 100-increment samples and two were used for the collection of 200 discrete samples. Positions were located by moving north from the south-east corner then west, for both the sub-grids and for the 20 × 20-m area.

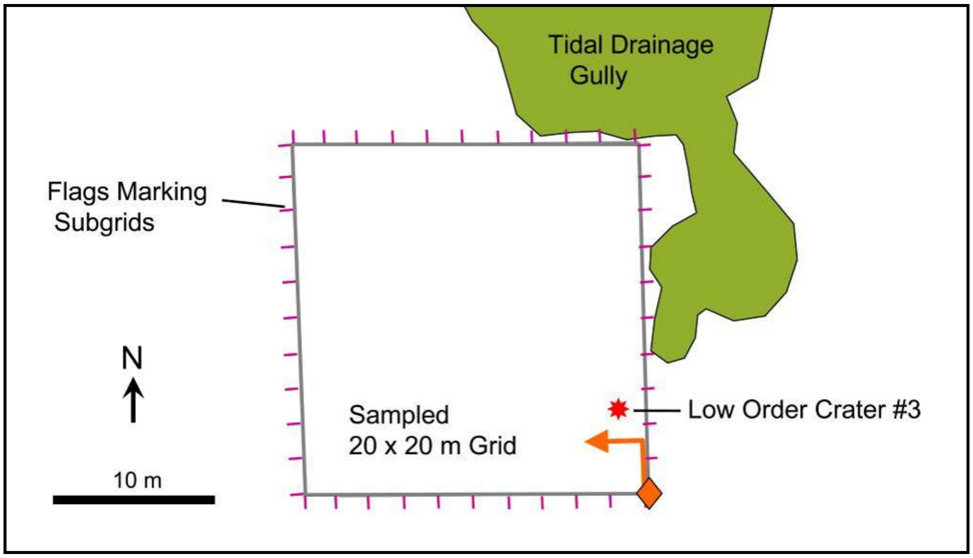

Figure 10. Sampling layout at Low Order #3 showing the 30 x 30-m grid orientation. Diamond symbol and bent arrow mark the corner and directions used to determine sample locations.

CFB Petawawa, Hand Grenade Range. The 20 × 20-m area selected for sampling at this site was 5 m in front of the grenade castle in the middle of the impact range (Figure 11). This range is used for training with the C13 (same as the M67) hand grenade, which contains Comp B. When grenades fail to detonate, they are blown-in-place with C4 (Pennington et al. 2006, Chapter 3). The surface of this range consisted of coarse sand with no vegetation. Pin flags were positioned at the four corners and at 2-m intervals around the exterior. Dice were used to randomly select locations for the box and wheel sampling locations within the 20 × 20-m area. They were also used to randomly select 10 different sampling positions within each 2 × 2-m sub-grid to be used for collecting the 100-increment samples. Positions were located by moving northward from the south-east corner then to the southwest, for both the sub-grids and for the 20 × 20-m area. A discrete sample was collected in the far right hand corner away from the firing line in each 2 × 2-m sub-grid, for a total of 100 discrete samples.

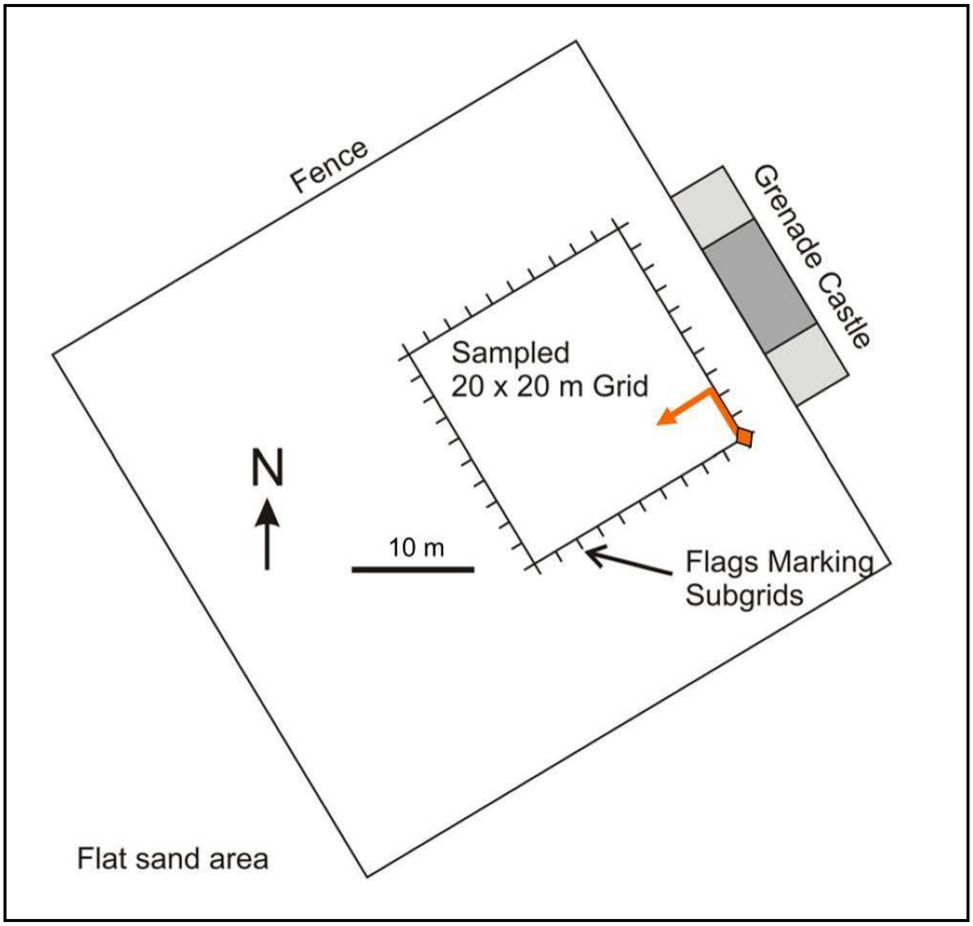

Figure 11. Sampling layout at the Hand Grenade Range. Diamond symbol and bent arrow mark the corner and directions used to determine sample locations.

CFB Petawawa, Firing Point Juliet Tower. The 30 × 30-m area chosen for sampling was heavily vegetated (grasses and short bushes), littered with pieces of plastic debris from large caliber salvo rounds, and had several vehicle ruts (Figure 12). This same approximate area had been sampled in 2005. Pin flags were positioned at the four corners and at 3-m intervals around the exterior. Dice were used to select sampling locations as was done previously. Positions were located by moving north from the south-east corner then west, for both the sub-grids and for the 30 × 30-m area. The 100 discrete samples were obtained by combining three increments collected with the coring tool from near the middle of each 3 × 3-m sub-grid. Two additional 100-increment samples were collected with each increment being split, separating the vegetation from the subsurface soil. The two portions were placed in separate bags, thereby creating two bags with mostly vegetation and two with a combination of soil and roots.

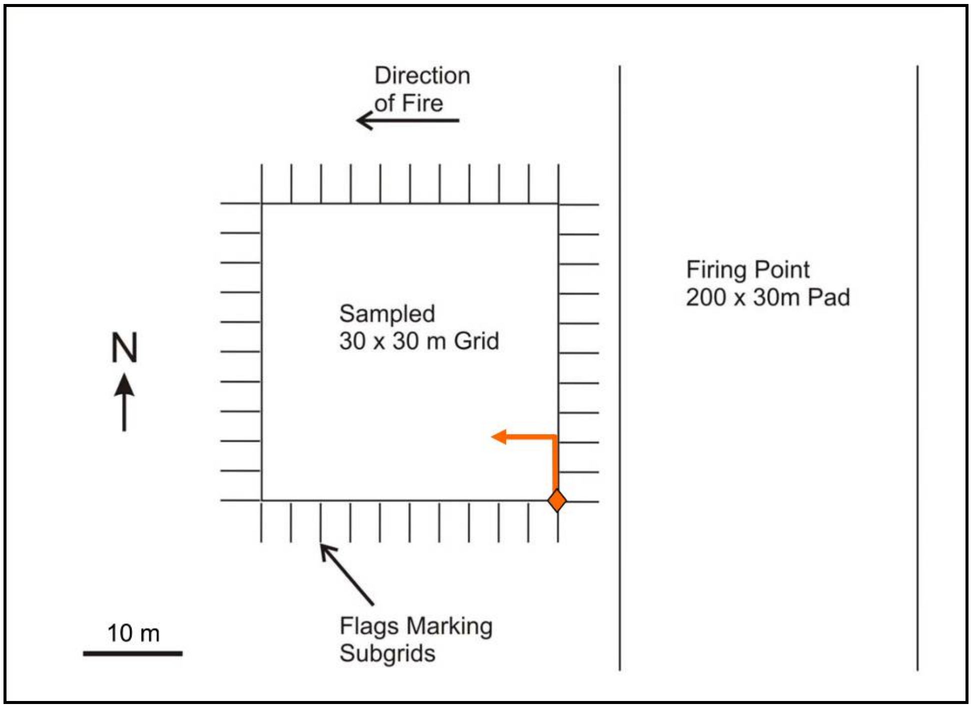

Figure 12. Sampling layout at FP Juliet Tower. Diamond symbol and bent arrow mark the corner and directions used to determine sample locations.

3.5.9 Sample handling and processing

Soil samples from each of the five chosen areas were stored, shipped, and processed together. Samples were collected with clean, stainless steel tools and were transported in either pre-cleaned 8-oz (239-mL) amber glass, wide-mouth bottles with Teflon-lined lids or in clean plastic bags. Each sample was labeled in the field at the time of collection with a permanent marker. The *MULTI INCREMENT* samples were double-bagged; the outside bag and a tag were both labeled and the tag was attached to the outer bag with a tie-wrap. The box, wheel, and a few discrete samples collected in jars were returned to cardboard boxes after the lids had been labeled. Discrete samples were collected in small plastic bags; each was labeled, then placed into a larger plastic bag containing several discrete samples. The information placed on each discrete sample included the sampling area and sampling location. The box and wheel samples also included the samplers' initials and the date. The *MULTI INCREMENT* samples included all of this information along with the number of increments. All of this information was also recorded in field notebooks. On site, the samples were placed in refrigerated storage and were transported within 24 hours to ERDC-CRREL, either by car or by contracted carrier.

At the ERDC-CRREL laboratory, all information placed on bags, lids or tags was transferred to the sample logs, and each sample was given a laboratory number. Samples were held in a refrigerated room at 4°C until they were air-dried. Samples were set out to air dry within one week of being received. *MULTI INCREMENT* samples were spread out on large sheets of aluminum foil; split samples from the box and wheel sampling designs were placed in aluminum pans; the majority of the discrete samples were dried in the original sample bag after opening and turning down the sides. All soil samples except those from the Fort Richardson impact range dried in 24 to 48 hours. The water-saturated, fine-grained silts from the Fort Richardson impact range took about a week to completely air dry.

Once air-dried, *MULTI INCREMENT* samples were weighed, then passed through a 10-mesh (2-mm) sieve; both fractions were placed into separate bags and re-weighed. The < 2-mm fraction of the *MULTI INCREMENT* samples was ground in aliquots not exceeding 600 g in a Lab TechEssa LM2[13] puck mill grinder for either 90 seconds or for 5 × 60-second cycles, with a 60-second cool-down period between each grinding cycle. Samples from the firing points and the demolition range received the longer grinding treatment, whereas those from the impact and hand grenade ranges received the shorter treatment. Samples with only crystalline energetic residues can be adequately ground in 90 seconds whereas samples with polymeric residues require additional grinding (Walsh et al. 2005). After grinding, each entire sample was thoroughly mixed by returning all of the aliquots to the sample bag and manually shaking. The mixed sample was then spread out on a large sheet of aluminum foil. Subsamples of 10.0 g (±0.1 g) were obtained by combining at least 30 increments from the entire exposed surface into 2-oz (59 mL) amber wide-mouth glass bottles with Teflon-lined lids. Subsample triplicates were removed from one out of every five *MULTI INCREMENT* samples. Likewise, subsamples for the matrix spike and matrix spike duplicate quality assurance tests were obtained following this same procedure. Once subsampling was completed, the remaining sample was returned to the bag and archived at ERDC-CRREL. Each 10.0g (±0.1 g) subsample was extracted with 20.0 mL of acetonitrile on a platform shaker overnight (18 hours at 150 rpm).

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[13] A product of Essa Worldwide, with headquarters in Bessendean, Western Australia. More information available at (accessed June 2009): www.essaaustralasia.com

Split samples from the box and wheel sampling protocols were transported to ERDC-CRREL in 8-oz (250-mL) jars with Teflon-lined lids. In addition, five discrete samples collected at FP Fox and five from the Demolition Range #3 were transported in these same 8-oz jars. Prior to air-drying any of the split samples and these 10 discrete samples, we chose certain box, wheel, and discrete samples from each area to undergo an additional subsampling experiment. One of the box and wheel splits chosen for subsampling at each site was from the position where the entire sample had been split into five or seven retained equal portions (Figure 7). The remaining samples (those not being subsampled) were transferred to large aluminum pans for air-drying. Once dry, they were passed through a 10-mesh sieve and the sieved portion was weighed. If that portion was under 120 g, it was returned to the 8-oz (239 mL) sample jar; if the sieved weight exceeded 120 g, it was placed in a new 16-oz (475 mL) jar with a Teflon-lined lid to accommodate the volume of solvent needed for extraction. This entire sieved fraction of the split sample was then extracted with a volume of acetonitrile (in milliliters) approximately double the mass (in grams). Extraction of these sample splits was performed on platform shaker overnight (18 hours at 150 rpm).

The samples selected for the additional subsampling experiment were first stirred with a stainless steel spatula. Triplicate subsamples were then obtained by removing and transferring approximately 20 g to small (~9-cm diam.) aluminum pans to air dry. The remainders of these split or discrete samples were transferred to large (~25-cm diam.) aluminum pans for air-drying. When they were dry, the subsamples were passed through a 10-mesh sieve and approximately 10 g of the sieved material was transferred to a 2-oz (59 mL) amber glass bottle and extracted as described above. The remainder of both fractions of each subsample that was not taken for extraction was then recombined with the appropriate split or discrete sample. After these split and discrete samples had air-dried, they each were processed and extracted as described in the previous paragraph.

Note: The sequence of steps described above, in which only a small portion is removed from a field-moist sample, is a common practice for laboratories that are still following Method 8330. That is, only a very small portion of the sample received by the laboratory is removed for further processing and analysis, a protocol that has been referred to as the “scoop off the top” subsampling method.

The air-dried discrete samples, a majority of which had been transported in small plastic bags, were passed through a #10 sieve, the sieved portion was weighed and transferred (returned in some cases) to either an 8-oz or a 16-oz jar with a Teflon-lined lid, depending if the weight of the sieved fraction was less than or exceeded 120 g, respectively. The entire sieved fraction of the discrete samples were extracted using a volume of acetonitrile (in milliliters) about double the mass of the sieved fraction (in grams) by shaking overnight on a platform shaker (18 hours at 150 rpm).

3.5.10 Sample analysis

All of the samples were analyzed at ERDC-CRREL following the guidelines in Method 8330B (USEPA 2006a). Briefly, following the 18-hour extraction period on a tabletop shaker, each sample was vigorously hand shaken and the soil was allowed to settle for one hour. An aliquot of each extract was then passed through a 0.45-µm Millex FH filter[14] and transferred to a 7-mL glass amber vial. These sample extracts were stored in a freezer until the day of analysis.

An analysis batch was typically composed of 20 to 50 samples and included at least one laboratory control sample and a sample preparation blank. On the day of analysis the sample extracts were allowed to warm to room temperature. While the extracts of samples from the impact and demolition ranges at Fort Richardson warmed, a small aliquot was removed and screened with the Expray™ Kit[15] colorimetric reagents (Bjella et al. 2005). The procedure involved removing a small portion with a disposable glass pipette, placing a drop on special paper, and spraying with the Expray™ Kit aerosol reagents. This screening step identified a few samples that should be diluted 10-fold prior to analysis, so as to keep the concentration estimates within the linear range of the calibration. The final preparation step prior to high performance liquid chromatography (HPLC) analysis was to mix one part of the acetonitrile extract (or diluted extract) with three parts reagent-grade water.

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[14] A product of Millipore, with headquarters in Billerica, Massachusetts. More information available at (accessed June 2009): www.millipore.com

[15] Manufactured by the Plexus Scientific, headquartered in Alexandria, VA. More information is available at (accessed June 2009): www.plexsci.com

Analysis was performed on a HPLC system from Thermo Finnigan. This modular system had SpectraSYSTEM® Model P1000[16] isocratic pump with a 100 µL sample loop, a SpectraSYSTEM UV2000 dual wavelength UV/Vis absorbance detector set at 210 and 254 nm (1 cm cell path), and a SpectraSYSTEM AS3000 auto-sampler. The primary separation was made on a 15-cm × 3.9-mm (4-µm) NovaPak C-8 column[17] maintained at 28°C and eluted with 15:85 isopropanol/water (v/v) at 1.4 mL/min. Secondary (confirmation) separation was made on a 25-cm × 4.6-mm (5-µm) Supelcosil Liquid Chromatograph-Cyanoproply (LC-CN) column (Supelco[18]) maintained at 30°C and eluted with 65:25:10 reagent-grade water, methanol, and acetonitrile (v/v) at 1.3 mL/min. Concentrations were estimated from peak height measurements compared to commercial (Restek Corp.[19]) multi-analyte and single analyte standards.

Table 3 lists the estimated reporting limits (ERL) used for energetic residues detected in this study. These limits were based on values five times the method detection limit (MDL) values that were established for this study (Federal Register 1984). The MDLs were determined by spiking 10.0 g of commercial sand with two quantities of analytes, 0.750 and 1.50 µg, then extracting with 20.0 ml of acetonitrile after allowing them to dry for 1 hour. These spiked samples were extracted for 18 hours on a shaker table (150 RPM).

Table 3. Method detection limits (MDL) and estimated reporting limits (ERL) for selected energetic residues in soil by RP-HPLC.

Analyte	mg/kg	
	MDL	ERL
HMX	0.0059	0.029
RDX	0.0076	0.038
TNT	0.0070	0.035
2,4-DNT	0.0062	0.031
2,6-DNT	0.0098	0.049

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[16] SpectraSYSTEM® products are produced by Thermo Fisher Scientific Inc., headquartered in Waltham, MA. More information available (accessed June 2009) at: www.thermo.com

[17] A product of Waters Chromatography Division, of Milford, MA. More information available (accessed June 2009) at: www.waters.com

[18] A brand name of the Sigma Aldrich Company. More information available (accessed June 2009) at: www.sigmaaldrich.com

[19] The Restek Corporation of Bellefonte, PA is a leading manufacturer of chromatography equipment. More information available (accessed June 2009) at: www.restek.com

Analyte	mg/kg	
	MDL	ERL
2AmDNT	0.0081	0.040
4AmDNT	0.022	0.11
NG	0.013	0.065

A selected number of sample extracts were prepared and analyzed three times, to allow estimation of analytical error. These extracts were placed within two different analytical run batches and thus, were analyzed over the course of 2 or 3 separate days.

Matrix spikes (MS) and matrix spike duplicates (MSD) were performed on a *MULTI INCREMENT* sample collected at each of the sampling locations. The spikes added to the sample matrix were approximately twice the estimated concentration that had been established during the initial analysis. Recoveries of the theoretical or targeted analyte concentrations in these MS and MSD ranged from 88% to 102%, suggesting that no apparent matrix interferences were present (Table 4).

Table 4. Matrix spike (MS) and matrix spike duplicate (MSD) results for the analytes detected at the five sampling areas.

Sample	Units	HMX	RDX	TNT	2,4-DNT	NG
Low Order #3						
MI-5	mg/kg	2.19	13.0	1.14		
Matrix Spike		2.28	13.3	0.560		
Target Value	mg/kg	4.47	26.3	1.70		
MS - result	mg/kg	4.54	24.8	1.66		
MSD - result	mg/kg	4.58	24.8	1.66		
MS - Recovery	%	101%	94%	98%		
MSD - Recovery	%	102%	94%	98%		
Demolition Range #3						
MI-10A,B,C	mg/kg	2.59	11.9	0.057	6.15	
Matrix Spike		1.82	10.6	0.448	4.95	
Target Value	mg/kg	4.41	22.5	0.505	11.1	
MS - result	mg/kg	4.12	21.4	0.460	11.4	
MSD - result	mg/kg	4.42	22.0	0.496	10.6	
MS - Recovery	%	93%	95%	91%	102%	
MSD - Recovery	%	100%	98%	98%	96%	

Sample	Units	HMX	RDX	TNT	2,4-DNT	NG
Hand Grenade Range						
MI-4 A,B,C	mg/kg	0.107	0.134			
Matrix Spike		0.100	0.100			
Target Value	mg/kg	0.207	0.234			
MS-result	mg/kg	0.194	0.228			
MSD-result	mg/kg	0.196	0.226			
MS - Recovery	%	94%	97%			
MSD - Recovery	%	95%	97%			
Firing Point Juliet Tower						
MI-6 A,B,C	mg/kg				0.690	2.42
Matrix Spike					1.00	1.00
Target Value	mg/kg				1.69	3.42
MS-result	mg/kg				1.49	3.28
MSD-result	mg/kg				1.61	3.18
MS - Recovery	%				88%	96%
MSD - Recovery	%				95%	93%
Firing Point Fox						
MI-10	mg/kg					4.99
Matrix Spike						4.52
Target Value	mg/kg					9.52
MS-result	mg/kg					8.92
MSD-result	mg/kg					9.40
MS - Recovery	%					94%
MSD - Recovery	%					99%

A dual column analysis was performed on a selected number of sample extracts of *MULTI INCREMENT* samples to confirm the identity of the analytes present at each of the 5 demonstration sampling locations (Table 5a and Table 5b). In addition, selected sub-samples and sample extracts from *MULTI INCREMENT* samples collected at two locations were analyzed by liquid chromatography – mass spectrometry (LC-MS) and liquid chromatography – tandem mass spectrometry (LC-MS-MS) at an independent commercial laboratory (Table 6). All of the analytes were confirmed by the analysis performed on the LC-CN column and by LC-MS and LC-MS-MS analysis that had been originally detected during the primary analysis. Moreover, in most cases very similar concentrations were obtained when compared to the primary column results. The one case where there was a consistent discrepancy was for the determination of HMX in the *MULTI INCREMENT* samples from the hand grenade range (Table 5). Because of generally low concentrations and poor agreement between the two analyses, caution should be used when interpreting these HMX results from the hand grenade range.

3.5.11 Experimental controls

Laboratory control sample (LCS) and blanks were processed and analyzed with every analysis batch. The laboratory control sample is commercial sand that is spiked and extracted with a batch of samples. In the majority of cases a 10.0 g quantity of sand was spiked with 10 µg/mL quantities of the analytes of interest. Table 7 shows the percent recoveries for the LCS run with the different batches of samples. With the exception of 2,6-DNT all of the recoveries were within 15% of the spiked concentration. In the one case where there was less than 80% recovery of 2,6-DNT, there was no detection of this analyte in the corresponding run batches, i.e., two batches of the discrete samples from the Low-order #3 area in the impact range at Fort Richardson (Table 7).

The blank was the same commercial sand that was used for the LCS. This soil was handled, processed, and analyzed along with a batch of samples. For the multi increment samples a 500 g quantity was set out to dry with a batch of field samples then sieved, ground, subsampled, extracted, diluted and analyzed. For the sample box and wheel split samples, and for the discrete samples, the blanks were composed of 100 g of soil that were placed out to dry with a batch then sieved, extracted, diluted and analyzed. No analytes were detected above their ERL in these laboratory blanks (Table 8).

Method 8330B recommends that entire < 2 mm fraction of the sample be pulverized prior to subsampling. When performed in the Lab TechEssa LM2 (LabTech Essa Pty. Ltd., Bessendean, WA, Australia) puck mill sited in section 3.5.4, the majority (>95%) of the matrix and constituents of concern are reduced to particles < 75 µm. To demonstrate that no analytes are lost during this operation as described in this report and in Method 8330B a performance evaluation (PE) material was ground for 90 sec and for an additional four, 60-second periods prior to subsampling, extraction, and analysis. Two grinding periods were used to address the protocols for the preparation of both crystalline and polymeric energetic residues. This PE material was a custom standard prepared for the USACE Hazardous, Toxic, and Radioactive Waste (HTRW) Center of Expertise (CX) in Omaha Nebraska, and was produced and certified by Environmental Resource Associates (ERA). The PE was composed of 500 g of soil that had been treated with all of the Method 8330B analytes with the exception of tetryl, and had also been treated with NG and PETN. The average recoveries for seven PE samples following the grinding protocols recommended in Method 8330B are shown in Table 9. In general, the analyte recoveries after grinding are greater than, or well within, the standard deviation of values from historical data.

Table 5a. Selected MULTI INCREMENT sample analyte confirmation by dual column analysis. — Demolition, Impact, Hand Grenade Range Results.

	C-8 Column Results (mg/kg)					LC-CN Column Results (mg/kg)				Ratio C-8 / LC-CN			
Sample	HMX	RDX	TNT	2,4-DNT	2,6-DNT	HMX	RDX	TNT	2,4-DNT	HMX	RDX	TNT	2,4-DNT*
Demolition Range #3													
MI-1	1.32	4.66	0.078	8.28		1.40	4.86	0.072	8.18	0.94	0.96	1.08	1.01
MI-2	5.54	27.4	0.128	9.04	0.116	5.36	28.0	0.118	8.82	1.03	0.98	1.08	1.04
MI-3	18.8	126		4.22		18.3	127		4.18	1.03	0.99		1.01
MI-4	2.46	9.44	0.258	5.58		2.24	9.44	0.276	5.52	1.10	1.00	0.93	1.01
MI-5A	1.63	7.20		6.36		1.55	7.28		6.26	1.05	0.99		1.02
Low Order #3													
MI-1	10.8	81.8	25.2			11.6	86.8	26.4		0.93	0.94	0.95	
MI-2	1.78	7.26	0.550			1.86	6.96	0.476		0.96	1.04	1.16	
MI-3	3.92	26.2	7.52			3.98	25.6	7.56		0.98	1.02	0.99	
MI-4	3.18	15.0	1.85			3.32	15.2	1.87		0.96	0.99	0.99	
MI-5A	2.16	12.8	1.11			2.14	12.7	1.11		1.01	1.01	1.00	
Hand Grenade Range													
MI-1	0.106	0.326				0.078	0.330			1.36	0.99		
MI-2	0.110	0.176				0.072	0.166			1.53	1.06		
MI-3	0.126	0.812				0.102	0.842			1.24	0.96		
MI-4A	0.107	0.134				0.080	0.134			1.33	1.00		
MI-5	0.144	0.138				0.070	0.136			2.06	1.01		

* C-8 column values for 2,4-DNT and 2,6-DNT were combined. These analytes co-elute on the LC-CN column.

Table 5b. Selected *MULTI INCREMENT* sample analyte confirmation by dual column analysis – firing point location results.

	C-8 Column Results (mg/kg)		LC-CN Column Results (mg/kg)		Ratio C-8 / LC-CN	
Sample	2,4-DNT	NG	2,4-DNT	NG	2,4-DNT	NG
Firing Point Fox						
MI-1		4.54		4.78		0.95
MI-2		4.94		5.08		0.97
MI-3		5.22		5.40		0.97
MI-4		7.00		7.48		0.94
MI-5A		65.2		64.4		1.01
Firing Point Juliet Tower						
MI-1	0.713	2.41	0.699	2.32	1.02	1.04
MI-3	0.581	3.13	0.591	3.08	0.98	1.02
MI-5	0.874	2.78	0.878	2.71	1.00	1.02
MI-6	0.696	2.47	0.690	2.42	1.01	1.02
MI-7	1.05	2.99	1.05	2.87	1.00	1.04

Table 6. Selected *MULTI INCREMENT* sample extracts and subsamples analyzed by HPLC-UV, LC/MS, and LC/MS/MS. Unless specified, results are from analysis of a single sample.

Sample	Analyte	Extract (mg/kg)			Subsample (mg/kg)		
		CRREL HPLC-UV	TA* LC/MS	TA LC/MS/MS	CRREL HPLC-UV	TA LC/MS	TA LC/MS/MS
					(n=3)		
LO#3 MI#5	HMX	2.16	2.68	3.10	2.16±0.06	2.65	2.85
	RDX	12.8	13.0	11.9	13.0±0.36	13.4	13.2
	TNT	1.11	1.11	1.22	1.14±0.04	1.10	1.25
					(n=3)		
LO#3 MI#10	HMX	6.64	9.07	10.2	6.67±0.09	7.70	8.39
	RDX	50.8	49.2	49.1	50.6±0.20	49.4	49.7
	TNT	25.6	25.8	26.6	25.5±0.12	27.7	26.0
LO#3 MI#2	HMX	1.78	2.00	2.36	1.78	1.79	2.04
	RDX	7.28	7.13	7.29	7.28	6.36	6.34
	TNT	0.55	0.454	0.516	0.55	0.454	0.505
LO#3 MI#6	HMX	1.76	2.09	2.40	1.76	1.81	2.07
	RDX	7.68	7.56	7.73	7.68	7.27	6.99
	TNT	0.800	0.800	0.738	0.8	0.871	0.794
LO#3 MI#3	HMX	3.92	5.37	5.94	3.92	4.89	6.07
	RDX	26.2	26.0	23.2	26.2	24.9	24.1
	TNT	7.52	7.80	8.02	7.52	7.65	6.89

Sample	Analyte	Extract (mg/kg)			Subsample (mg/kg)		
		CRREL HPLC-UV	TA* LC/MS	TA LC/MS/MS	CRREL HPLC-UV	TA LC/MS	TA LC/MS/MS
FP Fox MI#5	NG	61.0	60.0	57.60	(n=3) 62.8±2.16	57.3	51.5
FP Fox MI#10	NG	5.12	3.10	2.47	(n=3) 4.99±0.16	4.42	3.99
FP Fox MI#6	NG	3.98	4.60	4.16	3.98	3.98	3.79
FP Fox MI#2	NG	4.94	5.51	5.42	4.94	4.81	4.18

* TA – Test America

Table 7. Laboratory control sample recoveries for analysis batches.

Sampling area	Sample type	Percent Recovery (%)							
		HMX	RDX	TNT	2,4-DNT	2,6-DNT	2AmDNT	4AmDNT	NG
Demolition Range	Discrete	101	94	95	94	81	101	97	95
	Discrete	103	95	102	98	84	103	100	100
FP Fox	Discrete	100	94	97	94	82	99	97	95
	Discrete	105	97	104	100	86	104	101	100
Low-order# 3	Discrete	100	94	94	86	68	99	96	89
	Discrete	101	94	98	89	70	98	95	92
	MULTI INCR.	99	92	98	95	87	98	96	102
	Box/Wheel Subsamples	97	92	94	92	92	90	90	94
	Field Splits	95	98	100	99	98	95	96	97
FP Fox	*MULTI INCR.*	103	101	97	98	92	102	103	97
FP Fox and Demolition Range	Box/Wheel Subsamples	101	95	90	95	93	95	95	95
	Field Splits	100	97	97	94	86	97	95	91
Demolition Range	*MULTI INCR.*	104	99	95	100	97	100	98	99
Hand Grenade Range	*MULTI INCR.*	102	104	102	102	100	104	102	99
	Discrete	103	105	102	103	99	101	103	102
	Discrete	94	93	92	93	93	91	92	100
Hand Grenade Range and FP Juliet Tower	Box/Wheel Subsamples	96	96	94	95	95	104	97	98
	Field Splits	98	98	97	98	97	96	98	96
FP Juliet Tower	*MULTI INCR.*	98	97	97	97	98	99	99	98
	Discrete	99	99	97	98	97	97	98	97
	Discrete	95	94	98	95	94	92	95	96

Table 8. Results for laboratory sample processing blanks.

Sampling Area	Sample Type	Concentration (mg/kg)							
		HMX	RDX	TNT	2,4-DNT	2,6-DNT	2AmDNT	4AmDNT	NG
Demolition Range	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
FP Fox	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
Low-order# 3	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	MULTI INCR.	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Box/Wheel Subsamples	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Field Splits	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
FP Fox	*MULTI INCR.*	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
FP Fox and Demolition Range	Box/Wheel Subsamples	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Field Splits	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
Demolition Range	*MULTI INCR.*	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
Hand Grenade Range	*MULTI INCR.*	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
Hand Grenade Range and FP Juliet Tower	Box/Wheel Subsamples	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Field Splits	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
FP Juliet Tower	*MULTI INCR.*	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065
	Discrete	<0.029	<0.038	<0.035	<0.031	<0.049	<0.040	<0.11	<0.065

Table 9. Average percent recoveries of nitroaromatics and nitramines in Environmental Resource Associates (ERA) custom standards.

Analyte	Certified Value (µg/kg)	Recoveries* Mean	Number of Analyses	Recoveries† 90 s Mean	Recoveries† 90 + 4x60 s Mean
4AmDNT	946	72.3±13.0%	139	91.4±2.80%	85.1±2.66%
2AmDNT	650	81.9±12.9%	148	102±1.53%	97.9±2.08%
DNB	1010	92.7±11.8%	213	97.6±1.65%	95.3±1.88%
2,4-DNT	645	92.2±14.0%	257	95.2±1.73%	93.4±2.09%
2,6-DNT	1320	90.7±15.4%	242	97.5±1.65%	94.9±1.67%
HMX	620	84.5±15.4%	188	96.1±2.86%	91.8±2.67%
NB	1400	88.5±15.0%	257	83.7±1.50%	80.8±1.23%
2-NT (o)	1460	91.9±12.5%	197	98.0±1.49%	94.0±1.27%

Analyte	Certified Value (µg/kg)	Recoveries* Mean	Number of Analyses	Recoveries† 90 s Mean	Recoveries† 90 + 4x60 s Mean
3-NT (m)	1020	96.1±13.7%	162	91.4±2.32%	89.9±1.76%
4-NT (p)	1840	92.8±15.1%	195	93.0±2.33%	90.0±1.42%
RDX	583	88.0±14.2%	176	87.2±2.73%	84.7±2.86%
TNB	696	87.0±14.5%	198	99.8±2.05%	96.8±2.42%
TNT	808	82.4±10.8%	178	95.7±2.42%	93.4±2.23%
NG	1000			96.3±1.29%	90.1±2.54%
PETN	1000			102±4.57%	100±6.12%

* Environmental Resource Associates historical data.

† Analysis of seven PE samples that were pulverized in a puck mill.

3.5.12 Data quality parameters

The objective of this study was to demonstrate that *MULTI INCREMENT* sampling is more representative and more cost effective than discrete sampling. This was accomplished by assessing total measurement error, sampling splitting error, subsampling error, and analysis error. Although it will be impossible to show that these environmental sampling protocols are accurate (agreement with the true value), improved quality was inferred through the ability to reproduce the results (better precision).

Samples that are split in the field are assumed to be representative of the location from where the increments were obtained and mixed together. That is, the split sample from a box or wheel sampling design is assumed to contain the same concentration of analytes as the bulk sample. This assumption was evaluated through the analysis of samples for which the entire sample (composed of several splits) was processed and analyzed. The procedure allowed for an assessment of precision among splits and a measurement of bias of individual splits relative to the entire sample. A similar assessment was performed with subsamples removed from samples that have not been processed as recommended in Method 8330B. For this assessment, the precision among triplicate subsamples removed from a single sample and a measurement of bias of each individual subsample was made, relative to the estimated concentration for the entire sample.

All laboratory data was reviewed for its completeness, quality, quantity, and all the QA/QC analyses were compared to theoretical values. Results for all the field samples collected for this project are given in Appendix E, Tables E1 through E10. The response factors for continuing calibration validation (CCV) standards for all the values reported in Tables E-1 through E-10 stayed with ±20% of the initial calibration. Because of the very large number of samples, three analytical columns and three initial calibrations were performed. No analytes were detected above the estimated reporting limits for the laboratory blank samples (Table 8). The majority of the values for the laboratory control samples were within ±15% of theoretical values (Table 7). In a couple of cases the recoveries of 2,6-DNT were less than 80%; however, the batch of samples these two laboratory control samples were run with did not contain this analyte. After changing the commercial soil to another brand (that was used for the laboratory control samples) all of the remaining recoveries were with the ±15% of the theoretical values.

3.5.13 Calibration procedures, quality control checks, and corrective action

Commercial standards from Restek and AccuStandards were compared, to verify certified concentrations. A 5-point or greater linear calibration between 0.05 and 40 mg/L was constructed for each of the three C8 columns used during this study. A CCV standard of 1.00 mg/L was run after every 10 analyses, and all reported concentrations (response factors) had to remain within ±20% of the initial calibration for the analyses to be accepted.

3.5.14 Data quality indicators

Data quality will be based on the criterion specified in Tables 1 and 10. The variances (standard deviation) of 3, 5, 7 or 10 replicates will be computed and compared against the corresponding performance objectives shown in Table 1.

3.5.15 Demobilization

N/A

3.6 Selection of analytical/testing methods

Method 8330B is a document developed for the ESTCP ER-0628 program (USEPA 2006a).

3.7 Selection of analytical/testing laboratory

All of the samples were analyzed in house at ERDC-CRREL facilities. It should be recognized that analytical procedures for Method 8330B, its predecessor Method 8330, and Method 8095 were all developed by personnel at ERDC-CRREL (USEPA 1994, 1999, 2006a).

4 Performance Assessment

4.1 Performance criteria

The main objective of this demonstration was to compare the uncertainty associated with estimating mean concentrations of energetic residues in areas of concern when using the sampling strategy and sample processing protocols recommended in Method 8330B with the uncertainty resulting from other commonly used practices. The performance criteria for all of the protocols evaluated are again listed in Table 10. Another objective for this demonstration is to evaluate how removing vegetation prior to obtaining a surface soil sample could influence estimates of the mean concentration.

4.2 Performance confirmation methods

The error associated with splitting a sample in the field and the removal of only a small portion of the sample for further processing in the laboratory was assessed by measuring the variation among field splits and laboratory replicates. Likewise, the subsampling uncertainty associated with whole sample processing recommended in Method 8330B was evaluated. In addition, the values obtained from discrete and *MULTI INCREMENT* data sets were compared, as were their respective estimated UCLs of the mean, calculated using ProUCL Version 4.0 (US EPA 2007). The performance criterion is based on the capability of a sampling strategy to produce mean concentration estimates of energetic residues in an area of concern (decision unit or exposure unit) with the tightest tolerances. The targeted parameters for the performance criteria are given again in Table 10 (as were previously given in Table 1).

Table 10. Performance criteria.

Primary Performance Criteria	Expected Performance (Metric)	Actual Performance (%RSD; n = 3)
Subsampling error	Representative and precise subsamples, split samples, and reproducible field samples	< 10%
Sample splitting error		< 10%
Sampling error and collection bias		< 30%*

* Total measurement error.

4.3 Data analysis, interpretation, and evaluation

As noted in Section 2.1, this evaluation focuses on assessing the error (uncertainty) associated with some commonly practiced sample handling protocols. In particular, the practice of splitting the sample in the field and laboratory subsampling prior to adequate processing was evaluated. Uncertainty associated with these two protocols was explored by assessing the variation in concentration estimates among replicates and also by comparing (evaluating bias of) the concentration estimates obtained for split samples or subsamples to the value obtained for the entire sample. Data for samples that were not split in the field were evaluated for determining an acceptable range of uncertainty for estimating UCLs of the mean.

4.3.1 Sample extract preparation and analysis

Random error associated with preparation and analysis of the sample extracts was common to all the sample collection and handling protocols evaluated in this study. To assess the uncertainty associated with these two steps, three samples from each of the five sampling areas were selected after they had been initially analyzed. The sample selection criterion was based on the presence of one or more energetic residue concentrations at least twice the estimated reporting limits that were shown in Table 3. Since this selection occurred after the initial analysis, the subsequent preparation of sample extracts and analysis occurred on a separate occasion. Among the 15 samples selected, there were 32 sets of triplicate analyte concentrations (Table 11). Overall, the relative standard deviation (RSD) for these analytical replicates ranged from 0 to 6.19%, and averaged 1.53%. The median RSD for these triplicate determinations was 0.97%. Therefore, typically the uncertainty associated with the preparation and analysis of sample extracts will have little influence on the performance criteria listed in Table 10 (and also Table 1).

4.3.2 Subsampling error

The error associated with removing subsamples from sample jars, prior to processing the entire sample, was evaluated for a box and wheel split sample from each sampling area (Figure 7) and for two discrete samples from separate areas (Table 12). Similarly, subsampling error was evaluated for two *MULTI INCREMENT* samples from each of the five sampling areas (Table 13). For the box, wheel, and discrete samples, the 8-oz sample jar was opened and three separate quantities of approximately 20 g were removed with a stainless steel spatula after briefly stirring the contents. After air-drying, each subsample was passed through a #10 (2 mm) sieve and a 10±1.5 g portion of the sieved material was extracted for analysis. The remainder of both fractions (less than and greater than 2 mm) of each subsample was returned to the original sample prior to this larger portion being sieved and extracted for analysis. The *MULTI INCREMENT* samples were air-dried, sieved, ground, mixed, and spread out in a thin layer prior to removing triplicate 10.0±0.1 g subsamples by collecting $\geq$ 30 increments with a stainless steel spatula (USEPA Method 8330B).

Table 11. Analysis replicates.

Sample		Concentration (mg/kg)				
Site / Position	Replicate	HMX	RDX	TNT	2,4-DNT	NG
Low-Order #3						
Wheel 3G sub-3	Rep-1	2.10	14.8			
	Rep-2	2.08	14.9			
	Rep-3	2.08	14.9			
	Average	2.08	14.8			
	Std Dev	0.013	0.022			
	RSD	**0.65%**	**0.15%**			
Box 4E sub-2	Rep-1	41.8	269	28.7		
	Rep-2	41.5	272	28.7		
	Rep-3	41.8	274	28.9		
	Average	41.7	272	28.8		
	Std Dev	0.143	2.47	0.143		
	RSD	**0.34%**	**0.91%**	**0.50%**		
Wheel 4 sub-2	Rep-1	0.232	1.44	0.455		
	Rep-2	0.232	1.44	0.449		
	Rep-3	0.232	1.45	0.447		
	Average	0.232	1.44	0.450		
	Std Dev	0.000	0.009	0.004		
	RSD	**0.00%**	**0.60%**	**0.93%**		
Wheel 3G Sub-2	Rep-1	3.24	12.4		0.052	
	Rep-2	3.28	12.6		0.046	
	Rep-3	3.26	12.5		0.050	
	Average	3.260	12.5		0.049	
	Std Dev	0.020	0.083		0.0031	
	RSD	**0.61%**	**0.67%**		**6.19%**	

Sample		Concentration (mg/kg)				
Site / Position	Replicate	HMX	RDX	TNT	2,4-DNT	NG
Demo Range #3						
Box 1C Sub-3	Rep-1	0.496	0.460		0.108	
	Rep-2	0.500	0.466		0.114	
	Rep-3	0.506	0.472		0.114	
	Average	0.501	0.466		0.112	
	Std Dev	0.005	0.006		0.0035	
	RSD	1.01%	1.29%		3.09%	
Discrete-3 Sub-1	Rep-1	0.312	1.21		0.058	
	Rep-2	0.324	1.24		0.060	
	Rep-3	0.330	1.25		0.056	
	Average	0.322	1.23		0.058	
	Std Dev	0.009	0.020		0.0020	
	RSD	2.85%	1.60%		3.45%	
FP Fox						
Wheel 5D Sub-2	Rep-1					17.2
	Rep-2					17.2
	Rep-3					17.3
	Average					17.3
	Std Dev					0.046
	RSD					0.27%
Box 4E Sub-3	Rep-1					2.16
	Rep-2					2.18
	Rep-3					2.22
	Average					2.19
	Std Dev					0.031
	RSD					1.40%
Discrete 5 Sub-3	Rep-1					0.138
	Rep-2					0.152
	Rep-3					0.146
	Average					0.145
	Std Dev					0.0070
	RSD					4.83%
Hand Grenade Range						
Discrete 4,8	Rep-1	0.596	2.62			
	Rep-2	0.596	2.64			
	Rep-3	0.600	2.65			
	Average	0.597	2.64			
	Std Dev	0.0023	0.0153			
	RSD	0.39%	0.58%			

Sample		Concentration (mg/kg)				
Site / Position	Replicate	HMX	RDX	TNT	2,4-DNT	NG
Box 4A Sub-3	Rep-1	0.160	0.304			
	Rep-2	0.170	0.308			
	Rep-3	0.174	0.304			
	Average	0.168	0.305			
	Std Dev	0.0072	0.0023			
	RSD	4.29%	0.76%			
Wheel 2B Sub-1	Rep-1	0.288	2.82			
	Rep-2	0.290	2.80			
	Rep-3	0.298	2.84			
	Average	0.292	2.82			
	Std Dev	0.0053	0.0200			
	RSD	1.81%	0.71%			
FP Juliet Tower						
Discrete 1,4	Rep-1				1.32	0.732
	Rep-2				1.31	0.728
	Rep-3				1.33	0.708
	Average				1.32	0.723
	Std Dev				0.0100	0.0129
	RSD				0.76%	1.78%
Wheel 2B Sub-1	Rep-1				0.322	2.68
	Rep-2				0.328	2.56
	Rep-3				0.322	2.54
	Average				0.324	2.59
	Std Dev				0.0035	0.0757
	RSD				1.07%	2.92%
Box 5E Sub-2	Rep-1				0.276	2.62
	Rep-2				0.270	2.57
	Rep-3				0.270	2.56
	Average				0.272	2.58
	Std Dev				0.0035	0.0321
	RSD				1.27%	1.24%

Table 12 that follows shows results for the subsamples removed from the box, the wheel, and the two discrete samples. In all, there were potentially 37 sets of estimated analyte concentrations for the triplicate subsamples. However, in six cases, one or more of the subsamples failed to contain a concentration above the estimated detection limit. In the most aberrant case (Low-order #3, Wheel 3G), the TNT concentrations determined for the three subsamples ranged over three orders of magnitude (<0.035 to

262 mg/kg). Among the 31 triplicate sets with complete analytical results above the ERL, the RSD ranged from 8.4 to 155% and averaged 70.1%. The median RSD for these complete sets was 61.7%. The possibility of non-detections (e.g., false negatives), and the large and erratic variance among triplicate subsamples establishes a level of uncertainty that is much greater than the target threshold of 10% RSD for laboratory subsampling (See Table 1 or Table 10).

Table 12. Subsample replicates for box, wheel, and discrete designs.

Sample			Concentration (mg/kg)				
Site / Position	Portion	(grams)	HMX	RDX	TNT	2,4-DNT	NG
Low-Order #3							
Wheel 3G	Subsample-1	7.20	96.7	739	262		
	Subsample-2	10.6	7.72	55.8	1.33		
	Subsample-3	9.05	2.10	14.8	<ERL		
	Average		35.5	270			
	Std Dev		53.0	407			
	RSD		**149%**	**151%**			
	Total Sample	**183.8**	**11.1**	**63.3**	**18.7**		
Wheel 4	Subsample-1	10.5	0.076	1.11	<ERL		
	Subsample-2	9.98	0.232	1.44	0.45		
	Subsample-3	9.02	0.133	1.08	<ERL		
	Average		0.147	1.21			
	Std Dev		0.079	0.199			
	RSD		**54%**	**16%**			
	Total Sample	**182.5**	**0.301**	**1.41**	**0.284**		
Box 4E	Subsample-1	8.47	902	7410	2210		
	Subsample-2	8.09	41.8	269	28.7		
	Subsample-3	8.38	289	2130	499		
	Average		411	3270	913		
	Std Dev		443	3710	1150		
	RSD		**108%**	**113%**	**126%**		
	Total Sample	**202.9**	**135**	**1020**	**247**		
Box 3	Subsample-1	8.49	0.115	0.377	0.118		
	Subsample-2	9.46	<ERL	<ERL	<ERL		
	Subsample-3	8.13	0.263	<ERL	<ERL		
	Average						
	Std Dev						
	RSD						
	Total Sample	**214.1**	**0.117**	**0.067**	**<ERL**		

Sample			Concentration (mg/kg)				
Site / Position	Portion	(grams)	HMX	RDX	TNT	2,4-DNT	NG
Demo Range #3							
Wheel 3G	Subsample-1	10.0	1.32	4.40		0.090	
	Subsample-2	10.0	3.24	12.5		0.052	
	Subsample-3	10.0	8.96	49.8		0.274	
	Average		4.51	22.2		0.139	
	Std Dev		3.97	24.2		0.119	
	RSD		**88%**	**109%**		**86%**	
	Total Sample	**90.9**	**3.85**	**14.8**		**0.269**	
Box 1C	Subsample-1	10.0	0.640	0.662		0.022	
	Subsample-2	10.0	0.552	0.462		0.050	
	Subsample-3	10.0	0.496	0.466		0.108	
	Average		0.563	0.530		0.060	
	Std Dev		0.073	0.114		0.044	
	RSD		**13%**	**22%**		**73%**	
	Total Sample	**105**	**1.50**	**1.06**		**0.087**	
Discrete 3	Subsample-1	10.0	0.312	1.212		0.058	
	Subsample-2	10.0	0.212	0.440		0.144	
	Subsample-3	10.0	0.198	0.682		0.114	
	Average		0.241	0.778		0.105	
	Std Dev		0.062	0.395		0.044	
	RSD		**26%**	**51%**		**41%**	
	Total Sample	**77.80**	**0.760**	**2.83**		**0.172**	
FP Fox							
Wheel 5D	Subsample-1	10.0					27.0
	Subsample-2	10.0					17.3
	Subsample-3	10.0					0.240
	Average						14.8
	Std Dev						13.5
	RSD						**91%**
	Total Sample	**68.40**					**9.01**
Box 4E	Subsample-1	10.0					0.342
	Subsample-2	10.0					2.44
	Subsample-3	10.0					2.16
	Average						1.65
	Std Dev						1.14
	RSD						**69%**
	Total Sample	**70.20**					**1.89**

Sample			Concentration (mg/kg)				
Site / Position	Portion	(grams)	HMX	RDX	TNT	2,4-DNT	NG
Discrete 5	Subsample-1	10.0					0.202
	Subsample-2	10.0					0.258
	Subsample-3	10.0					0.138
	Average						0.199
	Std Dev						0.060
	RSD						**30%**
	Total Sample	**99.10**					**0.060**
Hand Grenade Range							
Box 1	Subsample-1	10.0	0.062	0.096			
	Subsample-2	10.0	0.086	0.364			
	Subsample-3	10.0	0.518	2.94			
	Average		0.222	1.13			
	Std Dev		0.257	1.57			
	RSD		**116%**	**139%**			
	Total Sample	**173**	**0.186**	**0.965**			
Wheel 1	Subsample-1	10.0	0.288	2.82			
	Subsample-2	10.0	0.064	0.100			
	Subsample-3	10.0	0.068	0.112			
	Average		0.140	1.01			
	Std Dev		0.128	1.57			
	RSD		**92%**	**155%**			
	Total Sample	**132**	**0.093**	**0.336**			
Box 4A	Subsample-1	10.0	0.116	0.262			
	Subsample-2	10.0	0.118	0.304			
	Subsample-3	10.0	0.160	0.304			
	Average		0.131	0.29			
	Std Dev		0.025	0.02			
	RSD		**19%**	**8.4%**			
	Total Sample	**143**	**0.136**	**0.278**			
Wheel 2C	Subsample-1	10.1	0.071				
	Subsample-2	10.1	0.063				
	Subsample-3	10.0	0.046				
	Average		0.060				
	Std Dev		0.013				
	RSD		**21%**				
	Total Sample	**118**	**0.066**				

Sample			Concentration (mg/kg)				
Site / Position	Portion	(grams)	HMX	RDX	TNT	2,4-DNT	NG
FP Juliet Tower							
Box 5E	Subsample-1	10.0				0.140	4.06
	Subsample-2	10.0				0.276	2.62
	Subsample-3	10.0				0.454	2.80
	Average					0.290	3.160
	Std Dev					0.157	0.785
	RSD					**54%**	**25%**
	Total Sample	**142**				**0.681**	**4.14**
Box 1	Subsample-1	10.0				0.068	0.314
	Subsample-2	10.0				0.186	0.346
	Subsample-3	10.0				0.118	2.50
	Average					0.124	1.05
	Std Dev					0.059	1.25
	RSD					**48%**	**119%**
	Total Sample	**70.6**				**0.125**	**1.03**
Wheel 3	Subsample-1	10.2					0.506
	Subsample-2	10.0					0.720
	Subsample-3	10.1					0.701
	Average						0.642
	Std Dev						0.119
	RSD						**18%**
	Total Sample	**130**				**0.167**	**0.844**
Wheel 2B	Subsample-1	10.0				0.322	2.68
	Subsample-2	10.0				<ERL	3.56
	Subsample-3	10.0				0.068	2.98
	Average						3.07
	Std Dev						0.447
	RSD						**15%**
	Total Sample	**160**				**0.247**	**3.35**

Table 12 also includes concentration estimates obtained for each entire sample (sub-samples and remainder of original sample combined). The ratio of the concentration of each of the subsamples to that of total sample represents the bias from the “true value” (subsample/total sample). In one case (Low-order #3, Box 3), the concentration estimate for the total sample was below the ERL, whereas one of the subsamples from this sample was above this level, thus indicating the potential to obtain a false positive determination. In 102 cases, both values were above the ERLs; in seven cases, one of the values was below the ERLs (Figure 13). Overall, the value determined for the subsample was lower in 67% of the cases than the value for the bulk, indicating a tendency to produce results that are biased low when subsamples are removed prior to processing the entire sample. The explanation for this trend is that 10 g is insufficient mass to address compositional heterogeneity.

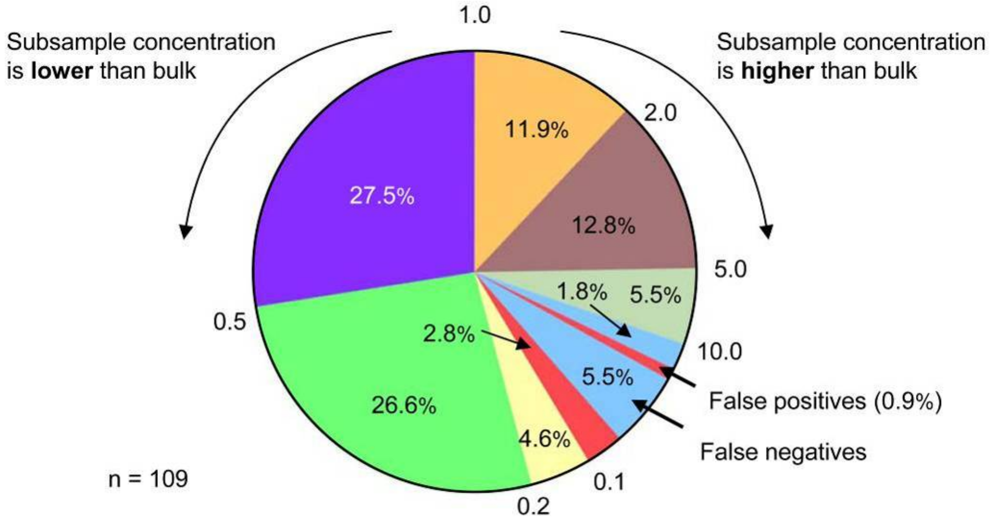

Figure 13. Distribution of the ratios of the subsample concentration to the total sample estimated concentration for 109 samples analyzed.

Table 13 presents analytical results for the triplicate subsamples removed from the processed *MULTI INCREMENT* samples. In this case, analytes were detected in all of the replicates that were collected after the mechanical pulverization of the entire sieved (< 2 mm) fraction of the bulk sample. In total, 27 triplicate sets of estimated analyte concentration were produced from the 10 *MULTI INCREMENT* samples that were subsampled. The range in RSD for these replicates was from 0.32 to 11.8%, the average was 4.16%, and the median RSD was 3.44%. It is clear that in the majority of cases, the uncertainty associated with the subsampling of ground *MULTI INCREMENT* samples is well below a target threshold of 10% RSD (as given in Table 1 and again in Table 10). Therefore, this type of sampling is a vast improvement over the commonly employed “scoop off the top” method.

Table 13. Subsample replicates for *MULTI INCREMENT* sampling processed according to Method 8330B.

Location/Sample	Portion	Concentration (mg/kg)						
		HMX	RDX	TNT	2,4-DNT	2AmDNT	4AmDNT	NG
Low-Order #3								
MI-5	Subsample-1	2.16	12.8	1.11				
	Subsample-2	2.26	13.4	1.19				
	Subsample-3	2.16	12.8	1.14				
	Average	2.19	13.0	1.14				
	Std Dev	0.058	0.364	0.040				
	RSD	**2.63%**	**2.80%**	**3.46%**				
MI-10	Subsample-1	6.64	50.8	25.6		0.080	0.124	
	Subsample-2	6.78	50.6	25.6		0.076	0.114	
	Subsample-3	6.60	50.4	25.4		0.082	0.108	
	Average	6.67	50.6	25.5		0.08	0.12	
	Std Dev	0.095	0.200	0.115		0.003	0.008	
	RSD	**1.42%**	**0.40%**	**0.45%**		**3.85%**	**7.01%**	
Demo Range #3								
MI-5	Subsample-1	1.63	7.20		6.36			
	Subsample-2	1.76	7.40		6.06			
	Subsample-3	1.59	7.00		6.68			
	Average	1.66	7.20		6.37			
	Std Dev	0.088	0.200		0.310			
	RSD	**5.29%**	**2.78%**		**4.87%**			
MI-10	Subsample-1	2.60	11.5	0.064	6.06			
	Subsample-2	2.62	12.2	0.052	5.96			
	Subsample-3	2.54	11.9	0.056	6.44			
	Average	2.59	11.9	0.06	6.15			
	Std Dev	0.042	0.325	0.006	0.253			
	RSD	**1.61%**	**2.74%**	**10.7%**	**4.12%**			
FP Fox								
MI-5	Subsample-1							65.2
	Subsample-2							62.2
	Subsample-3							61.0
	Average							62.8
	Std Dev							2.16
	RSD							**3.44%**
MI-10	Subsample-1							4.82
	Subsample-2							5.04
	Subsample-3							5.12
	Average							4.99

Location/Sample	Portion	Concentration (mg/kg)						
		HMX	RDX	TNT	2,4-DNT	2AmDNT	4AmDNT	NG
	Std Dev							0.155
	RSD							3.11%
Hand Grenade Range								
MI-5	Subsample-1	0.110	0.122					
	Subsample-2	0.104	0.128					
	Subsample-3	0.106	0.152					
	Average	0.107	0.134					
	Std Dev	0.003	0.016					
	RSD	2.86%	11.8%					
MI-10	Subsample-1	0.084	0.180					
	Subsample-2	0.086	0.198					
	Subsample-3	0.086	0.184					
	Average	0.09	0.19					
	Std Dev	0.001	0.009					
	RSD	1.35%	5.05%					
FP Juliet Tower								
MI-5	Subsample-1				0.954			2.52
	Subsample-2				0.776			2.68
	Subsample-3				0.904			2.94
	Average				0.88			2.71
	Std Dev				0.092			0.212
	RSD				10.5%			7.81%
MI-10	Subsample-1				1.13			3.34
	Subsample-2				1.03			3.14
	Subsample-3				1.04			3.04
	Average				1.07			3.17
	Std Dev				0.055			0.153
	RSD				5.16%			4.81%

To demonstrate that the subsamples removed for analysis were representative of the bulk sample, one *MULTI INCREMENT* sample from each sampling area was retrieved from storage. From each bulk sample, triplicate subsamples were collected; then, these subsamples and the remainder of each of bulk *MULTI INCREMENT* sample were extracted with acetone at the same time. A second set of triplicate subsamples were removed because this experiment was performed following 3–5 months of storage at room temperature. Acetone was used as a solvent instead of acetonitrile because of the large solvent volume required. The results of this study are given in Table 14. The relative percent difference (RPD) between the mean for the concentration estimates of the triplicate subsamples and the bulk sample ranged from 1.4–9.1%. These findings show that even though the subsamples removed from the *MULTI INCREMENT* samples for extraction were less than 1% of the total sample mass, they were representative of the concentration of energetic residues in the bulk processed sample.

Table 14. Comparison between bulk sample concentration and average subsample concentration after Method 8330B processing.

Sample		Mass (g)	Acetone (mL)	Concentration (mg/kg)					
Location	Portion			Statistic	HMX	RDX	TNT	2,4-DNT	NG
Demolition Range MI-9	Bulk	1766	3540		2.02	11.9		4.81	
	R1	10.0	20.0		1.98	11.7		4.58	
	R2	10.0	20.0		2.00	11.6		4.92	
	R3	10.0	20.0		1.98	11.8		5.22	
				Ave	1.99	11.7		4.91	
				Std Dev	0.009	0.090		0.320	
				RSD	0.48%	0.77%		6.53%	
				RPD *	1.4%	1.7%		2.1%	
Firing Point Fox MI-10	Bulk	1196	2400						4.21
	R1	10.0	20.0						4.00
	R2	10.0	20.0						5.04
	R3	10.0	20.0						4.06
				Ave					4.37
				Std Dev					0.584
				RSD					13.4%
				RPD					3.7%
Low Order #3 MI-5	Bulk	1278	2560		2.76	14.3	1.56		
	R1	10.0	20.0		2.72	14.1	1.60		
	R2	10.0	20.0		2.72	14.1	1.60		
	R3	10.0	20.0		2.60	13.9	1.63		
				Ave	2.68	14.0	1.61		
				Std Dev	0.035	0.125	0.016		
				RSD	1.26%	0.89%	1.00%		
				RPD	2.9%	2.1%	3.2%		

Sample		Mass (g)	Acetone (mL)	Concentration (mg/kg)					
Location	Portion			Statistic	HMX	RDX	TNT	2,4-DNT	NG
Hand Grenade Range MI5	Bulk	2526	5060			0.645			
	R1	10.0	20.0			0.592			
	R2	10.0	20.0			0.598			
	R3	10.0	20.0			0.576			
				Ave		0.589			
				Std Dev		0.011			
				RSD		1.93%			
				RPD		9.1%			
Firing Point Juliet Tower (MI-5)	Bulk	1935	3880					0.964	2.97
	R1	10.0	20.0					0.88	2.38
	R2	10.0	20.0					1.09	2.84
	R3	10.0	20.0					1.17	3.36
				Ave				1.05	2.86
				Std Dev				0.152	0.490
				RSD				14.5%	17.1%
				RPD				8.5%	3.8%
* RPD Relative percent difference between average and bulk concentration.									

4.3.3 Field sample splitting error

The random error associated with splitting a sample that was collected using the box or wheel sampling design in the field was evaluated by removing several splits from a single sample. This evaluation was performed on randomly selected samples from each of the five sampling areas. For the box and wheel sampling designs, 5 and 7 equal splits were taken, respectively. Each split sample was air-dried, sieved, and the entire < 2-mm fraction was extracted and analyzed. For those box and wheel split samples that had been subsampled, the mass of the analytes in each subsample was combined with the respective split sample to obtain a grand total estimate of the concentration. Of the 21 sets of replicate analyte concentrations available for this evaluation, only one set failed to have concentrations above the estimated reporting limits among all of the splits and for the total sample (Table 15, Wheel #2, Hand grenade range). Therefore, in this case the four splits with concentration estimates below the reporting limits would be considered false negatives. For the remaining 20 complete sets of replicate values, the RSD ranged from 4.7 to 120% and averaged 30.9%. The median RSD for these sets of data was 43.1%.

Although the uncertainty associated with this practice far exceeds the target threshold of 10% for field splitting (Table 1 and again in Table 10), it appears to be less erratic than the practice of removing subsamples prior to totally pulverizing the sample. This is consistent with compositional error, which is directly related to mass; the subsamples often represented less than 10% of the total laboratory sample mass, whereas the splits were between 16 and 20% of the total field sample mass. In addition, there were more replicates among the field sample splits (n=5 or 7) than the laboratory subsamples (n=3), which minimized the impact of a single very high concentration replicate. Overall, the largest RSDs indeed were associated with both the box and wheel splits from Low Order #3 and, in each case, a very high concentration split was the contributing factor.

Table 15. Sample field splits for box and wheel designs.

Sample		Mass (g) < 2mm	Concentration (mg/kg)				
Site / Position	Split		HMX	RDX	TNT	2,4-DNT	NG
Low-Order #3							
Box 4	4A	159	36.6	173	17.4		
	4B	155	29.4	144	13.5		
	4C	176	70.3	471	151		
	4D	140	25.2	153	14.9		
	4E	203	135	1020	247		
	Average		59.4	392	88.9		
	Std Dev		46.0	376	106		
	RSD		**77.5%**	**96.0%**	**120%**		
	Total Sample	**833**	**64.6**	**433**	**101**		
Wheel 3	3A	166	4.39	25.0	9.43		
	3B	184	3.50	15.2	5.02		
	3C	149	10.1	54.1	17.5		
	3D	148	23.6	166	84.8		
	3E	128	18.9	105	25.6		
	3F	200	10.5	51.1	10.4		
	3G	184	11.1	63.3	18.7		
	Average		11.7	68.6	24.5		
	Std Dev		7.30	52.0	27.4		
	RSD		**62.3%**	**75.8%**	**112%**		
	Total Sample	**1159**	**11.2**	**64.7**	**22.8**		

Sample		Mass (g) < 2mm	Concentration (mg/kg)				
Site / Position	Split		HMX	RDX	TNT	2,4-DNT	NG
Demo Range #3							
Box 1	1A	87.5	0.599	0.615		0.158	
	1B	106	1.10	0.981		0.057	
	1C	105	1.50	1.06		0.087	
	1D	114	1.22	1.45		0.056	
	1E	115	1.21	1.45		0.077	
	Average		1.13	1.11		0.087	
	Std Dev		0.331	0.352		0.042	
	RSD		29%	32%		48%	
	Total Sample	527.5	1.15	1.14		0.084	
Wheel 3	3A	98.4	6.99	37.2		0.278	
	3B	105	3.79	12.7		0.238	
	3C	108	6.23	26.1		0.191	
	3D	112	2.49	6.03		0.259	
	3E	106	3.30	12.1		0.211	
	3F	112	4.09	12.1		0.136	
	3G	90.9	3.85	14.8		0.269	
	Average		4.39	17.3		0.226	
	Std Dev		1.62	10.7		0.051	
	RSD		36.8%	61.7%		22.4%	
	Total Sample	732.3	4.36	17.0		0.224	
FP Fox							
Box 4	4A	91.4					3.71
	4B	82.4					3.20
	4C	52.2					2.09
	4D	69.4					1.72
	4E	70.20					1.89
	Average						2.522
	Std Dev						0.882
	RSD						35.0%
	Total Sample	365.6					2.64

Sample		Mass (g) < 2mm	Concentration (mg/kg)				
Site / Position	Split		HMX	RDX	TNT	2,4-DNT	NG
Wheel 5	5A	59.2					6.85
	5B	60.9					1.43
	5C	57.7					1.37
	5D	68.4					9.01
	5E	58.2					0.579
	5F	91.4					3.11
	5G	125					5.16
	Average						3.93
	Std Dev						3.18
	RSD						**80.8%**
	Total Sample	**520.8**					**4.13**
Hand Grenade Range							
Box 4	A	173	0.136	0.278			
	B	145	0.106	0.248			
	C	165	0.168	0.709			
	D	152	0.128	0.426			
	E	162	0.121	0.249			
	Average		0.132	0.382			
	Std Dev		0.023	0.197			
	RSD		**18%**	**52%**			
	Total Sample	**797**	**0.133**	**0.384**			
Wheel 2	A	109	0.044	<ERL			
	B	114	0.076	0.085			
	C	118	0.066	<ERL			
	D	82.0	0.106	<ERL			
	E	88.9	0.069	0.050			
	F	106	0.132	0.045			
	G	86.4	0.156	<ERL			
	Average		0.106				
	Std Dev		0.039				
	RSD		**37%**				
	Total Sample	**704.3**	**0.090**	**0.027**			

Sample		Mass (g) < 2mm	Concentration (mg/kg)				
Site / Position	Split		HMX	RDX	TNT	2,4-DNT	NG
Juliet Tower							
Box 5	A	130				0.961	5.66
	B	114				1.20	7.87
	C	140				0.611	4.70
	D	129				0.574	3.47
	E	142				0.681	4.14
	Average					0.805	5.169
	Std Dev					0.266	1.712
	RSD					**33%**	**33%**
	Total Sample	**655**				**0.790**	**5.08**
Wheel 2	A	155				0.299	3.16
	B	160				0.247	3.35
	C	127				0.224	2.95
	D	141				0.140	3.12
	E	131				0.186	3.31
	F	161				0.125	3.33
	G	141				0.356	3.09
	Average					0.225	3.186
	Std Dev					0.084	0.149
	RSD					**37%**	**4.68%**
	Total Sample	**1016**				**0.225**	**3.19**

Table 15 also contains the concentration estimate obtained for the entire sample (all of the splits combined). The ratio of the concentration of each split sample to the entire field sample represents the bias. Overall, 130 cases had both values above the ERLs (Figure 14). This comparison indicates that the majority (76%) of sample splits contained analyte concentrations that were within a factor of 2.0 of the “bulk concentration estimate” for the sample collected in the field.

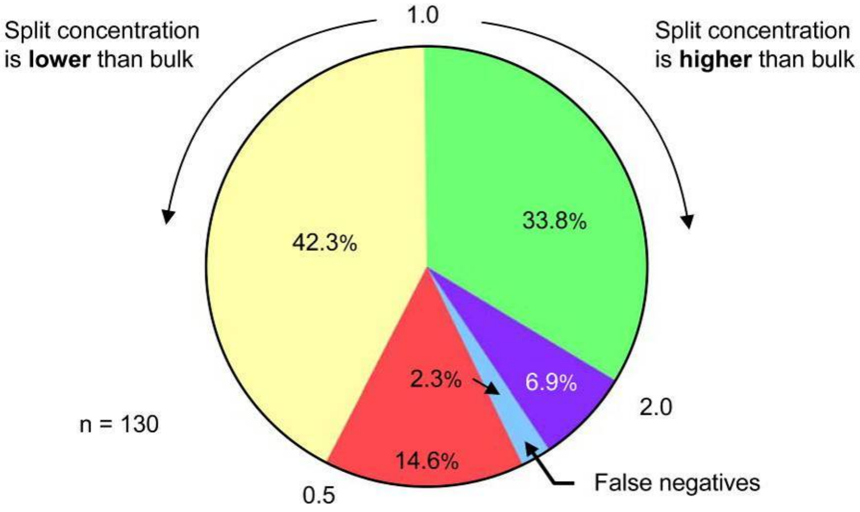

Figure 14. Distribution of the ratios of the sample split concentration to the total sample estimated concentration for 130 samples analyzed.

4.3.4 Uncertainty in the mean and the UCL of the mean for a single population

The 95% UCL of the mean was estimated using the Student's t-test for data sets with three values, and also with ProUCL version 4.0 when more than three values were available (USEPA 1992, 2007). This evaluation only addressed samples that were adequately processed prior to subsampling, or samples extracted in their entirety and therefore not subject to the uncertainty associated with splitting the sample in the field. In addition, only the energetic residues that pass through the #10 sieve (<2 mm) were addressed in this evaluation. Lastly, the variances and the subsequent calculation of the % RSD and the 95% UCL were tabulated for each sampling strategy, independent of establishing a normal distribution of values. Therefore, in some cases these values served only as qualitative inferences of uncertainty.

Firing Point Fox. An area on the north side of FP Fox, measuring 22 m x 36 m, has been sampled annually since 2005 (Area A in Figure 8). This firing point is primarily used for training exercises with 81- and 120-mm mortar rounds; NG has been the only energetic residue detected. Mean concentrations of NG for two separate sampling events of this area were 8.69 and 10.0 mg/kg (Walsh et al. 2007). The area chosen for this study encompassed most of the continuously monitored area and includes some previously unsampled terrain to the southwest (Figure 8).

Within the 40-m × 40-m area chosen for this study, NG was detected in 88% of the 100 randomly located discrete samples (Appendix E, Table E-1). The concentration of this compound ranged over three orders of magnitude, from less than the ERL of 0.065 mg/kg to 196 mg/kg. NG was detected in all of the *MULTI INCREMENT* samples (Appendix E, Table E-2). Table 16 reports the mean, median, variance, and percent RSD values for all the sampling strategies and designs. To estimate the mean and variance for the discrete samples, a value half of the ERL was substituted for the non-detects.

Even though the ratio of the maximum to minimum range for the NG concentration decreased by two orders of magnitude between the *MULTI INCREMENT* and 100 discrete samples, it was much larger than what has been reported for similar studies at this range and at other firing points (Walsh et al. 2005, 2007). Close examination of the NG concentrations estimated for the *MULTI INCREMENT* samples suggests that the sample with the highest concentration could be treated as an outlier (Appendix E, Table E-2).

One possible explanation for this disparate value is that it may have been the only sample that included one or more increments which contained either residues from the burning of excess propellant rings, or unburned propellant grains, or both. Burning of unused propellant rings at this firing point has been observed, and this activity may lead to localized areas with high concentrations due to the incomplete burning of grains (Walsh et al. 2007). The M45 propellant often used on this range consists of 1-mm diameter grains that weigh 4 mg and are 10% NG. The difference between the highest and next highest value among the *MULTI INCREMENT* samples was approximately 50 mg NG/kg, therefore, a concentration of around 5000 mg/kg would be required if this increase was the consequence of a single increment (i.e., 0.8 g of unburned grains within a 15 g sample increment). A study is presently underway to evaluate the residues remaining at locations where there were 10 or 11 M45 propellant rings which were burned on a frozen surface and on snow. The samples have been collected for this exercise and presently are awaiting analysis. At both locations, the surface was covered with hundreds to thousands of unburned propellant grains (Walsh M.R. et al. submitted). This potential point source of residue is much greater than determined for any of the discrete samples collected in this area, the highest of which was 196 mg NG/kg (Appendix E, Table E-1). Dixon's Q-test also supports the contention that this *MULTI INCREMENT* sample includes an aliquot (or a couple of aliquots) from a different population, by showing that the high value could be rejected at the 99% confidence level (USEPA 2007).

Table 16. Summary of sampling results at Firing Point Fox.

Parameter	NG Concentration (mg/kg)		
	Discrete	*MULTI INCREMENT*	
	N = 100	N = 10	N = 9 *
Min	<0.065	3.98	3.98
Max	196	62.8	10.7
Mean	7.29	11.3	5.64
Median	1.77	4.97	4.94
Variance	441	331	4.28
RSD	287%	160%	37%

* Results with outlier value not included. See text.

The combined mass of the NG and the soil processed for each of the 100 discrete samples was 35.6 mg and 6.28 kg, respectively; if treated as a single sample it would have a grand mean concentration of 5.67 mg/kg. This concentration falls among the mean concentrations of the nine *MULTI INCREMENT* samples (range 3.98 to 10.7 mg NG/kg; see Appendix Table E-2), suggesting that the discrete samples and the nine *MULTI INCREMENT* samples were representative of the same population. Taken together, this evidence supports the contention that there is a separate population (location influenced by an activity other than the firing of projectiles) where the concentration of NG is anticipated to be much greater than 200 mg/kg, of which, one increment was included in only one of the 10 *MULTI INCREMENT* samples. Moreover, since 1000 increments and 100 discrete samples were collected within this 40 × 40-m area, the size of this separate population is most likely relatively small. Excluding the *MULTI INCREMENT* sample that appeared to contain one or more increments from a separate population, the maximum value would decrease to 10.7 mg/kg; the mean, median and variance become 5.64, 4.94, and 4.28 mg/kg, respectively.

Table 17 lists ProUCL estimates of the 95% UCLs of the mean derived for the discrete and the *MULTI INCREMENT* sample sets (USEPA 2007). In the case of the 100 discrete samples, values for the non-detects were estimated using lognormal regression on order statistics (ROS). The selection of lognormal ROS was based on the ProUCLs goodness-of-fit evaluation of the discrete sample values above the ERL (USEPA 2007).

To further explore the range of possible 95% UCLs of the mean that could be obtained from these data sets, five replicates composed of 3 and 5 randomly selected *MULTI INCREMENT* samples and groups of 5 and 30 randomly selected discrete samples were evaluated using the Student's t-test and ProUCL (Table 18). Estimating 95% UCLs with the Student's t-test was necessary since ProUCL requires four values. The choice of 3 and 5 samples was based on the number of samples often collected for investigations to represent small areas of concern (< 2000 m^2); the 30-sample amount was based on studies by Jenkins et al. (2005) that showed a tendency for the distribution of means derived from data sets of this size to become more Gaussian. This exercise used a random number generator but did not allow the same sample to be selected twice within a single group; values for the non-detects were estimated with lognormal ROS. Therefore, it should be recognized that this exercise uses a prior knowledge of the distribution obtained from the collection of 100 discrete samples and applies it to the smaller sets.

Table 17. Estimates of the 95% upper confidence limit (UCL) of the mean NG concentration, derived by ProUCL using results of the sampling designs and strategies evaluated at Firing Point Fox.

Sampling design and strategy	95% UCL of the mean – and the parametric or non parametric computational method
100 Discrete – random	16.5 – 95% KM (Chebyshev) UCL*
9 *MULTI INCREMENT* – systematic/random	6.92 – 95% Student's-t UCL†
* nonparametric † parametric	

Table 18. Estimates of the 95% upper confidence limit (UCL) of the mean NG concentration (mg/kg), derived by using randomly selected combinations of results obtained at Firing Point Fox.

Number and type of samples randomly selected	Possible 95% UCL of the mean established by ProUCL or the Student's t-test
5 Discrete samples	9.28, 24.7, 13.7, 21.0* (12.3), 31.0* (28.2)
30 Discrete samples	40.9, 16.3, 10.2, 9.19, 9.50
3 *MULTI INCREMENT* samples**	5.48* (5.22), 12.6* (10.7), 7.62* (7.00), 5.30* (4.94), 7.72* (7.00)
5 *MULTI INCREMENT* samples	8.40, 8.45, 5.08, 8.40, 8.86
* 95% UCL greater than highest value; highest value shown in parentheses. ** Student's t-test, all others 95% UCLs were estimated with ProUCL	

The 95% UCLs of the mean based on 5 or 30 discrete samples were generally higher and much more variable than the individual values for the 100-increment samples and the UCLs of the mean based on 3 or 5 *MULTI INCREMENT* samples. Moreover, for two of five UCL values derived from five discrete samples, the highest value in the data set would be used for estimating the UCL (Mattuck, Blanchet, and Wait 2005). In one case, the highest value represented only half of the ProUCL-predicted 95% UCL of the mean.

Combining the mass of NG with that of the soils processed with the 100 discrete and 9 *MULTI INCREMENT* samples (18.1 kg) established a grand mean concentration of 5.70 mg NG/kg for the area sampled. The UCL derived from the 9 *MULTI INCREMENT* samples (absent the outlier) is the best UCL for the grand mean concentration of the population at this site. Moreover, UCL values derived from 3 or 5 *MULTI INCREMENT* samples were reasonable and varied by less than a factor of 2, which was only slightly better than the individual *MULTI INCREMENT* values. Clearly, a much better and more consistent estimate of the grand mean was obtained from individual values for the 100-increment samples as compared to the UCLs of the mean based on 5, 30, or 100 discrete samples.

Unfortunately, the comparison of data sets obtained with the discrete and the *MULTI INCREMENT* sampling strategies at FP Fox was compromised by the likely presence of a separate population with elevated NG concentrations within the area chosen for sampling. However, with the omission of one *MULTI INCREMENT* sample, the RSD among the remaining values was 37%, a value that approaches the target threshold listed in Table 1 (and again in Table 10).

Demolition Range #3. The area sampled during the earlier 2006 sampling at Demolition Range #3 included a small crater and a 10 × 10-m area surrounding the crater (Figure 9). This area was selected based on finding some pieces of C4 (91% RDX) on the ground surface inside and around the crater. All visible pieces of C4 on the surface were removed prior to sampling. Results showed that mean concentrations of HMX, RDX, and 2,4-DNT outside the crater ranged between 1 and 33 mg/kg; inside the crater, the range was between 28 and 200 mg/kg. RDX was the compound present at the highest concentration in both locations. Low levels (< 1 mg/kg) of TNT, 2,6-DNT, 2AmDNT and 4AmDNT were also present. Another activity performed during this sampling event was collecting three shallow (20-25 cm) profile samples. Visual inspection of two soil cores established the presence of pieces of energetic material beneath the surface.

The 30 × 30-m area selected for the demonstration encompassed the previously sampled location described above. The 100 randomly located discrete samples confirmed the presence of all the previously detected compounds at this range (Appendix E, Table E-3). The distribution was as follows: detectable levels of HMX and RDX in all samples; 2,4-DNT was found in approximately three quarters; the group TNT, 2,6-DNT, 2AmDNT, and 4AmDNT were found in 14% or less; and NG—which previously had not been detected—was found in one discrete sample. HMX, RDX, and 2,4-DNT were detected in all of the *MULTI INCREMENT* samples (Appendix E, Table E-4). Table 19 reports the mean, median, variance, and percent RSD values for HMX, RDX, and 2,4-DNT from the two sampling protocols used at this site. To estimate the mean and variance for 2,4-DNT in the discrete samples, a value equal to one-half the ERL was substituted for the non-detects.

Table 19. Summary of sampling results at Demolition Range #3.
Analyte concentrations are presented in mg/kg.

Statistic	Discrete Samples			*MULTI INCREMENT* Samples		
	HMX	RDX	2,4-DNT	HMX	RDX	2,4-DNT
Min	0.061	0.084	<0.031	1.32	4.66	4.22
Max	31.0	250	136	18.8	126	9.04
Mean	2.45	12.0	8.75	4.80	28.0	6.24
Median	0.925	2.04	0.160	2.77	13.5	6.15
Variance	18.9	930	4.28	26.4	1300	2.16
RSD	178%	255%	227%	107%	129%	24%

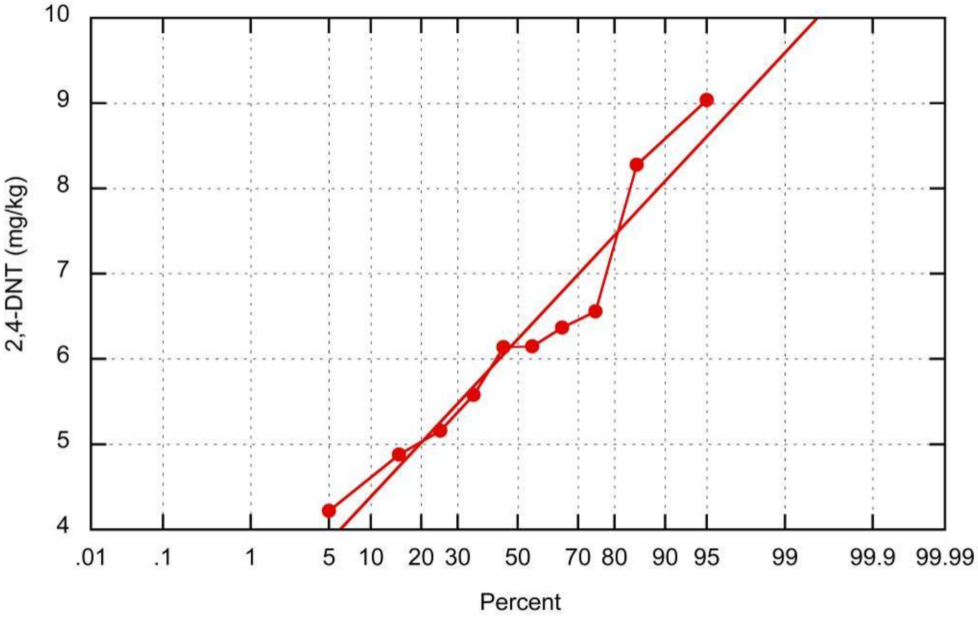

Figure 15. Probability plot of the 2,4-DNT concentration estimates for the 10 *MULTI INCREMENT* samples collected at Demolition Range #3.

Among these data sets, only the 2,4-DNT concentration estimates for the *MULTI INCREMENT* samples were normally distributed (Figure 15). The large range of values for HMX and RDX among the *MULTI INCREMENT* samples was the consequence of one elevated sample, suggesting the presence of a "nugget effect." The nugget effect suggests that small (< 2-mm) C4 pieces were at one or more locations within the chosen sampling area (see Table 19). This explanation is based on the presence of pieces of C4 on and beneath the surface of this range and, as a consequence, the nugget effect often will confound attempts to characterize HMX and RDX on demolition ranges. Because of this confounding variable, this evaluation focused on establishing 95% UCLs as before, only for the mean concentration of 2,4-DNT.

Table 20 lists the ProUCL estimates of the 95% UCL of the mean for 2,4-DNT derived from the discrete and the *MULTI INCREMENT* sampling strategies (USEPA 2007). In the case of the discrete samples, values for the non-detects were estimated using gamma regression on order statistics (ROS). No distribution was established by the ProUCL goodness-of-fit evaluation. The gamma regression was arbitrarily selected for this data set.

To explore the range of 95% UCLs of the mean that could be obtained from this data, five replicates of three and five randomly selected *MULTI*

INCREMENT samples, and groups of 5 and 30 randomly selected discrete samples were evaluated using the Student's t-test and ProUCL (Table 21). The same, previously described logic was used.

Table 20. Estimates of the 95% upper confidence limit (UCL) of the mean 2,4-DNT concentration derived by ProUCL using results of the sampling designs and strategies evaluated at Demolition Range #3.

Sampling design and strategy	95% UCL of the mean – and the parametric or non-parametric computational method
100 Discrete - random	17.4 – 95% KM (Chebyshev) UCL *
10 *MULTI INCREMENT* – systematic/random	7.10 – 95% Student's-t UCL †
* nonparametric; † parametric.	

Table 21. Estimates of the 95% upper confidence limit (UCL) of the mean 2,4-DNT concentration (mg/kg) derived by using randomly selected combinations of results obtained at Demolition Range #3.

Number and type of samples randomly selected	Possible 95% UCL of the mean established by the Student's t-test and ProUCL
5 Discrete samples	†† (0.242), 25.2, †† (28.4), 16.4, 817* (136)
30 Discrete samples	28.5, 10.4, 16.9, 29.2, 19.2
3 *MULTI INCREMENT* samples**	11.6* (9.04), 6.51, 10.2* (9.04), 9.38* (8.28), 6.47
5 *MULTI INCREMENT* samples	7.95, 7.77, 8.52, 7.76, 8.49

* 95% UCL greater than highest value, highest value in parentheses.

** Student's t-test, all others 95% UCLs were estimated with ProUCL

†† At least four values above the detection limits (ERL) are needed to perform ProUCL, highest value in parentheses.

The ProUCLs of the mean based on the 5 and 30 discrete samples were generally higher and much more variable than the individual values for the 100-increment samples and for the 95% UCLs based on 3 and 5 *MULTI INCREMENT* samples. Combining the mass of 2,4-DNT and soils processed among the 100 discrete and 10 *MULTI INCREMENT* samples (28.1 kg) yields an estimated grand mean concentration of 7.05 mg 2,4-DNT/kg for the area sampled. The UCL of the mean based on 10 *MULTI INCREMENT* and random sets of 5 *MULTI INCREMENT* samples were both very reasonable and consistent relative to the grand mean of this population. Indeed, the 95% UCLs based on 3 *MULTI INCREMENT* samples and the values for each of the 100-increment samples resulted in much more reasonable estimates relative to the grand mean concentration for this analyte than those based on 5, 30, or 100 discrete samples.

The nugget effect is likely to be a common problem when attempting to estimate mean concentrations for HMX and RDX on demolition ranges. This feature is prevalent for this type of range as a consequence of using C4 as a donor charge and as part of other training exercises. Another common activity on demolition ranges is the burning and blow-in-place of unused propellant. Frequent detection of 2,4-DNT in samples from this site indicates that one or both of these practices have taken place with single-based propellants. However, distribution of 2,4-DNT does not appear to be the same as distribution for HMX and RDX because it was present in only 75% of the discrete samples. The distribution of 2,4-DNT concentrations among the discrete samples was both skewed and left-censored. Using discrete data with a large number of non-detects also confounds efforts to estimate UCLs, based on statistical inference and not just the arbitrary selection of the highest value from a small set of values. Variance among the *MULTI INCREMENT* samples was below the targeted threshold listed in Table 1 (and again in Table 10) for 2,4-DNT at this sampling location. Very reasonable and reproducible UCLs of the mean concentration of this energetic residue can be obtained based on five or less of these samples.

Impact Range, Low Order #3. During a March 2006 training exercise at Fort Richardson, several 120-mm mortar rounds low-ordered within the central impact range. Around the detonation sampling area designated Low Order #3 (Figure 10), 133 pieces of Composition B (60% RDX, 39% TNT and 1% wax) were scattered over a 378-m^2 area[20]. When sampled previously in August 2006, discrete samples were collected using a nested sampling design as part of a probability study addressing the detection of elevated concentrations (i.e., hot spots). Analytes detected were HMX, RDX, TNT, and the two breakdown products of TNT: 2AmDNT and 4AmDNT. RDX was present in the greatest concentrations, ranging from below detection to about 2000 mg/kg.

The 20 × 20-m area sampled in this demonstration encompassed the location where the discrete samples had been collected and included the

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[20] Personal communication from Marianne Walsh to author Alan Hewitt.

crater formed by the low-order detonation. At this site, we collected 200 discrete samples. Among them, HMX was the most frequently detected compound, at 41% (Appendix E, Table E-5). RDX and TNT were detected in a slightly fewer number of discrete samples. In addition to these compounds, breakdown products of TNT were present in about 15% of the samples, and 2,4-DNT was present in two samples. With the exception of 2,4-DNT, these compounds were also present in the *MULTI INCREMENT* samples (Appendix E, Table E-6). HMX, RDX, and TNT were detected in all the *MULTI INCREMENT* samples.

Since detectable levels of compounds were present in less than half of the discrete samples, only the maximum value is reported; the minimum and median are displayed as ERL (Table 22). For the *MULTI INCREMENT* samples, Table 22 reports the mean, median, variance, and percent RSD values for HMX, RDX, and TNT.

Table 22. Summary of sampling results at Low Order #3.
Analyte concentrations are presented in mg/kg.

Statistic	Discrete Samples			*MULTI INCREMENT* Samples		
	HMX	RDX	TNT	HMX	RDX	TNT
Min	<0.029	<0.038	<0.035	1.76	7.26	0.55
Max	539	4450	1582	10.8	81.8	25.5
Mean				3.90	25.8	8.54
Median	<0.029	<0.038	<0.035	2.69	14.0	2.61
Variance				8.53	576	108
RSD				75%	93%	122%

Because RDX was present in the highest concentration in both data sets, it was selected for evaluating the 95% UCLs for the mean with the Students'-t test and ProUCL. Table 23 lists the estimates of the 95% UCLs of the respective means of RDX derived from the discrete and the *MULTI INCREMENT* sampling strategies (USEPA 1992, 2007). Non-detect values for the discrete samples were estimated using gamma regression on order statistics (ROS). No distribution was established by the ProUCL goodness-of-fit evaluation. The gamma regression was arbitrarily selected for this data set.

To further explore the range of possible 95% UCLs of the mean that could be obtained from these data if fewer samples had been collected, five replicate sets of three and five randomly selected *MULTI INCREMENT* samples, and groups of 30 and 50 discrete samples were evaluated with the Student's t-test and ProUCL (Table 24). Larger numbers of discrete samples were selected in this case because of the large number of non-detects. Aside from the number of discrete samples, this exercise used the same logic as presented previously.

Table 23. Estimates of the 95% upper confidence limit (UCL) of the mean RDX concentration derived by ProUCL, using results of the sampling designs and strategies evaluated at Low Order #3.

Sampling design and strategy	95% UCL of the mean established by ProUCL – and the parametric or non-parametric computational method
200 Discrete – systematic/random	153 – 95% KM (Chebyshev) UCL *
10 *MULTI INCREMENT* – systematic/random	44.2 – 95% Approximate Gamma UCL †
* nonparametric † parametric	

Table 24. Estimates of the 95% upper confidence limit (UCL) of the mean RDX concentration (mg/kg) derived by using randomly selected combinations of results obtained at Low Order #3.

Number and type of samples randomly selected	Possible 95% UCL of the mean established by the Student's t-test and ProUCL
30 Discrete samples	471, 91.5, 84.9, 79.4, 85.2
50 Discrete samples	302, 53.3, 272, 16.9, 33.4
3 *MULTI INCREMENT* samples**	104* (81.8), 62.5* (50.6), 41.7* (33.8), 103* (81.8), 63.2* (50.6)
5 *MULTI INCREMENT* samples	48.9, 42.6, 91.1* (81.8), 25.8, 226* (81.8)
* 95% UCL greater than highest value, highest value in parentheses. ** Student's t-test, all others 95% UCLs were estimated with ProUCL	

The possible 95% UCL of the mean based on 30 and 50 discrete samples, and 3 or 5 *MULTI INCREMENT* samples were erratic. Likewise, there was a large spread in values among the values for the individual 100-increment samples. Combining the mass of RDX and soils processed among the 200 discrete and ten *MULTI INCREMENT* samples (34.2 kg) provides an estimated grand mean concentration of 40.4 mg RDX/kg for the area sampled. The UCLs of the mean derived from the 10 *MULTI INCREMENT* samples yields the only reasonable estimate of upper confidence limit for this population.

These findings emphasize the futility of using discrete samples to characterize a location where extreme spatial heterogeneity is present, and indicates that several *MULTI INCREMENT* samples are needed to achieve a high degree of confidence in the UCL of the mean. However, it should be noted, that a single 100-increment sample provided a mean within a factor of 6 of the grand mean; also, the 95% UCLs based on 3 or 5 *MULTI INCREMENT* samples were within a factor of about two. The 95% UCLs of the mean based on 30 and 50 discrete samples ranged up to about an order of magnitude greater than the grand mean, and the one based on 200 discrete values was greater by a factor of 4.

Hand Grenade Range. Prior to this program, the active hand grenade range at CFB Petawawa was sampled in October 2004. At that time, the range was characterized using two sampling designs. One divided the impact range into several units to assess gradients; the other treated the entire impact range as a single population. Overall, the mean concentrations of HMX, RDX, and TNT were less than 1 mg/kg (Pennington et al. 2006, Chapter 3). Known sources for these energetic residues include Composition B (the explosive charge in the M67 hand grenade) and C4 (used to blow duds in place).

The 20 × 20-m area selected for sampling in this program was in the middle of the impact range, 5 m from the front wall of the grenade castle (Figure 11). HMX and RDX were present in concentrations above their ERLs in 93% of the discrete samples (Appendix E, Table E-7). TNT and its two breakdown products were only detectable in a couple discrete samples. All *MULTI INCREMENT* samples had detectable levels of HMX and RDX; only one had detectable TNT (Appendix E, Table E-8). The mean, median, variance, and percent RSD values for HMX and RDX are reported in Table 25. To estimate the mean and variance for HMX and RDX for the discrete samples a value equal to one-half of the ERL was substituted for the non-detects.

HMX concentrations among the *MULTI INCREMENT* samples appeared to be normally distributed; however, these data should be treated with caution due to the very low concentrations. For this reason, our determination of 95% UCLs only addresses RDX. Table 26 lists the ProUCL estimates of the 95% UCLs of the mean for RDX derived for the discrete and *MULTI INCREMENT* sampling strategies (USEPA 2007). Non-detect values for the discrete samples were estimated using gamma ROS.

No distribution was established by the ProUCL goodness-of-fit evaluation. The gamma regression was arbitrarily selected for this data set.

Table 25. Summary of sampling results at the Hand Grenade Range. Analyte concentrations are presented in mg/kg.

Statistic	Discrete		*MULTI INCREMENT*	
	HMX	RDX	HMX	RDX
Min	<0.029	<0.038	0.089	0.134
Max	1.28	13.7	0.154	0.812
Mean	0.133	0.763	0.122	0.374
Median	0.084	0.171	0.118	0.224
Variance	0.025	2.92	0.0004	0.0538
% RSD	119%	225%	17%	67%

Table 26. Estimates of the 95% upper confidence limit (UCL) of the mean RDX concentration (mg/kg) derived using results of the sampling designs and strategies evaluated at the Hand Grenade Range.

Sampling design and strategy	95% UCL of the mean established by ProUCL – and the parametric or non-parametric computational method.
100 Discrete – systematic/random	1.51 – 95% KM (Chebyshev) UCL *
10 *MULTI INCREMENT* – systematic/random	0.523 – 95% Approximate Gamma UCL †
* nonparametric † parametric	

Table 27. Estimates of the 95% UCL of the mean RDX concentration (mg/kg) derived using randomly selected combinations of results obtained at the Hand Grenade Range.

Number and type of samples randomly selected	Possible 95% UCL of the mean established by the Student's t-test and ProUCL
5 Discrete samples	0.616, 0.709, 1.23, 0.13, 6.47* (5.89)
30 Discrete samples	1.01, 2.87, 1.77, 3.29, 0.695
3 *MULTI INCREMENT* samples**	1.05* (0.812), 0.588* (0.482), 0.218* (0.192), 0.677* (0.554), 0.584* (0.482)
5 *MULTI INCREMENT* samples	0.403, 0.559* (0.554), 0.499, 0.696, 0.482
* 95% UCL greater than highest value, highest value in parentheses. ** Student's t-test, all others – 95% UCLs were estimated with ProUCL	

To further explore the range of possible 95% UCLs of the mean that could be obtained from these data, five replicates of three and five randomly selected *MULTI INCREMENT* and five and 30 randomly selected discrete samples were evaluated with the Student's t-test and ProUCL (Table 27). This exercise used logic as described previously.

The UCLs of the means based on 5 or 30 discrete samples were generally higher and more variable than those based on the 5 *MULTI INCREMENT* samples. In comparison to the UCL intervals based on 3 *MULTI INCREMENT* samples and the individual values for the 100-increment samples, the UCLs based on 5 discrete values were much more variable; those based on 30 discrete values tended to be higher. Combining the mass of RDX and that of the soils processed among the 100 discrete and 10 *MULTI INCREMENT* samples (45.4 kg) yields a grand mean concentration estimate of 0.528 mg RDX/kg for area sampled. The UCL derived from the 10 *MULTI INCREMENT* samples is the best UCL for the grand mean concentration of the population at this site; however; those derived from 5 *MULTI INCREMENT* samples were very reasonable. The UCLs of the mean based on 3 *MULTI INCREMENT* samples, and the individual values obtained for the 100-increment samples were a more reliable estimate of the grand mean than those based on 5, 30, and 100 discrete samples.

FP Juliet Tower. An area in front of a gravel pad was sampled in 2004 at this multi-purpose firing point. The gravel pad and the area in front of it are used mostly for training with vehicle-mounted guns. Overall, mean concentrations of NG and 2,4-DNT in this area of the firing point were 2 and 0.5 mg/kg, respectively (Pennington et al. 2006, Chapter 3).

Within the 30 × 30-m area selected for this program (Figure 12), we observed tracks from vehicles and spent cartridges from small- and medium-caliber guns. NG was detected in all of the discrete samples; 2,4-DNT was detected in 94% (Appendix E, Table E-9). Both energetic residues were detected in all the *MULTI INCREMENT* samples (Appendix E, Table E10). Table 28 reports the mean, median, variance, and percent RSD values for 2,4-DNT and NG for all the sampling strategies and designs. To estimate the mean and variance of 2,4-DNT in the discrete samples, a value equal to one-half of the ERL was substituted for the non-detects.

Among these data sets, the 2,4-DNTand NG concentration estimates for the *MULTI INCREMENT* samples were normally distributed (Figure 16). Table 29 lists the ProUCL estimates for the 95% UCL intervals of the means for 2,4-DNT and NG derived for the discrete and *MULTI INCREMENT* sampling strategies (USEPA 2007). In the case of the discrete samples, the non-detect values for 2,4-DNT were estimated using lognormal ROS. Selecting this method was based on the ProUCL's goodness-of-fit evaluation of discrete sample values above the ERL (ibid).

To further explore the range of possible UCLs of the mean that could be obtained from these data, five replicates of 3 and 5 randomly selected *MULTI INCREMENT* and 5 and 30 randomly selected discrete samples were evaluated using the Student's t-test and ProUCL (Table 30). This exercise used the same logic as described previously.

The ProUCLs of the mean based on 5 and 30 discrete samples were generally higher and much more variable than the individual means of the 100-increment samples and the UCLs based on 5 *MULTI INCREMENT* samples. Combining the mass of 2,4-DNT and NG and that of the soils processed among the 100 discrete and 10 *MULTI INCREMENT* samples (25.8 kg), yielded estimates of the grand mean concentrations of 0.771 and 2.79 mg/kg for 2,4-DNT and NG, respectively, within the area sampled. The UCL of the mean of the 10 *MULTI INCREMENT* and random sets of 3 and 5 *MULTI INCREMENT* samples were both very reasonable and consistent UCLs for this population. Indeed, values for the 100-increment samples were each more reasonable estimators of the grand mean concentrations for 2,4-DNT and NG than most of the UCLs of the mean, based on 5 or 30 discrete samples. Variance among the *MULTI INCREMENT* samples was below the targeted threshold listed in Table 1 (and Table 10) for both 2,4-DNT and NG at this sampling location; very reasonable and reproducible UCL intervals of the mean concentration for these two residues can be obtained based on five or fewer samples.

Table 28. Summary of sampling results at FP Juliet Tower. Analyte concentrations are presented in mg/kg.

	Discrete		*MULTI INCREMENT*	
Statistic	2,4-DNT	NG	2,4-DNT	NG
Min	<0.032	0.109	0.559	2.32
Max	7.23	16.0	1.12	3.25
Mean	0.700	2.79	0.800	2.79
Median	0.253	1.33	0.737	2.82
Variance	1.26	11.0	0.0467	0.104
RSD	160%	119%	27%	12%

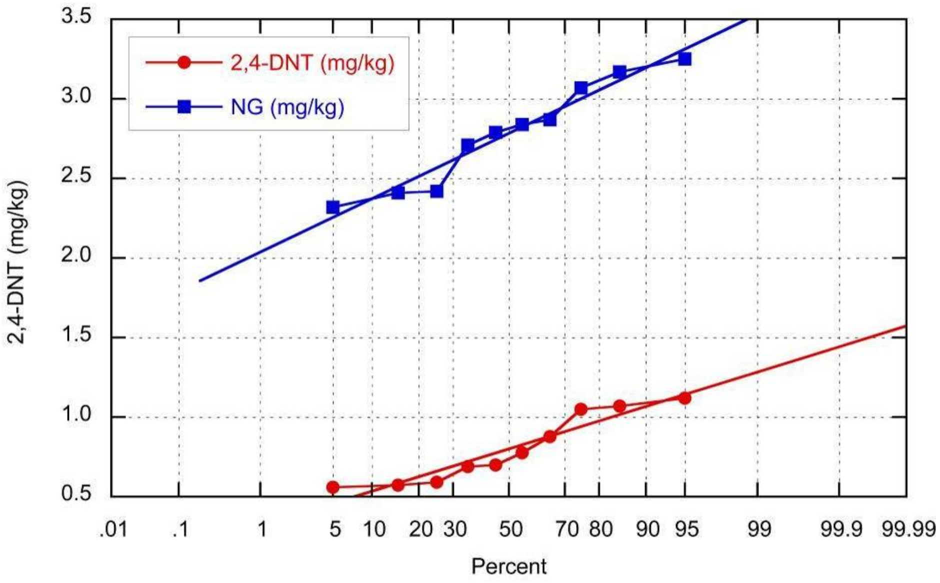


Figure 16. Normal probability plot of estimates of NG and 2,4-DNT in *MULTI INCREMENT* samples at FP Juliet Tower.

Table 29. Estimates of the 95% UCL of the mean NG and 2,4-DNT concentrations derived by ProUCL, using results of the sampling designs and strategies evaluated at FP Juliet Tower.

Sampling design and strategy	95% UCL of the mean established by ProUCL – and the parametric or non-parametric computational method.
100 Discrete – systematic/random	NG 4.24 – 95% Chebyshev (Mean Sd) UCL * 2,4-DNT 1.19 – 95% KM (Chebyshev) UCL *
10 *MULTI INCREMENT* – systematic/random	NG 2.98 – 95% Student's-t UCL † 2,4-DNT 0.926 – 95% Student's-t UCL †
* nonparametric † parametric	

Table 30. Estimates of the 95% UCL of the mean NG and 2,4-DNT concentrations (mg/kg) derived using randomly selected combinations of results obtained at FP Juliet Tower.

Number and type of samples randomly selected	Possible 95% UCL of the mean established by the Student's t-test and ProUCL
5 Discrete samples	NG – 8.37, 15.1* (14.1), 5.37, 9.61, 8.75 2,4-DNT – 2.67, 0.442, 7.27* (4.91), 11.9* (7.23), 3.00
30 Discrete samples	NG – 4.16, 5.97, 3.84, 3.85, 3.14 2,4-DNT – 2.25, 2.07, 1.12, 1.22, 1.51
3 *MULTI INCREMENT* samples**	NG – 3.40* (3.08), 3.43* (3.25), 3.54* (3.25), 3.03* (2.79), 3.72* (3.25) 2,4-DNT – 0.74* (0.70), 1.37 (1.05), 1.29* (1.12), 0.94* (0.88), 1.43* (1.12)
5 *MULTI INCREMENT* samples	NG – 3.18, 3.27* (3.25), 3.10, 3.11, 3.12 2,4-DNT – 0.976, 1.06, 0.903, 1.06, 0.952
* 95% UCL greater than highest value, highest value in parentheses. ** Student's t-test, all others – 95% UCL intervals were estimated with ProUCL	

4.3.5 Removal of vegetation

At FP Fox and FP Juliet Tower, studies were performed to determine if removing vegetative cover prior to sampling could potentially reduce the concentration of energetic residues in the sample. At both sampling areas, *MULTI INCREMENT* samples were collected using the same general sampling designs as used for the other *MULTI INCREMENT* samples. At FP Fox, the vegetative cover was removed prior to obtaining each increment for the five 100-increment samples. The RSD for these replicate field samples was 24%, which is below the targeted total measurement error threshold given in Table 1 (repeated in Table 10), with results shown in Appendix E, Table E-2. The Student's t-test comparison for this set of replicates versus the nine *MULTI INCREMENT* samples collected without removing the vegetative cover (potential outlier excluded) indicated a significant difference between the means at the 90% confidence level (vegetation not removed was higher). At FP Juliet Tower, each individual increment was separated into two portions during the collection of two 100-increment samples. The increments were divided, separating the vegetation from the subsurface soil. Estimated NG concentrations in the subsurface samples were 2.83 mg/kg and 2.91 mg/kg, compared with 4.07 mg/kg and 4.47 mg/kg in the vegetative portion. The 2,4-DNT concentrations were 0.652 mg/kg and 0.882 mg/kg in the subsurface fraction, and 0.611 mg/kg and 0.913 mg/kg in the corresponding portions of the vegetation layer. Collectively, these findings indicate that removing the vegetative cover could cause energetic residues to be lost; as a consequence, results established for surface soils at firing points can be biased low, when collected this way.

4.3.6 Summary

A large amount of uncertainty and bias is associated with the practice of splitting a sample in the field after mixing in a bowl. However, even greater uncertainty and greater bias were observed for the practice of removing only a small portion from a sample container for further processing and subsequent laboratory analysis. Independently or together, the uncertainty associated with these two practices far exceeded the 10% RSD performance criteria listed in Table 1 (repeated in Table 10), rendering the data suspect and scientifically indefensible. Indeed, concentration estimates obtained for samples that were field split and inadequately processed prior to subsampling can only be attributed to the fact that the subsample that was extracted and analyzed did not represent the field sample.

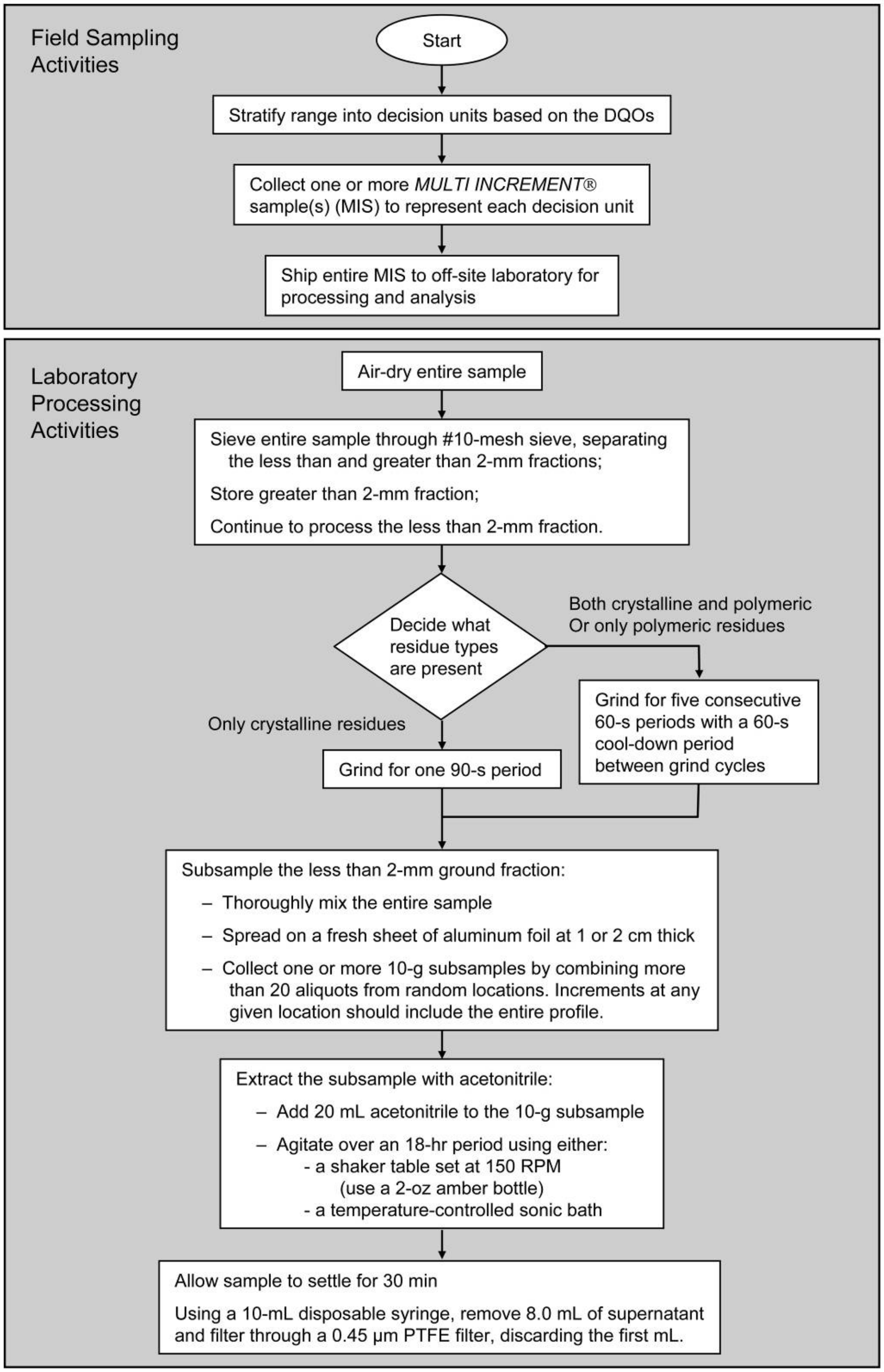
Method 8330B avoids the uncertainty associated with field splitting by recommending that the entire field sample be shipped offsite. To limit laboratory-based subsampling error, this method also recommends: (a) the entire sample be air-dried and sieved,; (b) the < 2-mm fraction be thoroughly pulverized (mechanically ground) and subsequently mixed; and (c) a *MULTI INCREMENT* subsample be collected for extraction and analysis. In the majority of cases, uncertainty was less than 10% RSD among triplicate subsamples removed from soil samples that were over 1200 g in mass. In addition, analyte concentration estimates for triplicate subsamples closely matched (< 10% RPD) the concentrations for the entire sample. These findings can be used to scientifically demonstrate that data generated for the *MULTI INCREMENT* samples indeed are representative of the field sample.

The ability to representatively sample an area of concern remains a formidable challenge for environmental studies. *MULTI INCREMENT* and discrete sampling strategies were evaluated in this study for establishing reliable estimates of mean. To judge which strategy provided the “best estimate” of the average concentration of energetic residues within a chosen area, a grand mean concentration was determined by combining the mass of constituent and soil obtained for all the discrete and *MULTI INCREMENT* samples. This grand mean was based on processing between 18 kg and 34 kg of soil.

The central limit theorem of statistics states that sets of means derived from non-normal populations will approach a normal distribution as the number of values (n) increases. Non-normal distributions of analyte concentrations were found for the discrete sample data sets collected for this study. In general, this phenomenon applies to most constituents distributed as particles. To help normalize data collected to estimate a mean concentration for an area influenced by a military activity, 100-increment samples were collected using a systematic-random sampling design. However, because the ability to collect representative (repeatable) samples depends on both the compositional and distributional heterogeneity of the constituents of concern, no single sampling protocol can assure normality in all cases. For this study a $\leq$ 30% RSD was established as a target for total measurement error. This threshold was obtained for the 100-increment samples collected at two of the five areas sampled. In these cases, the values were normally distributed and reliable 95% UCL intervals of the mean were determined based on only five 100-increment samples, and could have been based on even fewer replicates, since the individual 100-increment means were in all cases close to the grand mean.

In general, ProUCL estimates of the UCLs of the mean based on the 100 discrete samples were higher than: (a) individual 100-increment sample values, and (b) 95% UCLs of the mean based on 3 or 5 *MULTI INCREMENT* samples. Also, these UCLs were often greater than twice the grand mean estimated for the area sampled. The calculation of 95% UCLs of the mean based on 30 randomly selected discrete samples produced a range of values often twice that derived from 5 *MULTI INCREMENT* samples and greater than the variation among the means based on 10 individual 100-increment samples. In all cases, tighter tolerances were obtained for 5 *MULTI INCREMENT* samples as compared to 30 discrete samples. Estimates of the 95% UCL of the mean based on only 5 discrete samples were either unreasonable or unreliable (unrepeatable), or both. This finding is not surprising in that small data sets often have very large variances that result in predictions for UCL intervals that are both invalid and arbitrary (Jenkins et al. 2006, USEPA 2007).

These findings, along with those published during the SERDP ER-1155 program (Pennington et al. 2001-2006), have been used by several agencies to strongly recommend the use of *MULTI INCREMENT* sampling and Method 8330B for characterization activities on military training ranges. Figure 17 is a flow chart that provides an overview of the sampling and sample-handling protocols that result in scientifically defensible data.



Stratify range into decision units based on the DQOs

Collect one or more *MULTI INCREMENT®* sample(s) (MIS) to represent each decision unit

Ship entire MIS to off-site laboratory for processing and analysis

Air-dry entire sample

Sieve entire sample through #10-mesh sieve, separating the less than and greater than 2-mm fractions;

Store greater than 2-mm fraction;

Continue to process the less than 2-mm fraction.

Grind for five consecutive 60-s periods with a 60-s cool-down period between grind cycles

Grind for one 90-s period

Subsample the less than 2-mm ground fraction:

- Thoroughly mix the entire sample
- Spread on a fresh sheet of aluminum foil at 1 or 2 cm thick
- Collect one or more 10-g subsamples by combining more than 20 aliquots from random locations. Increments at any given location should include the entire profile.

Extract the subsample with acetonitrile:

- Add 20 mL acetonitrile to the 10-g subsample
- Agitate over an 18-hr period using either:
  - a shaker table set at 150 RPM (use a 2-oz amber bottle)
  - a temperature-controlled sonic bath

Allow sample to settle for 30 min

Using a 10-mL disposable syringe, remove 8.0 mL of supernatant and filter through a 0.45 µm PTFE filter, discarding the first mL.

Figure 17. Method 8330B field sampling and laboratory sample-processing steps for characterization of energetic residues on military training ranges.

5 Cost Assessment

5.1 Cost reporting

Costs associated with all field-sampling activities include travel, related lodging and meals, labor, and the shipment of samples off site. Unique to the costs associated with sampling activities on military training ranges is the need to acquire the services of EOD personnel or UXO technicians. The major cost differences between the recommended protocols in Method 8330B and those currently in practice appear to arise mainly from the shipping, handling, and processing of larger samples. However, this cost increase is likely to be offset by the need for fewer samples to adequately characterize an area of concern. As previously mentioned, the proposal for this program stated, “The primary focus of this effort is not to provide a less expensive characterization method, but to provide scientifically defensible environmental data.” Such a focus would better guide the stewardship of military training ranges, limiting the amount of costly remediation programs that would be incurred if residues migrate off base.

5.2 Cost analysis

Again, the purpose of this program is to demonstrate acceptable methods to provide scientifically defensible environmental data. To accomplish this goal, the entire sample must be either adequately processed or extracted in its entirety (Sections 4.3.2 and 4.3.3). To estimate a mean concentration or a 95% UCL of the mean concentration of energetic residues within a chosen sampling area, between one and five 100-increment samples or more than 30 discrete samples should be collected, based on the comparisons performed in Section 4.4.4. The expenses associated with gaining site assess, engaging EOD support, travel, and labor are anticipated to be equivalent for these two sampling procedures (Table 31). Given that each discrete sample weighs 150 g and each *MULTI INCREMENT* sample weighs 1500 g, it will likely cost more to ship and dispose of the *MULTI INCREMENT* samples. However, since this item should be based on mass, an estimate of $300 for every 5 kg was used.

The present cost of a Method 8330 analysis is around $150 per sample, including both processing and analysis. It should be recognized that typically this only involves removing 20–30 g from a field sample to perform this task. Indeed, costs may be higher if performing whole sample extraction on a routine basis is required. To dry, sieve, and adequately pulverize each 1500-g *MULTI INCREMENT* sample costs about $150, based on quotes from two separate commercial laboratories. These same laboratories stated that, subsequent to mechanical grinding, the cost of Method 8330B analysis is still about $150. Adding the two costs together means that processing and analysis of each *MULTI INCREMENT* sample costs $300. Another potential increase for the processing of *MULTI INCREMENT* sample, which is still being debated, is the appropriate quality control and assurance measures. Based on guidance issued by the DoD Environmental Data Quality Workshop (U.S. DoD 2008), the QA/QC costs associated with a batch of 20 samples might increase twofold for Method 8330B. This guide currently recommends that, at a minimum, a performance evaluation material should be ground with each batch (20 samples) or for each project. In addition, either duplicate or triplicate subsamples should be taken from processed samples at some pre-determined frequency. For purposes of our calculations, a set fee per batch of $150 (discrete) or $300 (*MULTI INCREMENT*) will be used to estimate the cost of this subcategory. In addition, for calculation purposes, 20 samples make up a batch of discrete samples, and 10 samples make up a batch of *MULTI INCREMENT* samples.

Table 31. Estimated cost comparison between *MULTI INCREMENT* and discrete sampling methods.

Cost Category	Cost Subcategory	Expense Comparison *MULTI INCREMENT* vs. Discrete
Field Sampling	Gaining Site Assess	Same
	EOD Personnel	Same
	Travel and Labor	Same
Samples	Shipment / Disposal	$300/5 kg for both
	Sample Processing/Analysis	$300 (Multi Increment) $150 (discrete)/batch
	Quality Control/Assurance	$300 (Multi Increment) $150 (discrete)/batch

Table 32 presents a cost comparison between expenses for 30 discrete samples and those for one, three, and five *MULTI INCREMENT* samples. The comparison uses only the categories with anticipated differences and puts a minimum cost for shipping/disposal and for QC/QA at $300. This simple analysis shows the potential for a 50% to 80% cost savings when using *MULTI INCREMENT* samples. It should be recognized that savings will be smaller on a project scale, since the total cost associated with field sampling and logistics (i.e., the first three subcategories in Table 31) are likely to be equal to or greater than that those associated with processing and analysis.

Table 32. Cost comparison between discrete samples and various *MULTI INCREMENT* samples.

Procedure	Cost related to sampling strategy			
	30 Discrete	1 *MULTI INCREMENT*	3 *MULTI INCREMENT*	5 *MULTI INCREMENT*
Shipment / Disposal*	$300	$300	$300	$600*
Sample Processing/ Analysis	$4,500	$300	$900	$1,500
Quality Control/ Assurance **	$300	$300	$300	$300
Total	**$5,100**	**$900**	**$1,500**	**$2,400**

* $300 / 5 kg discretes = 150 g; MULTI INCREMENT = 1500 g

** $300 for bath of 10 MUTI INCREMENT samples; $150 for batch of 20 discrete samples.

6 Implementation Issues

6.1 Environmental checklist

N/A.

6.2 Other regulatory issues

Presently, reluctance to adopt Method 8330B is based on a lack of regulatory implementation guidance. To facilitate this process, ERDC-CRREL has an open door policy to providing information and meeting the concerns of DoD agencies. For example, both the request to demonstrate that pulverization of soil would not cause losses of Method 8330 analytes (except tetryl) and the question of whether subsample concentrations were representative of the bulk *MULTI INCREMENT* sample contents were brought to our attention by the Environmental and Munitions Center of Expertise – Omaha (see results in Table 9 and Table 14). We've also supplied the DoD Environmental Data Quality Workgroup with comments on two separate "white papers" that are under development to provide guidance for the implementation of *MULTI INCREMENT* sampling and Method 8330B. Presently, we are engaged in an effort to support the ITRC in developing guidance for the implementation of *MULTI INCREMENT* sampling strategies for hazardous waste investigations. Lastly, we continue to invite representatives from other government agencies to join us in the field when we are performing studies concerning energetic residues on military training ranges.

6.3 End-user issues

We believe that military personnel, federal and state regulators, environmental consultants, and commercial laboratories are all potential end users of this technology. This sampling strategy and sample processing protocol has already been implemented at Hill AFB, MMR, and at Ravenna Army Depot. The states of Alaska and Hawaii have mandated that it be used on several projects, and Region 6 EPA has made strong recommendations for its implementation on MMRP FUDs and other related projects (see EPA letter, Appendix F).

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Acronyms and Annotations

2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
2AmDNT	2-amino-4,6-dinitrotoluene
4AmDNT	4-amino-2,6-dinitrotoluene
C4	91% RDX, 9% oil
CA	Canadian Army
CCV	Continuing Calibration Value
CFB	Canadian Forces Base
Composition B	60% RDX, 39% TNT, 1% wax
CRREL	Cold Regions Research and Engineering Laboratory
CX	Center of Expertise
DLE	Director of Land Environment
DNT	Dinitrotoluene
DoD	U.S. Department of Defense
DQO	Data Quality Objectives
DRDC	Defence Research and Development Canada
EIS	Environmental Impact Statement
EM-CX	Environmental and Munitions Center of Expertise
EOD	Explosive Ordnance Disposal
EPA	Environmental Protection Agency
ER	Environmental Restoration
ERA	Environmental Research Associates
ERDC	Engineering Research and Development Center
ERF	Eagle River Flats
ERL	Estimated Reporting Limit
ESTCP	Environmental Security Technology Certification Program
FATE	Field Analytic Technology Encyclopedia
FP	Firing Point
FUD	Formerly Used Defense
HASP	Health and Safety Plan
HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HTRW	Hazardous, Toxic, and Radioactive Waste
HPLC	High performance liquid chromatography

ITRC	Interstate Technology Regulatory Council
LC-MS	Liquid Chromatography – Mass Spectrometry
LC-MS-MS	Liquid Chromatography – Tandem Mass Spectrometry
LCS	Laboratory Control Spike; Laboratory Control Sample
MDL	Method Detection Limit
MMR	Massachusetts Military Reservation
MMRP	Military Munitions Response Program
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NARPM	National Association of Remedial Project Managers
NEPA	National Environmental Policy Act
NG	Nitroglycerin
OP	Observation Point
ORAP	Operational Range Assessment Program
OSHA	Occupational Safety and Health Administration
OSW	Office of Solid Waste
PE	Performance Evaluation
PETN	Pentaerythritol tetranitrate
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine
ROS	Regression on Order Statistics
RSD	Relative Standard Deviation
RPD	Relative Percent Difference; revolutions per minute
RPM	Remedial Project Manager
SSO	Site Safety Officer
SERDP	Strategic Environmental Research and Development Program
TMC	Troop Medical Clinic
TNT	2,4,6-Trinitrotoluene
UCL	Upper Confidence Limit
USAEC	U.S. Army Environmental Command
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
UXO	Unexploded Ordnance

Appendix A: Analytical Methods Supporting the Experimental Design

U. S. Environmental Protection Agency (2006) Method 8330B: Nitroaromatics, Nitramines, Nitrate Esters by High Performance Liquid Chromatography (HPLC). SW-846. Washington, DC: U. S. Environmental Protection Agency. http://www.epa.gov/osw//hazard/testmethods/pdfs/8330b.pdf

Appendix B: Analytical Methods Supporting the Sampling Plan

U. S. Environmental Protection Agency (2006) Method 8330B: Nitroaromatics, Nitramines, Nitrate Esters by High Performance Liquid Chromatography (HPLC). SW-846. Washington, DC: U. S. Environmental Protection Agency. http://www.epa.gov/osw//hazard/testmethods/pdfs/8330b.pdf

Appendix C: Quality Assurance Project Plan (QAPP)

Purpose and scope of the plan

The objective of this demonstration was to compare concentrations (and the uncertainty associated with them) for different sample collection strategies and sample handling and processing protocols that are used to characterize the concentration of energetic residues on military training and testing facilities. Sampling strategies included discrete, 5- and 7-increment, and *MULTI INCREMENT*. Statistical analyses was used to determine the uncertainty in the final concentration for each of these sampling strategies and their inherent sample handling and processing protocols. In addition, statistical tests assessed if a significant difference existed between the concentrations of samples collected when using the various sampling methods.

Quality assurance responsibilities

With respect to ensuring the integrity of the demonstration and the quality of the samples, Alan Hewitt, Thomas Jenkins, and Marianne Walsh had the following responsibilities.

Alan Hewitt oversaw all aspects of this project including the handling, processing and analysis of the samples and reviewed all data. Thomas Jenkins and Marianne Walsh served as QA Officers.

Data quality parameters

To insure data validity, standard laboratory practices for analyses included the following steps:

- receiving, log-in, and storage of field samples
- chain-of-custody documentation
- standards preparation and analysis
- instrument calibration

- instrumentation QC

Five or more field replicate samples were collected at each sampling location for each sampling strategy. The results from these analyses provided a measure of the precision (repeatability) of the field sampling methods.

Duplicate subsamples for each sampling location were evaluated with spike recoveries. These subsamples were taken for the purposes of preparing Matrix Spikes (MS) and Matrix Spike Duplicates (MSD). These samples helped to identify matrix effects on spiked analytes.

Labels on the sample bags and bottles were marked with water insoluble marker. They were given an identification number, and marked with the time, date, and initials of the sampling individual. All information about each sample was also recorded in a field notebook. Each sample bag or bottle was bagged or double bagged. Samples were stored on ice in a cooler until they were delivered to the analytical laboratory.

When the samples were ready to be sent to ERDC-CRREL for analyses, the cooler was packed with fresh ice. A chain-of-custody form was signed, placed in a zip-lock plastic bag and sealed; the bag was placed inside the cooler; the cooler was sealed with tape and a chain-of-custody seal (in a manner that the seal must be destroyed before accessing the cooler). The cooler was then shipped to the laboratory by overnight express (or equivalent) delivery.

Laboratory control samples and continuing calibration verification standard ensured calibration was maintained. One out of 50 analyses was made a laboratory control sample. One out of 10 analyses was used for continuing calibration verification standards.

Calibration procedures, quality control checks, and corrective action

Approximately 5% of the sample extracts were analyzed in triplicate to give a measure of the precision of the analyses.

The analytical laboratory used standard USEPA protocols for calibrating the analytical instrumentation, including calibration curves with a minimum of five concentrations and the analysis of sample processing blanks.

The data was reviewed by a second analyst to help ensure that all calculations were correct and also to identify any transcription errors.

Demonstration procedures

N/A

Calculation of data quality indicators

The data quality was judged on variation among replicates; therefore, the calculation of the percent relative standard deviation was applied. This was done by initially calculating the mean ($\bar{x}$) and the standard deviation (s) of the replicates with the equations:

$$\bar{x} = \sqrt{\frac{\sum_{i=1}^{n} x_i}{n}}$$

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}}$$

where: x_i = individual sample concentration

n = number of (laboratory or field) sample replicates

Percent relative standard deviation (*% RSD*), a useful statistic for comparing uncertainty between different measurements of varying absolute magnitude, can then be computed as follows:

$$\% \, RSD = \left(\frac{s}{\bar{x}} \right) \text{x}\, 100$$

The absolute or the relative standard deviation can be used to measure the indeterminate error of environmental measurement. To determine the accumulation of indeterminate errors, the relative standard deviation of individual portions are summed:

$$\frac{s_y}{y} = \sqrt{\left(\frac{s_a}{a}\right)^2 + \left(\frac{s_b}{b}\right)^2 + \left(\frac{s_c}{c}\right)^2}$$

where: s_y = absolute standard deviation

y = frequency of occurrence

a,b,c = measurable quantities

In addition, mean values were compared using the Student's t-test, and means for a given set of subsamples, were in some cases compared to the true (method established) concentration for that sample.

Performance and systems audits

N/A

Quality assurance reports

The QA reports contained the results for the continuing calibration verification standards, MS/MSD, and the laboratory control standards.

Data format

All field data was written in a notebook in ink, dated, and signed by the person making the entry.

Laboratory data was delivered in electronic format in a spreadsheet format suitable for easy data analyses (as specified by ERDC-CRREL in the laboratory contract). Any change in automated data entries was not to obscure the original entry. Updated entries also indicated the reason for the change, the date, and the person responsible for the change.

Data storage and archiving procedures

The data will be available as an Appendix in the final report which will be available electronically on the ERDC-CRREL website.

Appendix D: Health and Safety Plan

General

The Health and Safety Plan (HASP) represents the minimum health, safety, and emergency response activities required for Fort Richardson. Compliance with this HASP was mandatory for all personnel involved with this project. The HASP was available for inspection and review by all personnel participating in field sampling activities and also by Fort Richardson representatives. Prior to the initiation of field work, all project personnel read and understood the HASP as well as understood their responsibilities with respect to the HASP. Noncompliance with the HASP would have resulted in removal from active participation in the project.

Hazard assessment

A number of hazards exist at Fort Richardson during field activities. These hazards are primarily due to unexploded ordnance (UXO), possible chemical contamination, and other physical hazards associated with working on a military training facility.

The majority of the volatile chemicals that are of concern following the detonation of munitions are readily diluted and dispersed in the atmosphere. Past studies at the Eagle River Flats (ERF) impact range have failed to detect any toxic or dangerous chemicals other than white phosphorus (WP or P4) and a small amount of 2,4-dinitrotoluene (2,4-DNT). During 2003 – 2008, several smoke projectiles were discovered that, although fully detonated, still contained substantial masses (>200 g) of un-reacted WP that subsequently auto-ignited. WP is present in the mud of hot spots and will react when exposed to air. Other items of concern were low-ordered mortar projectiles. Any large chunks of high explosives associated with them were to be avoided.

A variety of physical hazards were present due to the poor walking surfaces encountered in the field, the use of hand tools, and motor vehicle operation. These hazards were not unique to personnel experienced with outdoor field activities, and were discussed during site-specific training and daily safety briefings.

The probability of encountering unexploded ordnance (UXOs) is moderate, but the consequences certainly warranted special precautions. Whenever encountered, UXO was to be avoided. Known UXO was marked or surface-cleared by the UXO contractor and EOD personnel, reducing the risk of an encounter. There were several 60-mm mortar rounds that have been found in the vicinity of our work areas; because they are painted green, they are difficult to spot, even when watching for them. Although small, they would be more than sufficient in size to kill anyone nearby if they detonated. Deterioration of any UXO in the field makes identification of the type of round difficult. Because of the presence of this UXO, the Army Project Manager and Range Control jointly authorized access to sampled training ranges. (Special precautions regarding UXOs are also further discussed throughout the following sections.)

Access requirements

Civilian personnel involved in activities on the training ranges at Fort Richardson must have completed the 40-hour HAZWOPER course from the Occupational Health and Safety Administration (OSHA) (or the 8-hour annual refresher course) before they were permitted to enter. The required certification must be current. The Army Remedial Project Manager (RPM), Cristal Fosbrook, made the determination that occasional site workers and personnel involved in non-invasive studies at Eagle River Flats (such as those periodically walking previously cleared mortality transects or personnel who only occasionally visit the site) were not required to have the 40-hour HAZWOPER, but must be accompanied by a HAZWOPER-certified individual.

All personnel participating in the sampling activities of this demonstration, whether or not they were required to have 40 hour HAZWOPER, received both a UXO familiarization, safety briefing, and a Range Control safety and protocol briefing before entry. They also had to familiarize themselves with the locations and best routes to the Troop Medical Clinic (TMC) and the emergency entrance to the hospital (at Elmendorf AFB). This HASP had to be read and understood. Individuals could not enter training ranges prior to completing these requirements. Only the Army RPM could waive a specific requirement for civilians. A roster of personnel completing the requirements was compiled by Public Works (Bill Smith) and provided to Range Control. Only personnel on the Range roster were permitted to enter the ranges being sampled. Visitors did not need to be on the Range roster, but did need to be called into and out of the areas being sampled.

Accident prevention

All personnel participating in the Fort Richardson field activities were expected to conduct themselves in a manner that minimized the potential for accidents. All field supervisors and team leaders were responsible for seeing that individuals under their direct supervision were aware of the standard operating procedures for field activities and that they carried out these procedures in a safe manner. A Site Safety Officer (SSO), either the UXO technician or the senior on-site investigator, gave a health and safety briefing prior to initiation of field activities. This meeting addressed all of the material covered in this HASP.

Prior to the start of field activities each day, a short health and safety briefing was conducted by the SSO to address the day's activities. The daily safety briefing served to notify individuals of any deficient areas that needed to be changed or corrected. The briefing emphasized specific concerns associated with the day's planned field activities.

All personnel working on the training ranges at Fort Richardson were to follow the buddy system, meaning that they worked in teams of at least two people and within the same area. All field teams carried standard Fort Richardson trunked radios (in waterproof cases) and had to remain in radio communication with Range Control at all times. In addition, ERDC-CRREL or other frequency radios were employed to reduce usage of the Fort Richardson trunked network. However, these radios are not linked to Range Control and could not be used as a substitute for the trunked radios.

Orange flags and flagging were reserved for use by the UXO tech in marking the location of possible unexploded ordnance. These areas were avoided when operating on Fort Richardson.

The following rules and restrictions were followed by all personnel participating in Fort Richardson field activities:

- The gate at OP Fagan remained locked at all times except when staffed. The lock was locked into a second lock on the two-lock chain to allow access by other entities, including Range, the Fire Department, and

Roads and Grounds. Gate keys were signed out by Bill Smith, Public Works.

- Entry into ranges with restricted access were permitted only when Range Control was manned and only after Range Control clearance had been obtained. All personnel were to be called *in* and called *out*. Team leaders contacted Range Control by radio, giving their last name, number in party, and the last names of all other personnel in the party. In case of duplicate last names, the members' initials were included.
- Detonated and unexploded ordnance was not to be handled under any circumstances by anyone except a member of the 176th EOD detachment or the UXO technician.
- Personnel identified in advance the area within the training ranges at Fort Richardson that required controlled entry. The UXO technician established acceptable routes to and from those identified areas. Personnel traveled on established routes unless they are accompanied by the UXO technician.
- No individuals engaged in field activities without proper authorization. The buddy system, requiring at least two people together at all times and within the same area, was always used for field activities.
- Because of the buddy system rule, if an individual needed to exit the range, the entire party had to exit if dropping a group member would have resulted in only one person left in the field. This did not apply to access and departure from less-restricted areas such as the EOD Pad, and non dud-producing ranges or firing points.
- The number of persons entering the impact area was kept to the minimum number necessary to accomplish the task safely.
- Bright orange safety vests were worn by all personnel. (These can be cloth surveyors' vests.)
- Alcoholic beverages were not allowed to be brought in or consumed on training ranges.
- Spills of hazardous material would be immediately reported to the Fort Richardson Fire Department (384-0774) and contained or cleaned up if safely possible. Any spill would have been reported as soon as possible to Public Works Spill Compliance (384-2711).
- A tag line was required when working in stable gullies if there was water flowing through these gullies. There was no entry allowed into gullies with more than 15 cm (6") of flowing water.
- If illegal hunting (poaching) was occurring on the ERF impact area, all personnel would have been evacuated immediately and Range Control contacted.

- A copy of this document as well as any accident report forms was provided to all personnel and the designated Site Safety Officer (SSO).
- Anyone had authority to cease operations any time procedures would have been violated or threats appeared. The site SSO would then write up the situation or incident for further review. The situation would be investigated by the Project Manager (or his representative), who has authority to allow work to resume when the condition is eliminated.

Accident investigation and reporting

All accidents or injuries (however slight), resulting from field activities, were to be reported to the Army Project Manager or one of her representatives within 24 hours. A determination would be made for further investigation and/or other actions.

Emergency response plan and procedures

The emergency response plan and procedures were instituted prior to any field activities. The emergency response plan and procedures covered the following topics: emergency procedures for field activities, first aid and emergency medical facilities, communications, and site evacuation. As part of the initial safety briefing, field personnel were instructed concerning all aspects of the emergency response plan and procedures. (A list of related phone numbers is provided in Table D-1.)

Table D- 1: Important phone numbers.

Name	Title	Organization	Telephone #
EMERGENCY		Fort Richardson	911
L.D. Fleshman	Range Officer	Fort Richardson	(907) 384-6233
Range Control	Range Dispatch	Fort Richardson	(907) 384-6230
Cristal Fosbrook	USAGAK Army RPM	Fort Richardson	(907) 384-2713
Bill Smith	ERF Logistics	Fort Richardson	(907) 384-3174
Chuck Canterbury	PAO	Fort Richardson	(907) 384-2072
David LaRose	Safety Director	Fort Richardson	(907) 384-2382
USAGAK EOD	EOD Cdr.	Fort Richardson	(907) 384-7600
Troop Medical Clinic	(Non-Emergency)	Fort Richardson	(907) 384-0600
Fire Department		Fort Richardson	(907) 384-0774
Spill Response		Fort Richardson	(907) 384-2711
CW4 Hamilton	AKANG Aviation Safety	Fort Richardson	(907) 384-4334
Search and Rescue		State of Alaska	(800) 478-5555
State Police		State of Alaska	(907) 269-5511
Emergency Services		State of Alaska	(907) 478-2337
Emergency Response		Elmendorf AFB	(907) 552-5555
Medical Hospital		Elmendorf AFB	(907) 552 2748

Emergency procedures

All personnel involved were made aware of the following procedures to be followed, if necessary:

Unexploded Ordnance — Immediate evacuation of the area (300 m radius) and cessation of all operations will be the first response when UXO is discovered by non-UXO personnel. Instructions and directions from the SSO and EOD personnel will be strictly followed.

Accident or Near Accident (Incident) — All accidents or injuries (however slight) that result from field activities must be reported to the Project Manager or their representative within 24 hours. In addition, the following cases require immediate notification (within one hour) by telephone or radio to the Project Manager and installation safety officer:

- fatalities
- lost-time injuries
- spill or exposure to hazardous materials
- theft of hazardous material (including fuel)
- fire or explosion
- property damage in excess of $300 due to an accident/incident
- loss of one day's scheduled activity due to an accident/incident.

Accident Report Information — The following information will be included in the accident report made to the Project Manager and the Installation Safety Officer:

1. Name, title, organization and telephone number of person(s) reporting;

2. Date and time of accident/injury;

3. Location of the accident/injury;

4. Description of the nature of the accident/injury and number of casualties (fatalities, disabling injuries, etc.);

5. Brief summary of the event, giving pertinent details including type of operation ongoing at the time of the accident;

6. Cause of the accident/injury, if known;

7. Details of any existing physical or chemical hazard or contamination;

8. Estimated property damage, if applicable;

9. Nature of the damage and the effect on the work schedule;

10. Action taken by the field supervisor to ensure safety and security;

11. Other damage or injuries sustained (public or private), and written confirmation of telephone reports should be submitted within 24 hours of initial report.

(Note: Form was made available from Project Coordinator and is included at the end of this Appendix D.)

Hospital Required Emergencies - Emergency evacuation in the event of an accident or illness during field activities to be provided by Fort Richardson personnel using their equipment. In cases of serious injury or accident, first contact Range Control on the FRA RANGE channel of your trunked, hand-held radio. If unable to contact Range Control, press the emergency button on top of the radio. On some models, this button is black with the word EMER. On others, it is red with no writing. Pressing this button will cut off all radio transmissions throughout the entire installation except yours on the emergency 911 frequency. Make sure the dispatcher understands clearly the nature of the injury/accident and the exact location. If the situation does not warrant an immediate emergency response, call Range Control and let them know if medical assistance is required and/or your intentions. If medical attention is needed, an ambulance and emergency response personnel will be deployed to the scene. The injured or ill person will be transported to either the Troop Medical Clinic (TMC) or the Elmendorf Air Force Base hospital, whichever is more appropriate. Fort Richardson personnel will make this determination based on their judgment of the situation and knowledge of the services provided at each facility. In less critical cases, it may be more expeditious and convenient for field personnel to transport the ill or injured person directly to the TMC or hospital. No matter what decision is made, Range Control must be informed of the situation.

Prior to entering the training ranges on Fort Richardson, a member of the sampling team shall be required to drive the routes from the study area to the TMC and the hospital on Elmendorf. Phone numbers and names for emergency personnel and facilities are listed in Table D-1.

On Site First Aid Treatment - Emergency first aid shall be applied on site as deemed necessary. At least one individual trained in first aid and CPR will be on-site during all field activities. At least one first aid kit will be readily available in a nearby vehicle that is used to transport field personnel to the study areas. Such equipment will be readily accessible and well marked. First aid kits will include the following:

- Analgesics (Aspirin, Tylenol, etc.)
- Antiseptics and Antihistamines
- Trauma Kit
- Safety pins
- CPR face mask
- Nitrile and latex gloves
- Tweezers; Scissors / Razor Blade
- Blankets
- Container of Clean Water
- Face mask
- Eye shield

It is the responsibility of every field supervisor (team leader) to ensure that his or her personnel are in the appropriate physical and mental condition to participate in fieldwork. It is their responsibility to ensure every team member has the proper safety equipment for their assignment and that the safety equipment is functional and properly fitted. It is also the supervisor's responsibility to be aware of any allergies or sensitivities their personnel may have with regard to medical treatment. It is the responsibility of each team member to notify his or her supervisor of these conditions and to wear the appropriate safety equipment.

Distribution of Safety Plan — A copy of this Safety Plan shall be distributed through the Fort Richardson RPM to all elements on base involved directly in the project, including EOD, Range Control, and Safety. The Alaska District project engineer will be responsible for distribution of this plan to contractors.

ERF Accident / Incident Report

1. Reporting Person

Name ____

Title ____

Telephone #/LID: ______________________________________

Project title: ERF REMEDIATION AND MONITORING PROJECT

P.I.: ____

SSO at the time of incident: ________________________________

Date of filing: ____

2. Details of accident / incident

Date of accident __________________________________

Time of accident __________________________________

Location __________________________________

____

3. Description of the nature of the accident / injury: __________________

____

____

____

4. Cause of the accident / injury, if known: __________________________

____

5. Number of casualties (fatalities, disabling injuries, etc.):_____________

____

____

6. Brief summary of the event, giving pertinent details including type of operation ongoing at the time of the accident: __________________

____

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____

____

7. Details of any existing physical or chemical hazard or contamination:

____

____

8. Estimated property damage, if applicable: ________________________

____

____

9. Nature of the damage and the effect on the work schedule:

____

____

____

10. Action taken by the field supervisor to ensure safety and security:

____

____

____

Other damage or injuries sustained (public or private) and written confirmation of telephone reports should be submitted within 24 hours of initial report to the Remedial Project Manager (Cristal Fosbrook, USARAG –PW).

Appendix E: Analysis Results from Comparative Study

Table E- 1. NG concentration (mg/kg) in discrete samples collected at Fort Richardson (Firing Point Fox).

CRREL #	Position†	Dry wt. (g) <2mm	NG (mg/kg)	CRREL #	Position†	Dry wt. (g) <2mm	NG (mg/kg)
FRA-07-1	0-0	56.7	6.94	FRA-07-34	37-23	70.2	1.46
FRA-07-2	24-5	52.3	18.1	FRA-07-35	1-16	88.2	2.35
FRA-07-3	9-30	43.4	<ERL	FRA-07-36	26-22	40.9	1.12
FRA-07-4	38-22	47.4	0.20	FRA-07-37	23-27	69.2	2.45
FRA-07-5	24-7	26.6	5.93	FRA-07-38	34-8	96.6	18.2
FRA-07-6	15-17	72.8	4.33	FRA-07-39	26-3	33.0	2.58
FRA-07-7	14-8	46.9	3.07	FRA-07-40	24-15	48.2	4.48
FRA-07-8	20-39	109	1.18	FRA-07-41	8-37	42.9	0.11
FRA-07-9	17-31	81.8	1.46	FRA-07-42	27-15	43.2	1.49
FRA-07-10	34-12	88.1	10.2	FRA-07-43	40-28	53.0	1.84
FRA-07-11	23-21	20.5	7.55	FRA-07-44	37-30	88.9	0.17
FRA-07-12	13-20	77.8	0.85	FRA-07-45	32-38	63.7	<ERL
FRA-07-13	39-19	52.4	9.50	FRA-07-46	32-2	64.2	21.9
FRA-07-14	7-0	83.7	1.13	FRA-07-47	24-34	69.3	0.33
FRA-07-15	6-26	29	5.03	FRA-07-48	5-17	63.5	0.32
FRA-07-16	31-10	36.7	10.6	FRA-07-49	23-23	47.3	1.70
FRA-07-17	33-10	24.8	28.2	FRA-07-50	21-4	77.8	4.63
FRA-07-18	9-18	61.4	5.92	FRA-07-51	32-38	97.8	<ERL*
FRA-07-19	37-33	39.3	0.145	FRA-07-52	3-30	88.3	0.76
FRA-07-20	31-34	31.6	196	FRA-07-53	34-35	91.8	<ERL
FRA-07-21	38-37	42.9	0.33	FRA-07-54	17-21	52.2	2.36
FRA-07-22	5-24	35.9	5.48	FRA-07-55	7-32	87.2	0.16
FRA-07-23	21-34	55.6	0.60	FRA-07-56	10-23	82.6	0.77
FRA-07-24	32-5	24.9	60.0	FRA-07-57	4-28	85.3	0.66
FRA-07-25	3-38	28.9	1.52	FRA-07-58	20-33	85.8	0.30
FRA-07-26	2-21	57.5	0.747	FRA-07-59	2-23	55.8	9.95
FRA-07-27	18-17	59.5	0.474	FRA-07-60	2-23	36.3	3.35
FRA-07-28	37-33	52.4	1.25	FRA-07-61	5-32	36.7	0.77
FRA-07-29	22-26	64.9	12.3	FRA-07-62	26-24	65.3	<ERL
FRA-07-30	27-1	69.1	<ERL	FRA-07-63	1-24	46.1	0.70
FRA-07-31	6-5	27.1	18.3	FRA-07-64	4-24	65.5	0.87
FRA-07-32	28-14	43.4	15.4	FRA-07-65	2-29	60.3	<ERL
FRA-07-33	40-30	75.6	<ERL	FRA-07-66	3-15	87.3	7.01

* Less than estimated reporting limit. † position in meters, north from the southern grid boundary - west from the eastern grid boundary

Table E1 (cont.). NG concentration (mg/kg) in discrete samples at FP Fox.

CRREL #	Position†	Dry wt. (g) <2mm	NG (mg/kg)
FRA-07-67	2-33	15.2	<ERL
FRA-07-68	2-35	65.3	0.28
FRA-07-69	22-15	90.4	10.7
FRA-07-70	18-29	80.2	1.46
FRA-07-71	2-30	115	<ERL
FRA-07-72	3-34	35.1	0.70
FRA-07-73	1-21	66.7	18.7
FRA-07-74	14-19	75.1	1.56
FRA-07-75	12-34	64.0	2.85
FRA-07-76	35-26	101	0.25
FRA-07-77	25-8	58.2	15.5
FRA-07-78	17-23	66.9	1.24
FRA-07-79	31-39	83.7	<ERL
FRA-07-80	17-9	27.1	2.83
FRA-07-81	27-24	58.2	0.34
FRA-07-82	35-16	98.3	0.11
FRA-07-83	33-19	68.9	38.0
FRA-07-84	5-11	89.5	7.48
FRA-07-85	28-1	60.4	2.54
FRA-07-86	13-18	74.9	2.11
FRA-07-87	14-18	78.8	2.21
FRA-07-88	27-17	90.8	4.72
FRA-07-89	22-6	54.9	17.2
FRA-07-90	33-37	86.3	<ERL
FRA-07-91	40-4	55.9	12.5
FRA-07-92	24-40	65.9	0.501
FRA-07-93	20-19	81.4	3.50
FRA-07-94	21-7	46.5	6.43
FRA-07-95	23-16	91.3	17.5
FRA-07-96	35-7	71.9	12.3
FRA-07-97	29-6	29.0	14.5
FRA-07-98	24-28	56.0	2.49
FRA-07-99	12-1	73.7	2.75
FRA-07-100	12-2	107	1.16

† position in meters, north from the southern grid boundary - west from the eastern grid boundary

Table E- 2. Concentration (mg/kg) of NG in box, wheel, and MULTI INCREMENT samples collected at Fort Richardson (Firing Point Fox).

Sample Type	CRREL #	Position†	Dry wt. (g) <2mm	NG (mg/kg)
Box	FP Fox Box -#1	6-22	90.3	0.775
	FP Fox Box -#2	20-29	59.1	0.201
	FP Fox Box -#3	28-21	79.9	11.6
	FP Fox Box -#4*	12-19	366	2.64
	FP Fox Box -#5	33-12	73.9	12.6
Wheel	FP Fox Wheel-#1	14-20	157	0.589
	FP Fox Wheel-#2	13-26	84.4	0.900
	Demo Wheel-#3	24-35	68.2	10.1
	FP Fox Wheel-#4	25-18	118	0.407
	FP Fox Wheel-#5*	17-6	520.8	4.13
MULTI INCREMENT With vegetation	FRA07-FP-MI-1	random††	1206	4.54
	FRA07-FP-MI-2	random	1305	4.94
	FRA07-FP-MI-3	random	1368	5.22
	FRA07-FP-MI-4	random	1243	7.00
	FRA07-FP-MI-5A,B,C**	random	1546	62.8 *†
	FRA07-FP-MI-6	random	1261	3.98
	FRA07-FP-MI-7	random	1279	4.44
	FRA07-FP-MI-8	random	1477	10.7
	FRA07-FP-MI-9	random	1344	4.94
	FRA07-FP-MI-10A,B,C	random	1380	4.99
MULTI INCREMENT Vegetation removed	FRA07-FP-MI-VR-1	random	1524	2.38
	FRA07-FP-MI-VR-2	random	1614	3.20
	FRA07-FP-MI-VR-3	random	1375	4.72
	FRA07-FP-MI-VR-4	random	1628	3.76
	FRA07-FP-MI-VR-5A,B,C	random	1519	3.55

* Box or Wheel sample that was split into 5 or 7 equal portions

** Value of subsample triplicates

† position in meters, north from the southern grid boundary - west from the eastern grid boundary

†† random location within each 3 x 3 subgrid

*† potential outlier

Table E- 3. Concentration (mg/kg) of energetic residues in discrete samples collected at Fort Richardson (Demolition Range).

CRREL #	Position†	Dry wt. (g) <2mm	Concentration (mg/kg)							
			HMX	RDX	TNT	2,4-DNT	2,6-DNT	2A-DNT	4A-DNT	NG
FRA-07-1	5-13	128	11.9	84.6	5.20	18.9	< ERL	< ERL	< ERL	< ERL
FRA-07-2	13-0	133	0.886	2.40	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-3	3-5	144	0.900	2.18	< ERL	1.22	< ERL	0.082	< ERL	< ERL
FRA-07-4	2-23	143	0.417	1.20	0.067	74.4	< ERL	< ERL	< ERL	< ERL
FRA-07-5	26-27	82.7	0.163	0.257	< ERL	0.093	< ERL	< ERL	< ERL	< ERL
FRA-07-6	23-0	120	0.460	0.811	< ERL	0.044	< ERL	0.104	0.105	< ERL
FRA-07-7	8-9	112	1.78	4.53	< ERL	0.865	< ERL	< ERL	< ERL	< ERL
FRA-07-8	2-20	98.8	1.07	2.11	< ERL	136	2.23	0.253	0.476	< ERL
FRA-07-9	14-2	97.5	2.09	8.29	< ERL	0.076	< ERL	< ERL	< ERL	0.232
FRA-07-10	14-12	110	6.92	32.8	< ERL	0.048	< ERL	< ERL	< ERL	< ERL
FRA-07-11	11-29	113	0.197	0.30	< ERL	1.65	< ERL	< ERL	< ERL	< ERL
FRA-07-12	27-4	119	0.811	2.76	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-13	5-5	124	0.521	1.02	< ERL	4.37	< ERL	< ERL	< ERL	< ERL
FRA-07-14	7-13	82.2	2.12	10.0	< ERL	14.8	< ERL	< ERL	< ERL	< ERL
FRA-07-15	3-12	80.7	3.81	26.0	20.4	45.8	0.341	0.375	0.521	< ERL
FRA-07-16	28-8	109	4.92	21.6	< ERL	0.042	< ERL	< ERL	< ERL	< ERL
FRA-07-17	24-16	121	1.20	3.37	< ERL	0.199	< ERL	< ERL	< ERL	< ERL
FRA-07-18	6-18	96.6	1.29	7.20	0.056	28.4	0.277	0.104	0.133	< ERL
FRA-07-19	21-25	89.9	0.725	0.559	< ERL	0.070	< ERL	< ERL	< ERL	< ERL
FRA-07-20	8-18	100	0.928	3.48	0.054	20.4	0.174	0.142	0.238	< ERL
FRA-07-21	7-10	95.6	10.2	50.8	< ERL	3.60	< ERL	0.084	< ERL	< ERL
FRA-07-22	21-26	124	1.63	1.63	< ERL	0.066	< ERL	< ERL	< ERL	< ERL
FRA-07-23	17-12	100	0.418	0.626	< ERL	0.04	< ERL	< ERL	< ERL	< ERL
FRA-07-24	13-21	112	0.257	0.350	< ERL	4.99	0.175	< ERL	< ERL	< ERL
FRA-07-25	6-4	116	0.480	0.958	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-26	22-25	68	13.0	78.0	< ERL	0.070	< ERL	< ERL	< ERL	< ERL
FRA-07-27	26-13	73.1	3.27	11.4	< ERL	0.067	< ERL	< ERL	< ERL	< ERL
FRA-07-28	29-17	47.8	0.760	2.83	< ERL	0.172	< ERL	< ERL	< ERL	< ERL
FRA-07-29	0-11	97.7	1.10	4.99	< ERL	30.7	< ERL	< ERL	< ERL	< ERL
FRA-07-30	4-0	117	5.91	37.1	< ERL	2.38	< ERL	< ERL	< ERL	< ERL
FRA-07-31	30-19	114	0.297	0.444	< ERL	0.058	< ERL	< ERL	< ERL	< ERL
FRA-07-32	27-26	89.3	20.2	118	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-33	5-19	119	6.76	33.5	0.058	37.9	0.450	0.081	0.119	< ERL
FRA-07-34	19-10	114	1.33	4.38	< ERL	0.054	< ERL	< ERL	< ERL	< ERL
FRA-07-35	20-27	78.3	0.828	0.550	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-36	19-21	106	2.38	4.17	< ERL	0.242	< ERL	< ERL	< ERL	< ERL
FRA-07-37	15-9	101	1.66	5.31	< ERL	0.133	< ERL	< ERL	< ERL	< ERL
FRA-07-38	5-12	92	0.479	0.984	< ERL	5.89	< ERL	< ERL	< ERL	< ERL
FRA-07-39	18-24	73	0.255	0.545	< ERL	0.067	< ERL	< ERL	< ERL	< ERL

CRREL #	Position†	Dry wt. (g) <2mm	Concentration (mg/kg)							
			HMX	RDX	TNT	2,4-DNT	2,6-DNT	2A-DNT	4A-DNT	NG
FRA-07-40	21-22	104	8.67	31.9	< ERL	0.233	< ERL	< ERL	< ERL	< ERL
FRA-07-41	9-11	110	1.18	3.00	< ERL	0.45	< ERL	< ERL	< ERL	< ERL
FRA-07-42	26-11	110	1.04	1.44	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-43	13-18	78.9	1.24	2.68	0.041	45.0	0.164	< ERL	< ERL	< ERL
FRA-07-44	13-28	74.3	0.143	0.322	< ERL	5.90	< ERL	< ERL	< ERL	< ERL
FRA-07-45	18-29	71.6	0.061	0.149	< ERL	1.07	< ERL	< ERL	< ERL	< ERL
FRA-07-46	11-6	95.4	8.53	52.6	< ERL	0.055	< ERL	< ERL	< ERL	< ERL
FRA-07-47	14-2	113	2.06	10.2	7.44	< ERL	< ERL	0.128	0.150	< ERL
FRA-07-48	1-30	80.3	0.161	0.281	0.062	35.1	0.139	< ERL	< ERL	< ERL
FRA-07-49	7-28	103	0.394	1.59	< ERL	2.70	< ERL	< ERL	< ERL	< ERL
FRA-07-50	9-24	86.1	0.213	0.54	< ERL	8.95	< ERL	< ERL	< ERL	< ERL
FRA-07-51	3-25	104	0.212	0.35	0.044	53.5	0.537	< ERL	< ERL	< ERL
FRA-07-52	7-29	80.8	0.141	0.242	< ERL	3.60	< ERL	< ERL	< ERL	< ERL
FRA-07-53	1-23	93.5	0.229	0.824	< ERL	18.2	0.144	< ERL	< ERL	< ERL
FRA-07-54	9-30	96.6	0.093	0.166	< ERL	6.83	< ERL	< ERL	< ERL	< ERL
FRA-07-55	19-8	104	0.596	4.08	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-56	7-6	85.9	0.997	1.94	< ERL	0.434	< ERL	0.105	< ERL	< ERL
FRA-07-57	7-14	103	2.47	10.1	1.75	15.4	< ERL	< ERL	< ERL	< ERL
FRA-07-58	5-3	113	1.05	2.73	< ERL	0.105	< ERL	0.086	< ERL	< ERL
FRA-07-59	21-3	98.3	0.391	1.08	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-60	20-19	87	6.08	20.0	0.089	0.174	< ERL	0.248	0.302	< ERL
FRA-07-61	16-16	84.9	0.693	0.698	< ERL	0.301	< ERL	< ERL	< ERL	< ERL
FRA-07-62	23-14	133	1.27	3.64	< ERL	0.135	< ERL	< ERL	< ERL	< ERL
FRA-07-63	29-3	137	0.084	0.170	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-64	18-18	98.4	1.27	3.39	< ERL	0.100	< ERL	< ERL	< ERL	< ERL
FRA-07-65	20-2	116	0.718	1.14	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-66	30-17	70.8	0.206	0.415	< ERL	0.042	< ERL	< ERL	< ERL	< ERL
FRA-07-67	26-23	71.1	0.146	0.104	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-68	24-22	109	0.922	1.19	< ERL	0.097	< ERL	< ERL	< ERL	< ERL
FRA-07-69	23-17	100	0.918	1.49	< ERL	0.146	< ERL	< ERL	< ERL	< ERL
FRA-07-70	15-28	90.4	0.327	0.708	< ERL	0.208	< ERL	< ERL	< ERL	< ERL
FRA-07-71	5-26	109	0.188	0.511	< ERL	24.0	0.115	< ERL	< ERL	< ERL
FRA-07-72	9-23	103	2.95	8.74	< ERL	14.9	< ERL	< ERL	< ERL	< ERL
FRA-07-73	30-5	98	0.404	1.08	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-74	27-8	113	0.991	4.26	< ERL	0.053	< ERL	0.086	< ERL	< ERL
FRA-07-75	8-17	118	6.57	25.6	< ERL	6.49	0.071	< ERL	< ERL	< ERL
FRA-07-76	5-15	104	0.781	1.57	< ERL	8.65	< ERL	< ERL	< ERL	< ERL
FRA-07-77	21-11	124	0.267	0.523	< ERL	0.064	< ERL	< ERL	< ERL	< ERL
FRA-07-78	17-28	98.6	0.071	0.085	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-79	8-4	132	0.443	1.85	< ERL	0.047	< ERL	< ERL	< ERL	< ERL

		Dry wt. (g)	Concentration (mg/kg)							
CRREL #	Position†	<2mm	HMX	RDX	TNT	2,4-DNT	2,6-DNT	2A-DNT	4A-DNT	NG
FRA-07-80	13-11	112	0.277	0.766	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-81	17-19	88.4	2.32	13.8	< ERL	0.248	< ERL	< ERL	< ERL	< ERL
FRA-07-82	8-17	116	4.37	11.0	< ERL	8.94	< ERL	< ERL	< ERL	< ERL
FRA-07-83	20-1	115	7.50	41.1	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-84	23-4	104	0.277	0.352	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-85	3-11	88.6	2.99	17.4	1.61	31.3	< ERL	0.120	0.160	< ERL
FRA-07-86	8-12	101	8.10	25.3	< ERL	16.3	< ERL	< ERL	< ERL	< ERL
FRA-07-87	26-16	135	1.17	2.33	< ERL	0.177	< ERL	< ERL	< ERL	< ERL
FRA-07-88	5-21	89.4	31.0	250	< ERL	12.5	0.123	< ERL	< ERL	< ERL
FRA-07-89	9-18	104	4.98	11.9	< ERL	14.4	< ERL	< ERL	< ERL	< ERL
FRA-07-90	5-29	81.6	0.186	0.225	< ERL	24.3	0.235	< ERL	< ERL	< ERL
FRA-07-91	22-25	143	1.53	1.63	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-92	29-9	184	4.32	18.5	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-93	8-29	125	0.062	0.139	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-94	25-22	123	0.427	0.250	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-95	11-22	107	0.378	1.33	< ERL	0.095	< ERL	< ERL	< ERL	< ERL
FRA-07-96	25-7	112	0.526	1.98	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-97	2-12	95.2	2.67	11.2	0.069	79.6	< ERL	0.147	0.151	< ERL
FRA-07-98	17-28	105	0.120	0.099	< ERL	0.055	< ERL	< ERL	< ERL	< ERL
FRA-07-99	30-25	111	1.98	9.04	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL
FRA-07-100	16-3	105	0.080	0.084	< ERL	< ERL	< ERL	< ERL	< ERL	< ERL

† position in meters, west from the eastern grid boundary - south from the northern grid boundary

Table E- 4 Concentration (mg/kg) of energetic residues in box, wheel and *MULTI INCREMENT* samples collected at Fort Richardson, Demolition Range.

			Dry wt. (g)	Concentration (mg/kg)							
Sample Type	CRREL #	Position†	<2mm	HMX	RDX	TNT	2,4-DNT	2,6-DNT	2A-DNT	4A-DNT	NG
Box	Demo Box -#1*	9-17	527.5	1.15	1.14	<ERL	0.084	<ERL	<ERL	<ERL	<ERL
	Demo Box -#2	8-23	104	13.4	76.9	<ERL	30.4	0.198	<ERL	<ERL	<ERL
	Demo Box -#3	16-24	95.4	1.17	2.29	<ERL	26.4	0.224	<ERL	0.096	<ERL
	Demo Box -#4	6-10	129	2.44	10.0	0.907	13.4	0.125	0.085	0.103	<ERL
	Demo Box -#5	10-23	117	3.20	11.2	<ERL	15.1	<ERL	<ERL	<ERL	<ERL
Wheel	Demo Wheel-#1	24-8	89.2	0.634	1.16	<ERL	18.9	0.115	<ERL	<ERL	<ERL
	Demo Wheel-#2	3-18	81.6	1.59	7.06	<ERL	3.02	<ERL	<ERL	<ERL	<ERL
	Demo Wheel-#3*	10-25	732.3	4.36	17.0	<ERL	0.224	<ERL	<ERL	<ERL	<ERL
	Demo Wheel-#4	8-27	116	1.62	4.59	<ERL	72.8	1.16	0.137	0.343	<ERL
	Demo Wheel-#5	18-22	95.5	0.896	1.89	<ERL	0.239	<ERL	<ERL	<ERL	<ERL
MULTI INCREMENT	Demo-MI-1	random††	1650	1.32	4.66	0.078	8.28	<ERL	<ERL	<ERL	<ERL
	Demo-MI-2	random	1730	5.54	27.4	0.128	9.04	0.116	<ERL	<ERL	<ERL
	Demo-MI-3	random	1730	18.8	126	<ERL	4.22	<ERL	<ERL	<ERL	<ERL
	Demo-MI-4	random	1710	2.46	9.44	0.258	5.58	<ERL	<ERL	<ERL	<ERL
	Demo-MI-5A,B,C**	random	1850	1.66	7.20	<ERL	6.37	<ERL	<ERL	<ERL	<ERL
	Demo-MI-6	random	1850	2.48	10.8	<ERL	6.56	<ERL	<ERL	<ERL	<ERL
	Demo-MI-7	random	1830	5.64	32.0	0.574	6.14	<ERL	<ERL	<ERL	<ERL
	Demo-MI-8	random	1820	4.58	35.2	0.042	4.88	<ERL	<ERL	<ERL	<ERL
	Demo-MI-9	random	1860	2.96	15.2	0.556	5.16	<ERL	<ERL	<ERL	<ERL
	Demo-MI-10A,B,C	random	1770	2.59	11.9	0.057	6.15	<ERL	<ERL	<ERL	<ERL

* Box or Wheel sample that was split into 5 or 7 equal portions

** Value of subsample triplicates

† position in meters, west from the eastern grid boundary - south from the northern grid boundary.

†† random location within each 3 x 3 subgrid

Table E-5. Concentration (mg/kg) of energetic residues in discrete samples collected at Fort Richardson, Low-Order #3. (Page 1 of 6)

			Concentration (mg/kg)					
CRREL #	Position*	Dry wt. (g)	HMX	RDX	TNT	2,4-DNT	2A-DNT	4A-DNT
FRA-07-1A	1,1 - 1/1	110	0.662	2.09	<ERL	<ERL	<ERL	<ERL
FRA-07-1B	1,1 - 1.7/0	83.0	3.14	10.6	<ERL	<ERL	<ERL	<ERL
FRA-07-2A	1,2 - 1/1	93.1	1.65	10.1	1.18	<ERL	<ERL	<ERL
FRA-07-2B	1,2 - 1.7/0	83.6	7.96	51.7	9.13	<ERL	0.094	0.105
FRA-07-3A	1,3 - 1/1	95.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-3B	1,3 - 1.7/0	84.6	1.33	9.10	3.37	<ERL	<ERL	<ERL
FRA-07-4A	1,4 - 1/1	69.8	2.25	9.60	0.074	<ERL	<ERL	<ERL
FRA-07-4B	1,4 - 1.7/0	66.6	0.088	0.042	<ERL	<ERL	<ERL	<ERL
FRA-07-5A	1,5 - 1/1	70.4	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-5B	1,5 - 1.7/0	66.1	1.79	10.5	0.290	<ERL	<ERL	<ERL
FRA-07-6A	1,6 - 1/1	76.4	0.061	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-6B	1,6 - 1.7/0	79.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-7A	1,7 - 1/1	78.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-7B	1,7 - 1.7/0	74.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-8A	1,8 - 1/1	74.7	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-8B	1,8 - 1.7/0	59.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-9A	1,9 - 1/1	86.8	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-9B	1,9 - 1.7/0	105	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-10A	1,10 - 1/1	68.3	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-10B	1,10 - 1.7/0	75.2	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-11A	2,1 - 1/1	66.8	31.2	203	101	<ERL	0.344	0.432
FRA-07-11B	2,1 - 1.7/0	90.8	27.0	104	0.852	<ERL	0.169	0.367
FRA-07-12A	2,2 - 1/1	89.8	10.6	25.1	0.509	<ERL	0.070	0.152
FRA-07-12B	2,2 1.7/0	89.8	26.3	164	69.4	<ERL	0.259	0.309
FRA-07-13A	2,3 - 1/1	89.5	1.87	6.92	0.451	<ERL	<ERL	<ERL
FRA-07-13B	2,4 - 1.7/0	103	12.5	95.5	48.3	<ERL	<ERL	<ERL
FRA-07-14A	2,4 - 1/1	77.1	31.3	249	94.6	<ERL	0.461	0.353
FRA-07-14B	2,4 - 1.7/0	107	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-15A	2,5 - 1/1	85.8	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-15B	2,5 - 1.7/0	79.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-16A	2,6 - 1/1	120	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-16B	2,6 - 1.7/0	65.7	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-17A	2,7 - 1/1	74.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-17B	2,7 - 1.7/0	95.6	<ERL	<ERL	0.038	<ERL	<ERL	<ERL

* Moving north from southeast corner, then west for each 2 x 2 m sub grid — sampling location within sub-grid: north from south edge, then west

Table E5 (cont.). Concentration of energetic residues in discrete samples collected at Fort Richardson, Low-Order #3. (Page 2 of 6)

			Concentration (mg/kg)					
CRREL #	Position*	Dry wt. (g)	HMX	RDX	TNT	2,4-DNT	2A-DNT	4A-DNT
FRA-07-18A	2,8 - 1/1	81.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-18B	2,8 - 1.7/0	73.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-19A	2,9 - 1/1	95.3	0.029	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-19B	2,9 - 1.7/0	80	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-20A	2,10 - 1/1	56.4	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-20B	2,10 - 1.7/0	81.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-21A	3,1 0 1/1	85.8	18.8	75.9	16.5	<ERL	0.166	0.222
FRA-07-21B	3,1 1.7/0	109	13.8	67.4	9.89	<ERL	0.121	0.163
FRA-07-22A	3,2 - 1/1	142	45.5	252	32.7	<ERL	0.724	0.992
FRA-07-22B	3,2 - 1.7/0	102	539	4450	1580	<ERL	16.5	10.4
FRA-07-23A	3,3 - 1/1	104	0.360	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-23B	3,3 - 1.7/0	87.4	0.144	0.422	0.039	<ERL	<ERL	<ERL
FRA-07-24A	3,4 - 1/1	85.2	0.450	2.18	<ERL	<ERL	<ERL	<ERL
FRA-07-24B	3,4 - 1.7/0	73.9	0.138	0.294	<ERL	<ERL	<ERL	<ERL
FRA-07-25A	3,5 - 1/1	98.7	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-25B	3,5 - 1.7/0	105	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-26A	3,6 - 1/1	104	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-26B	3,6 - 1.7/0	106	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-27A	3,7 - 1/1	102	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-27B	3,7 - 1.7/0	132	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-28A	3,8 - 1/1	114	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-28B	3,8 - 1.7/0	94.4	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-29A	3,9 - 1/1	121	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-29B	3,9 - 1.7/0	52.3	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-30A	3,10 - 1/1	87.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-30B	3,10 - 1.7/0	86.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-31A	4,1 - 1/1	96	7.63	31.5	0.97	<ERL	0.063	0.140
FRA-07-31B	4,1 - 1.7/0	103	2.23	10.7	1.31	<ERL	<ERL	0.192
FRA-07-32A	4,2 - 1/1	65.7	146	980	247	0.111	3.60	4.90
FRA-07-32B	4,2 - 1.7/0	103	2.80	0.899	0.043	<ERL	<ERL	<ERL
FRA-07-33A	4,3 - 1/1	30.9	11.7	56.3	1.79	<ERL	<ERL	<ERL
FRA-07-33B	4,3 - 1.7/0	83	18.1	92.0	46.7	<ERL	0.096	0.147
FRA-07-34A	4,4 - 1/1	90.6	13.6	97.0	52.8	<ERL	0.163	0.201
FRA-07-34B	4,4 - 1.7/0	112	3.36	17.8	8.05	<ERL	<ERL	<ERL

* Moving north from southeast corner, then west for each 2 x 2 m sub grid — sampling location within sub-grid: north from south edge, then west

Table E5 (cont.). Concentration of energetic residues in discrete samples collected at Fort Richardson, Low-Order #3. (Page 3 of 6)

			Concentration (mg/kg)					
CRREL #	Position*	Dry wt. (g)	HMX	RDX	TNT	2,4-DNT	2A-DNT	4A-DNT
FRA-07-35A	4,5 - 1/1	94.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-35B	4,5 - 1.7/0	90.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-36A	4,6 - 1/1	119	0.123	0.052	<ERL	<ERL	<ERL	<ERL
FRA-07-36B	4,6 - 1.7/0	83.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-37A	4,7 - 1/1	137	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-37B	4,7 - 1.7/0	104	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-38A	4,8 - 1/1	64.3	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-38B	4,8 - 1.7/0	88.2	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-39A	4,9 - 1/1	83.4	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-39B	4,9 - 1.7/0	91.2	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-40A	4,10 - 1/1	78.2	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-40B	4,10 - 1.7/0	89.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-41A	5,1 - 1/1	102	6.71	33.5	1.00	<ERL	0.129	0.188
FRA-07-41B	5,1 - 1.7/0	107	2.82	16.4	1.79	<ERL	<ERL	<ERL
FRA-07-42A	5,2 - 1/1	61.1	0.69	0.475	<ERL	<ERL	<ERL	<ERL
FRA-07-42B	5,2 - 1.7/0	78	41.0	190	8.04	<ERL	0.423	0.658
FRA-07-43A	5,3 - 1/1	89.3	42.5	310	83.0	<ERL	0.639	0.734
FRA-07-43B	5,3 - 1.7/0	86.7	12.4	78.9	16.9	<ERL	0.154	0.193
FRA-07-44A	5,4 - 1/1	115	0.113	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-44B	4,4 - 1.7/0	97.1	0.130	<ERL	0.097	<ERL	<ERL	<ERL
FRA-07-45A	5,5 - 1/1	102	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-45B	5,5 - 1.7/0	108	<ERL	<ERL	0.043	<ERL	<ERL	<ERL
FRA-07-46A	5,6 - 1/1	96.6	0.093	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-46B	5,6 - 1.7/0	94.2	0.055	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-47A	5,7 - 1/1	89	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-47B	5,7 - 1.7/0	75	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-48A	5,8 - 1/1	85.7	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-48B	5,8 - 1.7/0	103	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-49A	5,9 - 1/1	105	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-49B	5,9 - 1.7/0	101	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-50A	5,10 - 1/1	87.5	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-50B	5,10 - 1.7/0	81.3	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-51A	6,1 - 1/1	79.5	11.7	65.2	16.4	<ERL	0.608	0.574
FRA-07-51B	6,1 - 1.7/0	119	10.1	75.2	28.4	<ERL	0.242	0.270

* Moving north from southeast corner, then west for each 2 x 2 m sub grid – sampling location within sub-grid: north from south edge, then west

Table E5 (cont.). Concentration of energetic residues in discrete samples collected at Fort Richardson, Low-Order #3. (Page 4 of 6)

			Concentration (mg/kg)					
CRREL #	Position*	Dry wt. (g)	HMX	RDX	TNT	2,4-DNT	2A-DNT	4A-DNT
FRA-07-52A	6,2 - 1/1	107	18.3	73.2	4.21	<ERL	0.335	0.485
FRA-07-52B	6,2 - 1.7/0	76.3	7.38	43.2	8.70	<ERL	0.199	0.281
FRA-07-53A	6,3 - 1/1	74.0	1.18	0.200	<ERL	<ERL	<ERL	<ERL
FRA-07-53B	6,3 - 1.7/0	93.4	3.87	33.1	8.61	<ERL	0.096	0.108
FRA-07-54A	6,4 - 1/1	120	7.30	54.4	16.2	<ERL	<ERL	<ERL
FRA-07-54B	6,4 - 1.7/0	89.5	3.68	17.8	4.16	<ERL	0.080	<ERL
FRA-07-55A	6,5 - 1/1	85.3	0.099	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-55B	6,5 - 1.7/0	87.3	0.087	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-56A	6,6 - 1/1	107	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-56B	6,6 - 1.7/0	103	0.085	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-57A	6,7 - 1/1	76.5	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-57B	6,7 - 1.7/0	82.7	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-58A	6,8 - 1/1	70.2	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-58B	6,8 - 1.7/0	97.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-59A	6,9 - 1/1	75.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-59B	6,9 - 1.7/0	95.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-60A	6,10 - 1/1	66.8	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-60B	6,10 - 1.7/0	92.2	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-61A	7,1 - 1/1	75.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-61B	7,1 - 1.7/0	83.6	0.633	1.97	<ERL	<ERL	<ERL	<ERL
FRA-07-62A	7,2 - 1/1	96.0	0.194	0.121	<ERL	<ERL	<ERL	<ERL
FRA-07-62B	7,2 - 1.7/0	108	1.76	8.33	0.424	<ERL	<ERL	<ERL
FRA-07-63A	7,3 - 1/1	99.2	0.980	3.95	<ERL	<ERL	<ERL	<ERL
FRA-07-63B	7,3 - 1.7/0	104	99.8	804	408	0.062	1.11	0.888
FRA-07-64A	7,4 - 1/1	95.3	2.31	3.23	<ERL	<ERL	<ERL	<ERL
FRA-07-64B	7,4 - 1.7/0	74.0	0.086	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-65A	7,5 - 1/1	108	0.136	0.562	<ERL	<ERL	<ERL	<ERL
FRA-07-65B	7,5 - 1.7/0	90.8	0.145	0.301	<ERL	<ERL	<ERL	<ERL
FRA-07-66A	7,6 - 1/1	90.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-66B	7,6 - 1.7/0	78.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-67A	7,7 - 1/1	80.3	0.076	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-67B	7,7 - 1.7/0	86.5	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-68A	7,8 - 1/1	86.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-68B	7,8 - 1.7/0	51.3	0.090	<ERL	<ERL	<ERL	<ERL	<ERL

* Moving north from southeast corner, then west for each 2 x 2 m sub grid — sampling location within sub-grid: north from south edge, then west

Table E5 (cont.). Concentration of energetic residues in discrete samples collected at Fort Richardson, Low-Order #3. (Page 5 of 6)

			Concentration (mg/kg)					
CRREL #	Position*	Dry wt. (g)	HMX	RDX	TNT	2,4-DNT	2A-DNT	4A-DNT
FRA-07-69A	7,9 - 1/1	83.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-69B	7,9 - 1.7/0	101	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-70A	7,10 - 1/1	86.8	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-70B	7,10 - 1.7/0	72.8	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-71A	8,1 - 1/1	88.0	0.127	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-71B	8,1 - 1.7/0	100	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-72A	8,2 - 1/1	74.9	0.047	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-72B	8,2 - 1.7/0	104	0.669	1.96	<ERL	<ERL	<ERL	<ERL
FRA-07-73A	8,3 - 1/1	82.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-73B	8,3 - 1.7/0	84.2	0.851	3.67	0.067	<ERL	<ERL	<ERL
FRA-07-74A	8,4 - 1/1	78.2	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-74B	8,4 - 1.7/0	75.6	0.091	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-75A	8,5 - 1/1	96.8	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-75B	8,5 - 1.7/0	98.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-76A	8,6 - 1/1	106	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-76B	8,6 - 1.7/0	111	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-77A	8,7 - 1/1	93.2	0.050	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-77B	8,7 - 1.7/0	78.5	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-78A	8,8 - 1/1	71.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-78B	8,8 - 1.7/0	124	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-79A	8,9 - 1/1	77	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-79B	8,9 - 1.7/0	66.3	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-80A	8,10 - 1/1	107	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-80B	8,10 - 1.7/0	100	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-81A	9,1 - 1/1	132	0.140	0.177	<ERL	<ERL	<ERL	<ERL
FRA-07-81B	9,1 - 1.7/0	75.4	<ERL	0.123	<ERL	<ERL	<ERL	<ERL
FRA-07-82A	9,2 - 1/1	60.3	0.541	1.95	0.111	<ERL	<ERL	<ERL
FRA-07-82B	9,2 - 1.7/0	96.5	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-83A	9,3 - 1/1	86.7	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-83B	9,3 - 1.7/0	91.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-84A	9,4 - 1/1	83.4	91.9	787	468	<ERL	0.72	0.591
FRA-07-84B	9,4 - 1.7/0	53.7	0.084	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-85A	9,5 - 1/1	92.8	46.9	308	37.2	<ERL	0.910	1.28

* Moving north from southeast corner, then west for each 2 x 2 m sub grid — sampling location within sub-grid: north from south edge, then west

Table E5 (cont.). Concentration of energetic residues in discrete samples collected at Fort Richardson, Low-Order #3. (Page 6 of 6)

			Concentration (mg/kg)					
CRREL #	Position*	Dry wt. (g)	HMX	RDX	TNT	2,4-DNT	2A-DNT	4A-DNT
FRA-07-85B	9,5 - 1.7/0	95.2	12.5	52.5	9.00	<ERL	0.097	0.132
FRA-07-86A	9,6 - 1/1	82.9	0.050	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-86B	9,6 - 1.7/0	74.4	0.068	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-87A	9,7 - 1/1	89.0	0.038	0.101	0.176	<ERL	<ERL	<ERL
FRA-07-87B	9,7 - 1.7/0	120	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-88A	9,8 - 1/1	72.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-88B	9,8 - 1.7/0	82.3	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-89A	9,9 - 1/1	100	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-89B	9,9 - 1.7/0	76.7	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-90A	9,10 - 1/1	77.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-90B	9,10 - 1.7/0	73.2	<ERL	0.268	<ERL	<ERL	<ERL	<ERL
FRA-07-91A	10,1 - 1/1	93.4	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-91B	10,1 - 1.7/0	95.2	0.091	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-92A	10,2 - 1/1	96.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-92B	10,2 - 1.7/0	71.2	0.100	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-93A	10,3 - 1/1	63.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-93B	10,3 - 1.7/0	84.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-94A	10,4 - 1/1	73.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-94B	10,4 - 1.7/0	116	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-95A	10,5 - 1/1	62.6	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-95B	10,5 - 1.7/0	83.4	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-96A	10,6 - 1/1	75.5	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-96B	10,6 - 1.7/0	54.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-97A	10,7 - 1/1	77.1	0.193	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-97B	10,7 - 1.7/0	65.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-98A	10,8 - 1/1	81.4	<ERL	0.271	<ERL	<ERL	<ERL	<ERL
FRA-07-98B	10,8 - 1.7/0	68.2	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-99A	10,9 - 1/1	79.9	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-99B	10,9 - 1.7/0	76.1	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-100A	10,10 - 1/1	75.8	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
FRA-07-100B	10,10 - 1.7/0	75.0	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL

* Moving north from southeast corner, then west for each 2 x 2 m sub grid — sampling location within sub-grid: north from south edge, then west

Table E- 5. Concentration (mg/kg) of energetic residues in box, wheel and *MULTI INCREMENT* samples collected at Fort Richardson, Low-Order #3

Sample Type	CRREL #	Position†	Dry wt. (g) <2mm	Concentration (mg/kg)					
				HMX	RDX	TNT	2,4-DNT	2A-DNT	4A-DNT
Box	LO3 Box 1	3,15	185	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
	LO3 Box 2	7,8	173	11.5	91.3	60.5	<ERL	<ERL	<ERL
	LO3 Box 3	11,19	188	0.116	0.060	<ERL	<ERL	<ERL††	<ERL††
	LO3 Box 4*	13,14	833	64.6	433	101	<ERL	<ERL	<ERL
	LO3 Box 5	14,13	215	<ERL	0.066	<ERL	<ERL	<ERL	<ERL
Wheel	LO3 Wheel 1	2,19	176	0.076	0.243	<ERL	<ERL	<ERL	<ERL
	LO3 Wheel 2	5,18	170	<ERL	<ERL	<ERL	<ERL	<ERL	<ERL
	LO3 Wheel 3*	7,5	1159	11.2	64.7	22.8	<ERL	<ERL††	<ERL††
	LO3 Wheel 4	14,1	153	0.331	1.45	0.309	<ERL	<ERL	<ERL
	LO3 Wheel 5	19,9	174	0.634	1.40	<ERL	<ERL	<ERL	<ERL
MULTI INCREMENT	MI-1	1.7 / 0.7	1761	10.8	81.8	25.2	<ERL	0.268	0.354
	MI-2	0.7 / 0.3	1448	1.78	7.26	0.550	<ERL	<ERL	<ERL
	MI-3	1 / 1	1906	3.92	26.2	7.52	<ERL	0.098	0.118
	MI-4	1.3 / 0.7	1385	3.18	15.0	1.85	<ERL	<ERL	<ERL
	MI-5 A,B,C**	0 / 0.7	1933	2.19	13.0	1.14	<ERL	<ERL	<ERL
	MI-6	1 / 1.3	1498	1.76	7.68	0.802	<ERL	<ERL	<ERL
	MI-7	1/ 0	1940	1.78	10.2	0.954	<ERL	<ERL	<ERL
	MI-8	0 / 0.7	1449	2.16	12.7	3.38	<ERL	<ERL	<ERL
	MI-9	1 / 1.7	1829	4.68	33.8	18.5	<ERL	0.140	0.176
	MI-10 A,B,C	0.3 / 0	1453	6.67	50.6	25.5	<ERL	0.079	0.115

* Box or Wheel sample that was split into 5 or 7 equal portions

** Value of subsample triplicates

† Moving north from southeast corner, then west by the designated number of meters for box and wheels sample center points, and within each 2 x 2 m sub-grid for MULTI INCREMENT samples

†† Some of the sample splits contained detectable levels of 2A-DNT and 4A-DNT.

Table E- 6. Concentration (mg/kg) of energetic residues in discrete samples collected at CFB Petawawa, Hand Grenade Range.

CRREL #	Position†	Dry wt. (g) < 2mm	Concentration(mg/kg)		
			HMX	RDX	TNT
07Pet-13	1,1	136	<ERL	<ERL	<ERL
07Pet-14	1,2	163	0.044	0.094	<ERL
07Pet-15	1,3	170	0.048	0.085	<ERL
07Pet-16	1,4	204	0.040	0.125	<ERL
07Pet-17	1,5	217	0.052	0.101	<ERL
07Pet-18	1,6	189	0.075	0.359	<ERL
07Pet-19	1,7	208	0.421	4.97	<ERL
07Pet-20	1,8	202	0.491	5.89	<ERL

CRREL #	Position†	Dry wt. (g) < 2mm	Concentration(mg/kg) HMX	RDX	TNT
07Pet-21	1,9	230	0.120	0.135	<ERL
07Pet-22	1,10	245	1.28	13.7	0.432
07Pet-23	2,1	189	0.030	0.870	<ERL
07Pet-24	2,2	201	0.055	0.179	<ERL
07Pet-25	2,3	229	0.052	0.138	<ERL
07Pet-26	2,4	154	0.056	0.132	<ERL
07Pet-27	2,5	173	0.060	0.111	<ERL
07Pet-28	2,6	146	0.061	0.117	<ERL
07Pet-29	2,7	177	0.056	0.137	<ERL
07Pet-30	2,8	223	0.052	0.123	<ERL
07Pet-31	2,9	144	0.100	0.265	<ERL
07Pet-32	2,10	212	0.142	1.46	<ERL
07Pet-33	3,1	204	<ERL	1.04	<ERL
07Pet-34	3,2	169	0.101	0.655	<ERL
07Pet-35	3,3	177	0.045	0.111	<ERL
07Pet-36	3,4	198	0.045	0.142	<ERL
07Pet-37	3,5	173	0.046	0.147	<ERL
07Pet-38	3,6	164	0.145	0.846	<ERL
07Pet-39	3,7	195	0.095	0.258	<ERL
07Pet-40	3,8	172	0.198	1.41	<ERL
07Pet-41	3,9	190	0.135	0.784	<ERL
07Pet-42	3,10	213	0.100	0.423	<ERL
07Pet-43	4,1	98.4	<ERL	<ERL	<ERL
07Pet-44	4,2	110	0.060	0.062	<ERL
07Pet-45	4,3	114	0.054	0.127	<ERL
07Pet-46	4,4	184	0.091	0.048	<ERL
07Pet-47	4,5	174	0.320	0.670	0.048
07Pet-48	4,6	187	0.105	0.054	<ERL
07Pet-49	4,7	190	0.226	2.02	<ERL
07Pet-50	4,8	198	0.361	1.59	<ERL
07Pet-51	4,9	234	0.087	0.087	<ERL
07Pet-52	4,10	309	0.056	0.172	<ERL
07Pet-53	5,1	161	<ERL	<ERL	<ERL
07Pet-54	5,2	184	0.050	0.056	<ERL
07Pet-55	5,3	77	0.041	<ERL	<ERL
07Pet-56	5,4	102	0.057	0.057	<ERL
07Pet-57	5,5	82.8	0.528	6.42	<ERL
07Pet-58	5,6	119	0.101	0.206	<ERL
07Pet-59	5,7	93.9	0.153	0.253	<ERL
07Pet-60	5,8	119	0.173	0.222	<ERL
07Pet-61	5,9	133	0.065	0.143	<ERL

CRREL #	Position†	Dry wt. (g) < 2mm	Concentration(mg/kg) HMX	RDX	TNT
07Pet-62	5,10	117	0.230	0.509	0.111
07Pet-63	6,1	177	0.038	0.294	<ERL
07Pet-64	6,2	177	0.076	0.203	<ERL
07Pet-65	6,3	162	0.052	0.083	<ERL
07Pet-66	6,4	156	0.097	0.171	<ERL
07Pet-67	6,5	178	0.225	0.481	<ERL
07Pet-68	6,6	163	0.484	1.60	<ERL
07Pet-69	6,7	181	0.180	0.418	<ERL
07Pet-70	6,8	178	0.173	0.922	<ERL
07Pet-71	6,9	198	0.080	0.051	<ERL
07Pet-72	6,10	112	0.116	0.043	<ERL
07Pet-73	7,1	209	0.041	<ERL	<ERL
07Pet-74	7,2	135	0.270	3.00	<ERL
07Pet-75	7,3	140	0.053	0.055	<ERL
07Pet-76	7,4	213	0.174	0.952	<ERL
07Pet-77	7,5	182	0.261	2.11	<ERL
07Pet-78	7,6	227	0.110	0.575	<ERL
07Pet-79	7,7	189	0.117	0.255	<ERL
07Pet-80	7,8	135	0.137	0.176	<ERL
07Pet-81	7,9	174	0.065	0.127	<ERL
07Pet-82	7,10	204	0.081	0.047	<ERL
07Pet-83	8,1	170	<ERL	<ERL	<ERL
07Pet-84	8,2	160	0.038	0.060	<ERL
07Pet-85	8,3	197	0.106	0.546	<ERL
07Pet-86	8,4	145	0.061	0.051	<ERL
07Pet-87	8,5	190	0.072	0.230	<ERL
07Pet-88	8,6	139	0.079	0.086	<ERL
07Pet-89	8,7	186	0.151	0.111	<ERL
07Pet-90	8,8	199	0.093	0.092	<ERL
07Pet-91	8,9	147	0.313	0.475	<ERL
07Pet-92	8,10	172	0.179	1.41	<ERL
07Pet-93	9,1	183	0.286	0.849	0.050
07Pet-94	9,2	216	0.039	0.069	<ERL
07Pet-95	9,3	168	0.051	<ERL	<ERL
07Pet-96	9,4	188	0.153	1.35	<ERL
07Pet-97	9,5	198	0.138	1.10	<ERL
07Pet-98	9,6	216	0.243	2.21	<ERL
07Pet-99	9,7	260	0.110	0.858	<ERL
07Pet-100	9,8	257	0.157	1.35	<ERL
07Pet-101	9,9	236	0.051	0.049	<ERL
07Pet-102	9,10	259	0.033	<ERL	<ERL

CRREL #	Position†	Dry wt. (g) < 2mm	Concentration(mg/kg) HMX	RDX	TNT
07Pet-103	10,1	163	<ERL	<ERL	<ERL
07Pet-104	10,2	201	0.066	0.324	<ERL
07Pet-105	10,3	229	0.074	0.131	<ERL
07Pet-106	10,4	244	0.066	0.087	<ERL
07Pet-107	10,5	237	0.266	2.93	<ERL
07Pet-108	10,6	157	0.060	0.119	<ERL
07Pet-109	10,7	237	0.057	<ERL	<ERL
07Pet-110	10,8	169	0.271	1.62	0.187
07Pet-111	10,9	228	0.112	0.065	<ERL
07Pet-112	10,10	168	0.187	0.876	<ERL

* Moving NW from SE corner, then SW for each 2 x 2 m sub-grid; discrete sample collected in westernmost corner of each 2 x 2 m sub-grid

Table E- 7. Concentration (mg/kg) of energetic residues in box, wheel, and *MULTI INCREMENT* samples collected at CFB Petawawa, Hand Grenade Range.

Sample Type	CRREL #	Position†	Dry wt. (g) <2mm	Conc. (mg/kg) HMX	RDX
Box	HGR Box-1	4,8	143	0.178	0.930
	HGR Box-2	6,12	131	0.098	0.158
	HGR Box-3	12,8	121	0.100	0.132
	HGR Box-4*	8,12	797	0.133	0.384
	HGR Box-5	14,16	112	0.074	0.094
Wheel	HGR Wheel-1	2,12	102	0.080	0.137
	HGR Wheel-2*	10,4	704.3	0.090	0.027
	HGR Wheel-3	2,16	135	0.095	0.155
	HGR Wheel-4	10,16	138	0.133	0.457
	HGR Wheel-5	14,16	113	0.140	0.153
MULTI INCREMENT	MI-1	random	2965	0.154	0.554
	MI-2	random	2876	0.110	0.176
	MI-3	random	2606	0.126	0.812
	MI-4	random	2925	0.107	0.134
	MI-5 A,B,C**	random	3023	0.144	0.138
	MI-6	random	2474	0.106	0.520
	MI-7	random	3069	0.110	0.256
	MI-8	random	2379	0.126	0.180
	MI-9	random	2125	0.089	0.192
	MI-10 A,B,C	random	2886	0.140	0.482

* Box or Wheel sample that was split into 5 or 7 equal portions

** Value of subsample triplicates

† Moving northwest from southeast corner, then west by meters

Table E- 8. Concentration of energetic residues in discrete samples collected at CFB Petawawa, Firing Point Juliet Tower.

CRREL #	Position*	Dry wt. (g) <2mm	Concentration (mg/kg)	
			2,4-DNT	NG
Pet-152	1,1	56.7	4.91	5.23
Pet-153	1,2	63	0.545	1.76
Pet-154	1,3	69.8	0.132	0.883
Pet-155	1,4	55.2	1.44	0.796
Pet-156	1,5	68.4	0.037	0.456
Pet-157	1,6	64.6	0.162	0.327
Pet-158	1,7	64.9	0.043	0.791
Pet-159	1,8	67.1	0.230	0.303
Pet-160	1,9	69.6	0.286	0.109
Pet-161	1,10	59.1	0.039	0.242
Pet-162	2,1	65.93	0.646	3.78
Pet-163	2,2	60.2	0.239	3.15
Pet-164	2,3	65.4	0.137	1.35
Pet-165	2,4	62.3	0.081	1.29
Pet-166	2,5	65.4	2.04	1.00
Pet-167	2,6	71.8	0.078	1.69
Pet-168	2,7	44.1	0.241	8.33
Pet-169	2,8	54.9	0.710	1.66
Pet-170	2,9	51.8	0.201	0.286
Pet-171	2,10	55.9	0.211	0.252
Pet-172	3,1	48.7	0.825	11.4
Pet-173	3,2	63.2	0.372	5.34
Pet-174	3,3	67.6	0.567	5.67
Pet-175	3,4	62.6	0.069	0.880
Pet-176	3,5	69.9	1.77	0.525
Pet-177	3,6	64.4	0.326	2.25
Pet-178	3,7	63.5	0.055	0.675
Pet-179	3,8	54.6	0.538	1.77
Pet-180	3,9	62.2	0.102	0.249
Pet-181	3,10	64.6	<ERL	0.182
Pet-182	4,1	59.4	1.05	10.3
Pet-183	4,2	64.9	2.07	5.86
Pet-184	4,3	59.2	0.683	3.69
Pet-185	4,4	69.7	0.237	1.17
Pet-186	4,5	60.3	0.975	0.925
Pet-187	4,6	61.4	0.199	1.35
Pet-188	4,7	60.3	0.221	1.48

CRREL #	Position*	Dry wt. (g) <2mm	Concentration (mg/kg)	
			2,4-DNT	NG
Pet-189	4,8	61.6	<ERL	1.61
Pet-190	4,9	61.6	0.051	0.732
Pet-191	4,10	59.2	0.067	0.934
Pet-192	5,1	80.1	2.18	6.47
Pet-193	5,2	57.5	0.753	6.64
Pet-194	5,3	65.5	0.207	2.05
Pet-195	5,4	53.3	1.99	1.11
Pet-196	5,5	54.9	0.087	1.44
Pet-197	5,6	62.4	0.444	0.875
Pet-198	5,7	65.9	0.083	1.26
Pet-199	5,8	57.2	0.136	0.493
Pet-200	5,9	60.9	0.183	1.24
Pet-201	5,10	39.6	1.22	0.788
Pet-204	6,1	77.9	0.388	1.22
Pet-204	6,2	67.8	0.900	8.07
Pet-204	6,3	48.9	0.268	6.69
Pet-205	6,4	65.2	0.264	1.57
Pet-206	6,5	53.7	0.171	1.23
Pet-207	6,6	66.8	0.080	2.33
Pet-208	6,7	66.9	<ERL	0.674
Pet-209	6,8	63.3	0.091	1.04
Pet-210	6,9	60.6	0.095	0.550
Pet-211	6,10	40.9	4.87	0.554
Pet-212	7,1	77	2.24	8.42
Pet-213	7,2	71	0.243	11.3
Pet-214	7,3	76.2	0.466	1.51
Pet-215	7,4	56.4	0.100	2.60
Pet-216	7,5	70.2	1.56	1.53
Pet-217	7,6	64.4	0.061	1.94
Pet-218	7,7	64.4	0.145	0.770
Pet-219	7,8	58.5	0.059	0.942
Pet-220	7,9	55.4	0.905	2.64
Pet-221	7,10	60.5	0.438	1.95
Pet-222	8,1	76.4	0.461	3.18
Pet-223	8,2	50.6	7.23	14.1
Pet-224	8,3	67.5	0.203	1.12
Pet-225	8,4	69.2	0.905	2.65
Pet-226	8,5	60.4	0.517	3.04
Pet-227	8,6	59.7	0.050	0.90
Pet-228	8,7	56.3	0.637	1.22
Pet-229	8,8	55.8	<ERL	1.20

CRREL #	Position*	Dry wt. (g) <2mm	Concentration (mg/kg)	
			2,4-DNT	NG
Pet-230	8,9	52.5	0.145	1.31
Pet-231	8,10	53.8	2.73	1.09
Pet-232	9,1	65.2	0.842	6.83
Pet-233	9,2	71.1	2.19	4.41
Pet-234	9,3	68.1	0.446	10.7
Pet-235	9,4	60.4	1.33	8.01
Pet-236	9,5	69.7	0.229	1.60
Pet-237	9,6	58.3	0.058	1.41
Pet-238	9,7	59	0.279	0.919
Pet-239	9,8	57.8	0.596	0.899
Pet-240	9,9	60.6	0.091	0.917
Pet-241	9,10	54.7	0.146	0.850
Pet-242	10,1	60.9	1.06	10.6
Pet-243	10,2	68.5	0.995	16.0
Pet-244	10,3	72	0.410	6.14
Pet-245	10,4	54.2	3.51	4.58
Pet-246	10,5	58.9	0.192	1.54
Pet-247	10,6	68.4	0.911	1.16
Pet-248	10,7	51.1	0.204	0.366
Pet-249	10,8	56.5	0.278	0.678
Pet-250	10,9	61.8	0.078	0.571
Pet-251	10,10	61.9	0.052	0.601

* Moving north from SE corner, then west for each 3 x 3 m sub grid; discrete sample collected in middle of each 3 x 3 m subgrid

Table E- 9. Concentration (mg/kg) of energetic residues in box, wheel, and MULTI INCREMENT samples collected at CFB Petawawa, Firing point Juliet Tower.

Sample Type	CRREL #	Position†	Dry wt. (g) <2mm	Concentration (mg/kg)	
				2,4-DNT	NG
Box	FP JT Box-1	6,27	40.6	0.126	1.02
	FP JT Box-2	18,6	61.1	0.420	4.05
	FP JT Box-3	21,6	128	0.299	5.46
	FP JT Box-4	21,21	79.1	0.158	2.00
	FP JT Box-5*	24,9	655	0.790	5.08
Wheel	FP JT Wheel-1	3,9	85.6	1.81	2.17
	FP JT Wheel-2*	27,3	1016	0.225	3.19
	FP JT Wheel-3	12,18	99.7	0.218	0.905
	FP JT Wheel-4	24,3	151	2.32	7.82
	FP JT Wheel-5	24,27	117	0.127	1.08

Sample Type	CRREL #	Position†	Dry wt. (g) <2mm	Concentration (mg/kg)	
				2,4-DNT	NG
MULTI INCREMENT	MI-1	random	1864	0.699	2.32
	MI-2	random	1941	0.559	2.84
	MI-3	random	1868	0.591	3.08
	MI-4	random	2030	0.573	3.25
	MI-5 A,B,C**	random	1913	0.878	2.71
	MI-6	random	2015	0.690	2.42
	MI-7	random	2039	1.05	2.87
	MI-8	random	2037	0.776	2.79
	MI-9	random	1993	1.12	2.41
	MI-10 A,B,C	random	1968	1.07	3.17

* Box or Wheel sample that was split into 5 or 7 equal portions

** Value of subsample triplicates

† Moving north from southeast corner, then west by designated number of meters

Appendix F: Letter of Support – EPA Region 6

UNITED STATES ENVIRONMENTAL
PROTECTION AGENCY
REGION 6
1445 Ross Avenue, Suite 1200
Dallas, Texas 75202-2733

March 14, 2008

Mr. Frank McStay, Jr., P.E.
Attn: CESWD-MTM
FUDS Program Manager
Southwestern Division
US Army Corps of Engineers
1100 Commerce Street
Dallas, Texas 75242

Vincent Del Greco
ATTN: CESPD-PDM
Environmental/HTRW Program Manager
Program Manager, SPD Range Support Center
Military Integration Division
Department of the Army
South Pacific Division,
US Army Corps of Engineers
1455 Market Street
San Francisco, CA 94103-1399

RE: New Region 6 FUDS Coordinator; Recent MIS Protocol Agreement

Dear Messrs. McStay and Del Greco:

The purpose of this letter is to inform you that I have accepted a new position in the Water Division of the Environmental Protection Agency (EPA) Region 6 office, and will no longer be the Region 6 Formerly Used Defense Sites (FUDS) Coordinator. Effective immediately, Mr. Gary Miller will be the new FUDS coordinator. Gary has been our Texas FUDS Coordinator for many years and will do a fine job. I also wanted to thank both of you for the cooperative relationship that has existed between our offices, and the Corps' Districts, for many years now, and which is exemplified by our recent agreement on implementing an approach to multi-increment sampling (MIS) as part of your Military Munitions Response Program's (MMRP's) Site Inspections (SI's) effort.

I especially appreciate the efforts of Ms. Monique Ostermann of the Corps' Albuquerque District in setting up the meeting January 14-16, 2008, in Fort Worth, Texas, to discuss the MIS issue. Personnel from the Corps' Albuquerque and Fort Worth Districts, the Corps' SI contractors, EPA Region 6, the Arkansas Department of Environmental Quality, the Texas Commission on Environmental Quality, and (by telephone) the New Mexico Environment Department met to discuss how to implement MIS during the SI process. My colleagues at EPA and I were very pleased with the discussions and the consensus we reached through that process. It was extremely important to have this face-to-face discussion so that we could efficiently and openly discuss the data quality objectives (DQOs) for these SI projects and how we can best achieve them. By having this opportunity, we now have an approach which we all agreed is likely to achieve our DQOs at a manageable cost and level of effort (LOE) for the majority of our sites in Region 6.

I also wanted to thank Alan Hewitt of the Corps' Cold Regions Research and Engineering Lab (CRREL) in Hanover, New Hampshire, and Hugh Rieck of the Corps' Huntsville Engineering and Support Center, Omaha group, for participating in our meetings. Their first hand knowledge of conducting these types of investigations and the scientific basis for the sampling design and laboratory processes greatly aided our understanding of the best way to modify the design in order to meet DQOs and maintain LOE.

I want to thank all of the Corps' members who attended the meeting, including Don Silkebakken and his staff from Parsons, and our state partners from Arkansas, New Mexico, and Texas, for their outstanding efforts at resolving this contentious issue. I fully expect other EPA Regions and many States will also seek to utilize this approach in the near future.

Finally, regardless of our consensus reached at this meeting, all participants understood each site will still have to be considered individually, with an appropriate set of DQOs and sampling design for the site discussed at the TPP meetings. And of course, our State counterparts will always reserve their rights to act independently, too.

Here is the outline of the general consensus points I believe we reached either at the meeting, or at more recent follow-up meetings.

1. One data quality objective of the SI process is to achieve a level of information that would allow the Corps and the regulators to agree on whether further sampling for munitions constituents (MC) would need to be continued into the remedial investigation phase of these sites.

2. Multi-increment sampling will implemented at most, if not all, MMRP SI's to be started in Region 6 this FY. Exceptions should be discussed on a case by case basis.

3. When arriving on a FUDS munitions response site (MRS), a large area encompassing the targets or areas of interest will be visually inspected as the quick reconnaissance (QR). In the case of the practice bombing range (PBR) in our example, the proposed area was approximately 500 meters by 1,000 meters and encompassed all target areas. The visual inspection proposed in that example included walking transects approximately 50 meters apart. This will not result in a complete visual inspection in most cases due to limited site lines, but is expected to be capable of achieving the data quality objectives to confirm target locations and assess whether munitions debris (MD) found on the site are consistent with those expected based on the Archive Search Report (ASR), and whether munitions and explosives of concern (MEC) or munitions constituents (MC) are readily identifiable.

4. If MC is identified on the site, its presence would be sufficient to require MC sampling would be carried forth into the remedial investigation (RI) for the site.

5. For PBRs that have multiple targets, the primary target (in the case of our example PBR, the swastika) would have a 100 m x 100 m grid laid over it, with 100 increments collected for the sample.

6. Secondary targets would have a 50 increment sample collected from within the target outline, but the entire area within the outline would be visually inspected.

7. No replicate or triplicate samples would be collected at this stage, because the purpose of those efforts is to assess the precision and accuracy of the sample results. Because the sample results on these sites will only be compared to a detect/non-detect DQO standard for carrying MC sampling into the RI, such QA evaluations are not necessary at this time.

8. When MC HE (high explosives) are detected in any sample at the MRS, MC HE sampling will be carried forward into the RI. However, if no MC HE is detected, and the sampling design and number of samples was adequate, it is reasonable to conclude MC HE sampling will not be conducted in the RI, unless additional information is found to support that need.

9. Some sites, such as the munitions burial pit on the Naval Auxiliary Air Station (NAAS) site we discussed, cannot be adequately screened in the SI phase, and therefore MC sampling will be carried forward to the RI. The potential for this situation should be discussed at the TPP phase of the project.

10. Small sites, such as pistol or skeet ranges with an identifiable location, may or may not be able to collect a single MIS sample for multiple analyses (e.g., explosives/propellants, metals, PAHs). That determination will best be made on a site by site basis. However, while MIS seems promising for those types of non-volatile constituents, its performance has not yet been documented on other constituents.

11. Some sites may be too complicated, or in the opinion of the regulators require a large number of samples (e.g. there may be a large number of targets present on the site), to achieve the DQO of assessing whether MC sampling should continue into the RI within the limits of the Corps' LOE. To the extent possible, the Corps will accommodate those requests, but may not be able to sample all areas of interest in the SI. When the SI report is prepared, if the Corps concludes that no further MC sampling is warranted based on the results of this limited sampling effort, the regulators will be unlikely to agree with this conclusion. The potential for this situation should be discussed at the TPP phase of the project.

I am sure there will continue to be individual issues and sites which the above points will not adequately address, however, these guidelines do provide a solid foundation for us to improve the quality of the SI's being done and to increase the ability to reach consensus on conclusions and recommendations.

In conclusion, I want to again express my thanks for the cooperation I have received from the Corps on resolving this and other issues. I know that Gary can look forward to the same cooperation as he assumes the position of Regional FUDS coordinator. He may be reached at (214) 665-8306, or *miller.gary@epa.gov*. His mailing address is the same as mine. My telephone number and email address will remain the same, should you need to contact me in the future.

Best regards,

Michael Overbay

Michael Overbay, P.G.
Region 6 FUDS Coordinator

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE (DD-MM-YYYY)
June 2009

2. REPORT TYPE
Final

3. DATES COVERED (From - To)

4. TITLE AND SUBTITLE
Validation of Sampling Protocol and the Promulgation of Method Modifications for the Characterization of Energetic Residues on Military Testing and Training Ranges

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Alan D. Hewitt, Dr. Thomas F. Jenkins, Marianne E. Walsh, Susan R. Bigl, and Dr. Sylvie Brochu

5d. PROJECT NUMBER
ER-0628

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
U. S. Army Engineer Research and Development Center (ERDC)
Cold Regions Research and Engineering Laboratory (CRREL)
72 Lyme Road
Hanover, NH 03755-1290

8. PERFORMING ORGANIZATION REPORT NUMBER
ERDC/CRREL TR-09-6

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Environmental Security Technology Certification Program
901 N. Stuart St., Ste 303
Arlington VA 22203

10. SPONSOR/MONITOR'S ACRONYM(S)
ESTCP

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

The Military maintains sustainability and environmental stewardship responsibilities for training ranges. One of the environmental challenges is to monitor the surface loading of energetic residues. Method 8330B, which was developed to assist in this task, recommends the use of a *MULTI INCREMENT®* sampling strategy and total sample processing prior to subsampling and analysis. This demonstration/validation project evaluated scientifically defensible sampling and sample-processing protocols used for the characterization of energetic residues on military training ranges. This project also specifically assessed the scientific uncertainty involved with the now-common practice of splitting a sample in the field and subsampling it prior to processing the entire laboratory sample. To address the cost aspect, this study then evaluated the cost benefits of coupling *MULTI INCREMENT* sampling methodology with recently developed sample handling and processing protocols designed to address the unique attributes of energetic residues. Consequently, the authors recommend that *MULTI INCREMENT* sampling and the U.S. Environmental Protection Agency (USEPA) Method 8330B be used for future characterization activities on military training ranges.

15. SUBJECT TERMS
ESTCP, range management, military training, propellants, explosives, training ranges

16. SECURITY CLASSIFICATION OF:

a. REPORT
Unclassified

b. ABSTRACT
Unclassified

c. THIS PAGE
Unclassified

17. LIMITATION OF ABSTRACT

18. NUMBER OF PAGES
146

19a. NAME OF RESPONSIBLE PERSON

19b. TELEPHONE NUMBER (include area code)