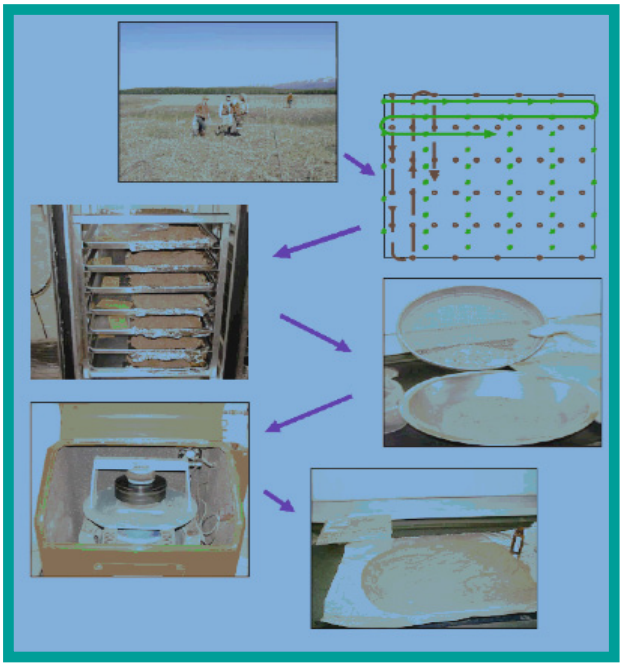
ESTCP
Cost and Performance Report

(ER-0628)



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

April 2009

ENVIRONMENTAL SECURITY
TECHNOLOGY CERTIFICATION PROGRAM

U.S. Department of Defense

COST & PERFORMANCE REPORT

ESTCP Project: ER-0628

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ACRONYMS AND ABBREVIATIONS

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
CFB	Canadian Forces Base
Composition B	60% RDX, 39% TNT, 1% wax
CRREL	Cold Regions Research and Engineering Laboratory
CSM	conceptual site model
DoD	U.S. Department of Defense
DQO	data quality objectives
DRDC-Val	Defence Research and Development Canada-Valcartier
EDQW	Environmental Data Quality Workgroup
EOD	explosive ordnance disposal
ERDC	Engineer Research and Development Center
ER	Environmental Restoration
ERL	estimated reporting limit
ESTCP	Environmental Security Technology Certification Program
FP	Firing Point
FUDS	Formerly Used Defense Sites
HEER	Hazard Evaluation and Emergency Response
HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HPLC	high performance liquid chromatography
ITRC	Interstate Technology & Regulatory Council
MIS	*MULTI INCREMENT* sampling
MMR	Massachusetts Military Reservation
MMRP	Military Munitions Response Program
NARPM	National Association of Remedial Project Managers
NG	Nitroglycerin
ORAP	Operational Range Assessment Program
OSW	Office of Solid Waste
PE	performance evaluation

QA	quality assurance
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
SERDP	Strategic Environmental Research and Development Program
TGM	technical guidance manual
TNT	2,4,6-trinitrotoluene
UCL	upper confidence limit
USEPA	U.S. Environmental Protection Agency
UXO	unexploded ordnance

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Technical material contained in this report has been approved for public release.

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1.0 EXECUTIVE SUMMARY

1.1 BACKGROUND

The U.S. Department of Defense (DoD) and U.S. Environmental Protection Agency (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) that have requirements to perform 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 Massachusetts Military Reservation (MMR) (USEPA 2000).

1.2 OBJECTIVES OF THE DEMONSTRATION

The objectives of this demonstration/validation project were to promote scientifically defendable sampling and sample-processing protocols for the characterization of energetic residues on military training ranges. The principal mechanism for meeting this objective has been to inform potential users of the sampling and sample processing protocols developed under the Strategic Environmental Research and Development Program (SERDP) Environmental Restoration (ER) projects ER-1155 and ER-1418. These objectives were promoted by:

- Posting of Method 8330B on the Web (USEPA, 2006a)
- Performing two field demonstrations
- Hosting workshops to promote the attributes of *MULTI INCREMENT*® sampling (MIS) and to highlight the laboratory modifications required for representative subsampling and analysis
- Actively assisting commercial laboratories prepare for certification in Method 8330B
- Demonstrating use of the MIS strategy at three MMRP Formerly Used Defense Sites (FUDS)
- Assisting the South Pacific Division Corps of Engineers, USEPA Region 6, the Corps Environmental and Munitions Center of Expertise, and the states of Texas, Utah, New Mexico, and Arizona in the development of MIS strategies for several different types of MMRP FUDS (see ER-0628 Final Report, Appendix F)
- Developing guidance for the implementation of Method 8330B by the DoD Environmental Data Quality Workgroup (EDQW) (http://www.navylabs.navy.mil/Archive/Final%208330B%20Implementation%20Guide%20070708.pdf)

- Accepting an MIS Interstate Technology & Regulatory Council (ITRC) project proposal (see Appendix B).

1.3 DEMONSTRATION RESULTS

The sample processing recommended in Method 8330B was demonstrated to be scientifically sound for obtaining representative subsamples. In the majority of cases, uncertainty among triplicate subsamples removed from soil samples over 1,200 g in mass was less than 10% relative standard deviation (RSD), and the average was within 10% relative percent difference (RPD) of the concentration obtained for the remainder of the sample.

In all cases, tighter tolerances were obtained for five *MULTI INCREMENT* samples as compared to 30 discrete samples for the calculation of upper confidence limits (UCL). That is, 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 five *MULTI INCREMENT* samples, and greater than the variation among the values established for 10 *MULTI INCREMENT* samples. Estimates of the 95% UCL of the mean based on only five discrete samples were either unreasonable or unreliable (unrepeatable), or both.

ProUCL, the statistical software, estimates of the UCLs of the mean based on 100 discrete samples were higher than:

- Individual *MULTI INCREMENT* samples
- 95% UCLs of the mean based on three or five *MULTI INCREMENT* samples
- Often greater than twice the grand mean (concentration established for between 18 and 34 kg) estimated for the area sampled.

1.4 IMPLEMENTATION ISSUES

The posting of Method 8330B on the Web (USEPA, 2006a) addressed most of the issues initially expressed by potential stakeholders and endusers. Within 18 months of posting, commercial laboratory services became available, removing the only remaining limitation to full-scale implementation.

2.0 INTRODUCTION

2.1 BACKGROUND

The Environmental Security Technology Certification Program (ESTCP) project ER-0628 recognized that use of different sampling and sample processing protocols would impact the data used to estimate mass loading of energetic residues on DoD training and testing ranges. These concerns were based on several sampling and sample-processing studies performed during the SERDP projects ER-1155 and ER-1481. Briefly stated, these projects showed that using a MIS strategy and a systematic-random sampling design routinely produced data sets with much less uncertainty and provided a better estimate of the mean concentrations of energetic residues within an area of concern than other commonly practiced procedures (Jenkins et al., 20050a; Jenkins et al., 2006; Hewitt et al., 2007a; Jenkins et al., 2008). In addition, whole sample pulverization (particle size reduction) was necessary to attain the degree of blending 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, ER-0628 revised Method 8330 by working with the USEPA Office of Solid Waste (OSW) organic methods working group. A consequence of this effort was the posting of Method 8330B on the USEPA's SW-846 Web page in November 2006. The demonstration phase of this program documented the attributes of Method 8330B and exposed the large uncertainty associated with the collection of discrete samples, field splitting of large samples, and subsampling of an inadequately processed field sample. Establishing that reliable and defensible environmental data is paramount to site characterization programs tasked with determining if remedial activities are needed to support training sustainability and stewardship responsibilities.

2.2 OBJECTIVES OF THE DEMONSTRATION

The principal objective of this effort has been to inform potential users of the sampling and sample processing protocols developed under ER-1155 and ER-1418, and consequently recommended in Method 8330B. During the demonstration portion of ER-0628, 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. In total, seven workshops attended by over 200 participants were held at the following locations: MMR; Engineering Research and Development Center (ERDC)-Cold Regions Research and Engineering Laboratory (CRREL); Army Environmental Command in Aberdeen, Maryland; Washington State; and the annual SERDP and ESTCP Partners in Environmental Technology Technical Symposium & Workshop in Washington, D.C.

The initial product of this demonstration was the posting of Method 8330B on the Web (USEPA, 2006a). Once posted, we offered laboratory services to a couple of groups to facilitate its rapid implementation. This offer was made because, at the time, commercial

laboratories were not prepared to perform the whole sample pulverization procedure. Today, at least four commercial laboratories offer the full sample-preparation protocol recommended in Method 8330B. We've also demonstrated the MIS strategy at three MMRP FUDS and assisted the South Pacific Division Corps of Engineers, USEPA Region 6, the Corps Environmental and Munitions Center of Expertise, and the states of Texas, Utah, New Mexico, and Arizona in developing MIS strategies for various types of ranges (see ER-0628 Final Report, Appendix F). As a consequence of commercial availability, several site investigations at MMRP FUDS have implemented Method 8330B.

2.3 REGULATORY DRIVERS

DoD and USEPA both have programs tasked with determining if military training and testing facilities present a risk to human health and the environment. DoD has established the MMRP and ORAP (DoD Directive 4715.11), which have requirements to perform site investigations. 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 the application of MIS strategy for the characterization of energetic residues on military training ranges have been given at the USEPA's National Association of Remedial Projects Managers (NARPM) and the USEPA's Quality Assurance (QA) meetings for the past couple of years. These two meetings—USEPA's Risk Assessors Meeting and Region III FUDS Partnership Conference—requested presentations in 2008. Furthermore, it is anticipated that meetings, conferences, and guidance focused on the implementation of the MIS strategy to meet the needs of risk assessment will be forthcoming in 2009. Indeed, ITRC accepted a proposal for a 2009 start to develop a guidance manual for MIS strategy (Appendix B). In addition, the State of Hawaii Hazard Evaluation and Emergency Response (HEER) office is currently developing a technical guidance manual (TGM) for the implementation of MIS strategies (http://hawaii.gov/health/environmental/hazard/ index.html).

3.0 TECHNOLOGY

3.1 TECHNOLOGY DESCRIPTION

3.1.1 Description

Energetic residues are deposited on DoD training ranges as particles of military explosives and as irregular fibers or pieces of propellants and rocket fuels. These energetic residue particles 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 (UXO) are found or are placed and then blown-in-place as part of a range clearance or training activity (Jenkins et al., 2001, 2004a, 2004b, 2006, 2008; Pennington et al., 2001–2006).

To representatively sample an area where particles have been dispersed, the sampling strategy must address the compositional and distribution 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 population (e.g., decision unit, 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 of >1 kg (Jenkins et al., 2004a, 2004b, 2005a, 2006, 2008; Walsh et al., 2006; Hewitt et al., 2005, 2007a). Figure 1 shows examples of teams surveying a range and collecting *MULTI INCREMENT* samples. The objective of this sampling technique is to obtain a representative amount of every particle size, composition (Tritonal, 60% RDX, 39% TNT, 1% wax [Composition B], H6, etc.), and configuration (e.g., crystalline spheres or elongated fibers), and to not oversample or miss any portion of the decision unit. The decision unit size is a function of the data quality objectives (DQO) and can be as small or as large as necessary to satisfy the DQO. The ability to meet the objective of high certainty depends on the type of range and training activity being performed. To empirically estimate the 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.

To obtain representative subsamples from a field sample, the processing protocol must also address the compositional and distribution heterogeneity. Method 8330B recommends the entire field sample be air dried and then passed through a #10 (2-mm) sieve (Figure 2). The sieved fraction of the sample is then mechanically pulverized to reduce the particle size of both the matrix and constituents of concern to <0.075 mm (Figure 3). 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

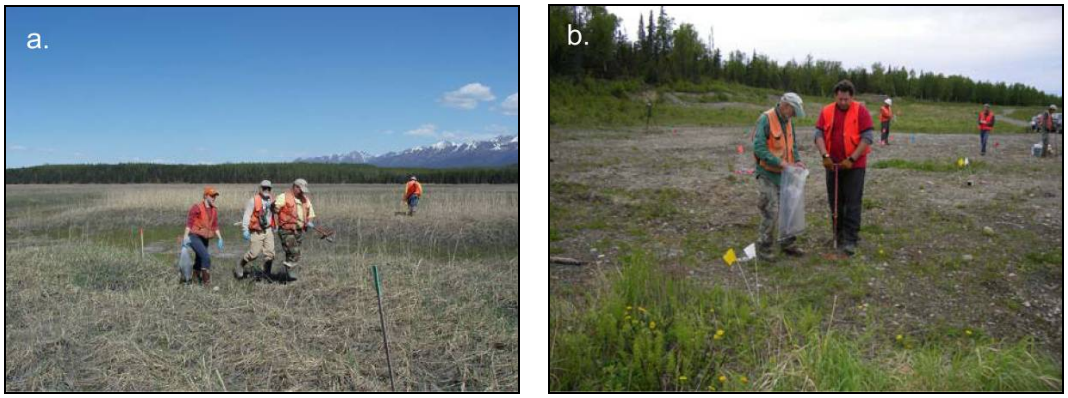

Figure 1. Field Sample Collection Techniques.
([a] Team surveying impact range for areas to sample [b] Area within a demolition range selected for sampling based on crater and pieces of 91% RDX, 9% oil (C4) observed on surface and sampling team collecting one of 10 *MULTI INCREMENT* samples)

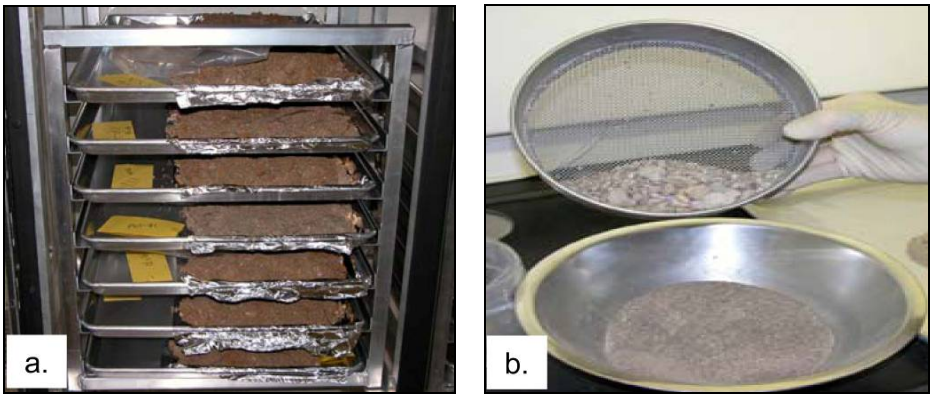

Figure 2. Laboratory Processing Steps.
([a] *MULTI INCREMENT* samples air-drying in rack
[b] Passing sample through #10 [2-mm] sieve)

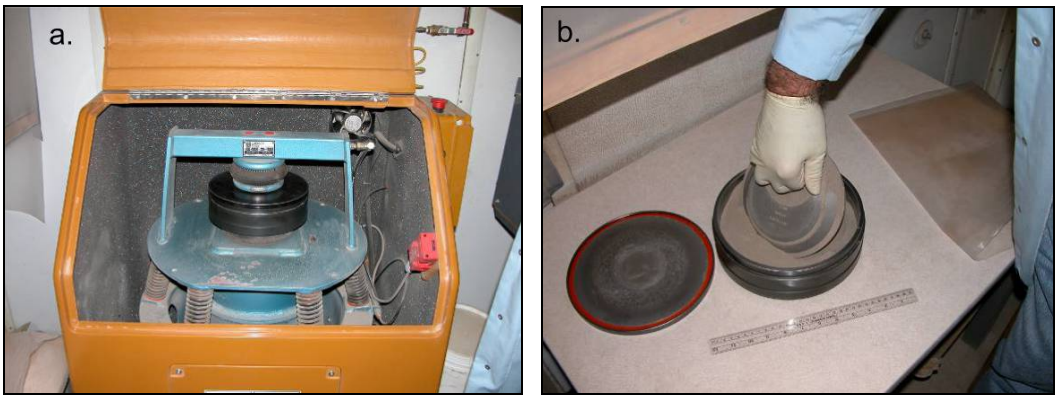

Figure 3. Sample Pulverization Step.
([a] Ring puck mill [b] Ground portion of *MULTI INCREMENT* sample)

dissolved and consistent with the classification of soil used in risk models for human exposure (Pennington et al., 2004). 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), and because of this is unacceptable.

To further reduce the uncertainty among subsamples, Method 8330B recommends a 10-g subsample size be obtained by combining many (>20) smaller increments (Figure 4). 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, instead of the 1:5 ratio cited in earlier Methods 8330 and 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, subsampling error cannot be assessed.

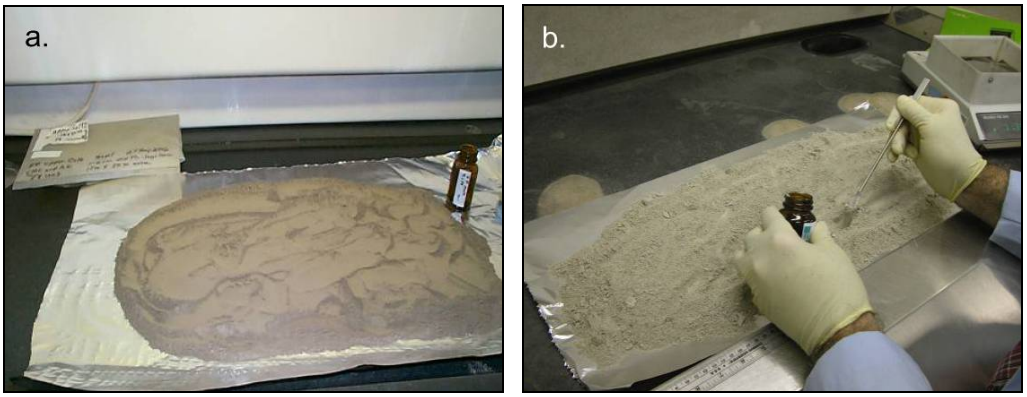

Figure 4. Subsampling Step.
([a] Mixed *MULTI INCREMENT* sample spread out to be 1–2 cm thick [b] Collecting 20 aliquots of ground and mixed multi-increment sample)

3.1.2 Schematic Diagram

The sampling and sample processing steps are shown in Figure 5 in simplified schematic form. This diagram covers the basic sample collection and processing steps; the project DQOs and Conceptual Site Model (CSM) should be used to guide the selection of the sampling decision unit or units and the number of increments needed to address the compositional and distribution heterogeneity of the energetic residues. As general rule, more increments and sample mass are needed to address these two sources of sampling error in locations where partial detonations are likely to have occurred as compared to locations where munitions are fired at both line-of-sight and remote targets.

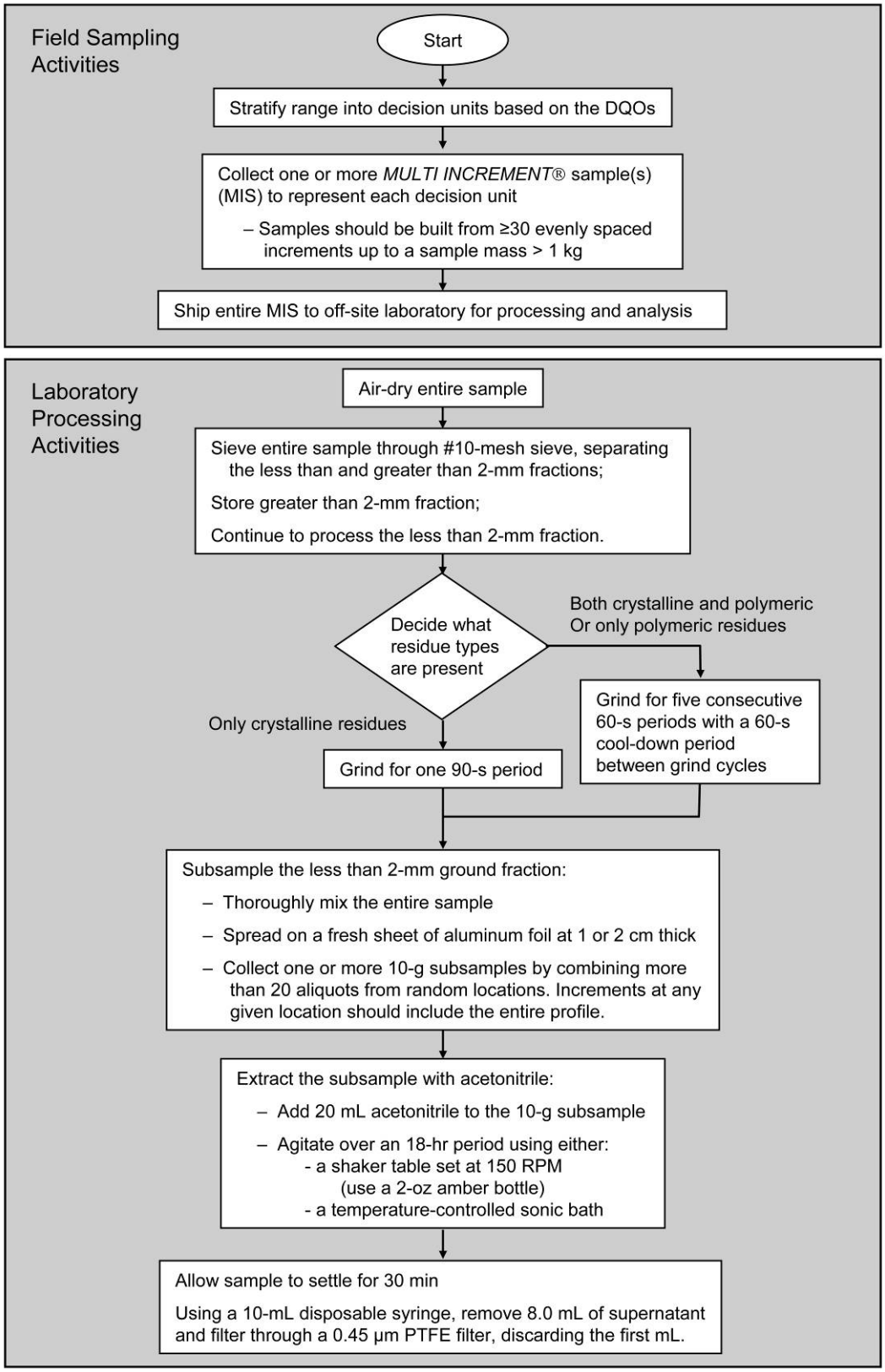

Figure 5. Method Schematic—Field and Laboratory Sample-Processing Steps Recommended by Method 8330B.

3.1.3 Chronological Summary of Technology Development

3.1.3.1 Early Studies

Modifications to our sampling strategy and sample processing protocols started in the middle of the 1990s. Below are some of the publications that show the transition between collecting discrete and *MULTI INCREMENT* samples for characterizing military training ranges and the processing of these larger samples to obtain representative estimates of energetic residue concentrations.

1995 to 2003 – Initial MIS Studies

Simple field screening method for white phosphorus in sediment. M.E. Walsh, C.M. Collins, C.H. Racine, C. Bouwkamp, and P.G. Thorne. CRREL Special Report 95-25.
http://www.crrel.usace.army.mil/techpub/CRREL_Reports/reports/SR95_25.pdf

Composite Sampling of Sediments Contaminated with White Phosphorus. M.E. Walsh, C.I. Collins, R.N. Bailey, and C.L. Grant. CRREL Special Report 97-30, December 1997.
http://www.crrel.usace.army.mil/techpub/CRREL_Reports/reports/SR97_30.pdf

Assessment of sampling error associated with the collection and analysis of soil samples at a firing range contaminated with HMX. T.F. Jenkins, M.E. Walsh, P.G. Thorne, S. Thiboutot, G. Ampleman, T.A. Ranney, and C.L. Grant. CRREL Special Report 97-22.
http://www.crrel.usace.army.mil/techpub/CRREL_Reports/reports/SR97_22.pdf

Monitoring of remediation of a salt marsh contaminated with white phosphorus. Marianne E. Walsh, Charles M. Collins, and Ronald N. Bailey. In Wetlands and Remediation. J.L. Means and R.E. Hinchee, Eds. Battelle Press, 2000, pp 349-356.

Sampling for Explosives Residues at Fort Greely, Alaska: Reconnaissance Visit July 2000. M.E. Walsh, C.M. Collins, C.H. Racine, T.F. Jenkins, A.B. Gelvin, and T.A. Ranney. ERDC/CRREL TR-01-15, November 2001.
http://www.crrel.usace.army.mil/techpub/CRREL_Reports/reports/TR-01-15.pdf

Sampling for Explosives-Residues at Fort Greely. M.E. Walsh, C.M. Collins, T.F. Jenkins, A.D. Hewitt, J. Stark, and K. Myers. Soil and Sediment Contamination, 12:631-645 (2003).

2002 to 2007 – Sample Processing Development

The Effect of Particle Size Reduction on Subsampling Variance for Explosives Residues in Soil. M.E. Walsh, C.A. Ramsey, and T.F. Jenkins. Chemosphere, 49:1265-1271 (2002).

Collection methods and laboratory processing of samples from Donnelly Training Area firing points Alaska 2003. M.E. Walsh, C.A. Ramsey, C.M. Collins, A.D. Hewitt, M.R. Walsh, K. Bjella, D. Lambert, and N. Perron, ERDC/CRREL TR 05-06 (March 2005).
http://www.crrel.usace.army.mil/library/technicalreports/TR05-6.pdf

Subsampling Variance for 2,4-DNT in Firing Point Soils, M.E. Walsh, C.A. Ramsey, S. Taylor, A.D. Hewitt, K. Bjella, and C.M. Collins, Soil and Sediment Contamination, An International Journal, 16 (5):459 (2007).

3.1.3.2 SERDP-Funded Studies

Funding for the SERDP project ER-1155 "Distribution and Fate of Energetics on DoD Testing and Training Ranges: Characterization of Explosives Contaminates," started in 2000. The second annual Interim Report (2002) and all subsequent reports included papers describing the use of MIS strategy to representatively characterize training ranges. Moreover, a collection of these works led to three journal publications.

2000 to 2005 – SERDP Products

Distribution and fate of energetics on DoD test and training ranges: Report 2. Pennington, J.C., T.F. Jenkins, G. Ampleman, S. Thiboutot, J.M. Brannon, J. Lynch, T.A. Ranney, J.A. Stark, M.E. Walsh, J. Lewis, C.H. Hayes, J.E. Mirecki, A.D. Hewitt, N.M. Perron, D.J. Lambert, J. Clausen, and J.J. Delfino. 2002. Vicksburg, MS: U.S. Army Engineer Research and Development Center. ERDC TR-02-8.
http://el.erdc.usace.army.mil/elpubs/pdf/tr02-8.pdf

Distribution and fate of energetics on DoD test and training ranges: Report 3. Pennington, J.C., T.F. Jenkins, G. Ampleman, S. Thiboutot, J.M. Brannon, J. Lewis, J.E. Delaney, J. Clausen, A.D. Hewitt, M.A. Hollander, C.A. Hayes, J.A. Stark, A. Marois, S. Brochu, H.Q. Dinh, D. Lambert, R. Martel, P. Brousseau, N.M. Perron, R. Lefebvre, W. Davis, T.A. Ranney, C. Gauthier, S. Taylor, and J.M. Ballard. 2003. Vicksburg, MS: U.S. Army Engineer Research and Development Center. ERDC TR-03-2.
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3.1.3.3 ESTCP-Funded Studies

Funding from the ESTCP started in 2006 for ER-0628, "Validation of Sampling Protocol and the Promulgation of Method Modifications for the Characterization of Energetic Residues on Military Testing and Training Ranges." In the first year, Method 8330B was posted on the EPA SW-846 Web page. Following posting of this revised method, a guide for sampling military training ranges and a report addressing laboratory protocols for sample grinding were published. Lastly, *MULTI INCREMENT*® sampling became a registered trademark.

2006 to 2009 – ESTCP Products

Nirtoaromatics, Nitramines, Nitrate Esters by High Performance Liquid Chromatography (HPLC). U. S. Environmental Protection Agency. 2006a. Washington, D.C.: U.S. Environmental Protection Agency. SW-846 Method 8330B.
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Protocols for collection of surface soil samples at military training and testing ranges for the characterization of energetic munition constituents. Hewitt, A.D., T.F. Jenkins, M.E. Walsh, M.R. Walsh, S.R. Bigl, and C.A. Ramsey. 2007. Hanover, New Hampshire: U.S. Army Engineer Research and Development Center. ERDC/CRREL TR-07-10.
http://www.crrel.usace.army.mil/library/technicalreports/TR07-10.pdf

Processing of training range soils for the analysis of energetic compounds. Hewitt, A.D., S.R. Bigl, M.E. Walsh, and S. Brochu. 2007. Hanover, New Hampshire: U.S. Army Engineer Research and Development Center. ERDC/CRREL TR-07-15.
http://www.crrel.usace.army.mil/library/technicalreports/TR07-15.pdf

Publication of Method 8330B led to the rapid increase in use of the term multi-increment. Since in many cases this term was being interchanged with composite and or the sampling strategy and design failed to address compositional and distribution error, a formal definition was published under the registered trade name of *MULTI INCREMENT*® to limit confusion. The definition is as follows:

MULTI INCREMENT—a comprehensive sample collection methodology used to represent a specific population (decision unit) and provide a foundation for defensible decision making. The term *MULTI INCREMENT* incorporates in a systematic approach many disciplines into the final sampling plan design and evaluation. The process begins with proper development of project objectives (e.g., USEPA's 7-step DQO process). Next, the tenants of Sampling Theory are employed to determine the appropriate sample mass, number of increments, and correct selection and use of sampling tools. Quality control is then incorporated for assessment of the sampling plan design and, ultimately, data quality. The combination of these disciplines forms the foundation of the *MULTI INCREMENT* methodology that generates a representative sample that directly provides data to meet project objectives.

MIS methodology can be applied to all project objectives, all media, and all analytes. It can be applied in the field and in the laboratory when subsampling of the field sampling is required. Users of *MULTI INCREMENT* must be aware that they cannot pick and choose only certain components of the method for their own convenience as that will not lead to a representative sample. If applied correctly *MULTI INCREMENT* is by definition the most cost-effective, defensible sampling collection methodology that can be implemented. *MULTI INCREMENT*® is a registered trademark of EnviroStat, Inc. (http://www.envirostat.org).

3.1.4 Technology Development Under ESTCP

The list below chronicles events that occurred during the ESTCP-funded project ER-0628. In addition to the listed events, several conference presentations were given and four workshops were held.

March 2006	Project awarded
November 2006	Method 8330B posted on USEPA Web page
June 2007	First field demonstration: Fort Richardson, Alaska
October 2007	Second field demonstration: Canadian Forces Base Petawawa
January 2008	Region 6 issues letter recommending the use of MIS and Method 8330B for MMRP-FUDS
June 2008	DoD Environmental Data Quality Workshop issues a "Guide for Implementing EPA SW-846 Method 8330B"
September 2008	ITRC accepts the project proposal "*MULTI INCREMENT* Sampling (MIS)" (Appendix B)

3.1.5 Applications of Technology

This technology was developed for site investigation and remedial investigations of formerly used and active military training and testing ranges. To date, Method 8330B and the MIS strategy have been implemented at the MMR on Cape Cod, Hill Air Force Base demolition range, Ravenna Army Ammunition Depot, and at the following MMRP-FUDS: Kannu Puu and Mahukona bombing ranges on the Hawaiian Islands and a practice bombing range in Idaho. The issuing of a letter from USEPA Region 6 that recommends the use of Method 8330B and MIS should facilitate implementation at some 200 MMRP-FUDS (available in Appendix F of the ER-0628 Final Report).

3.2 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

Sampling and analysis plans based on scientifically sound (defendable) studies will expedite range characterization programs addressing the burden of energetic residues on military training ranges. Once the initial site investigation studies have been performed, the proper remediation and management investigations 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.

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. 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 regraded, residues can become buried some 1 to 2 m below the surface. Development of sampling designs for practical implementation of the MIS strategy along with Triad, for the characterization of a three dimensional area of concern, is a research topic that should receive attention in the future.

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4.0 PERFORMANCE OBJECTIVES

Performance objectives targeted in ER-0628 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%**

*RSD = relative standard deviation
**Total measurement error

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5.0 SITE DESCRIPTION

5.1 SITE LOCATIONS

5.1.1 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 6) 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. The current Fort Richardson mission is to support the rapid deployment of Army forces from Alaska to the Pacific Theater and worldwide.

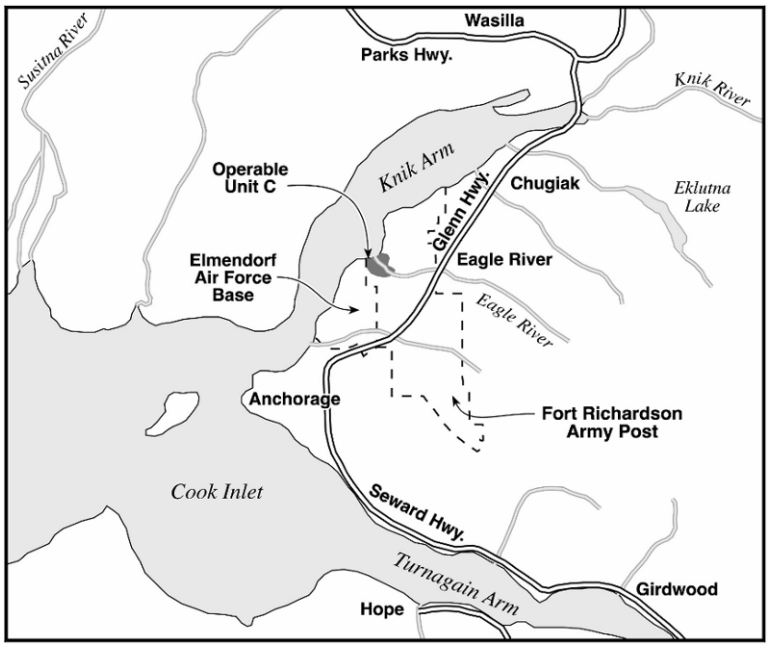


Figure 6. Fort Richardson and Surrounding Areas.

5.1.2 CFB Petawawa

CFB Petawawa is located approximately 160 km northwest of Ottawa, in the province of Ontario, Canada (Figure 7). Military activities at Petawawa began in 1905 on a 90-km^2 area located adjacent to the Ottawa River. The base has grown in importance since the mid 1990s with the closure of many Canadian Forces army bases.

5.2 SITE GEOLOGY/HYDROLOGY

The advantages of conducting the study at these two sites were not based on either geological or hydrological factors, but on our close working relationship with Range Control. As a consequence, both the Defence Research and Development Canada-Valcartier (DRDC-Val) and ERDC-CRREL, respectively, experienced good communications and expediency of field operations during the two field demonstrations.

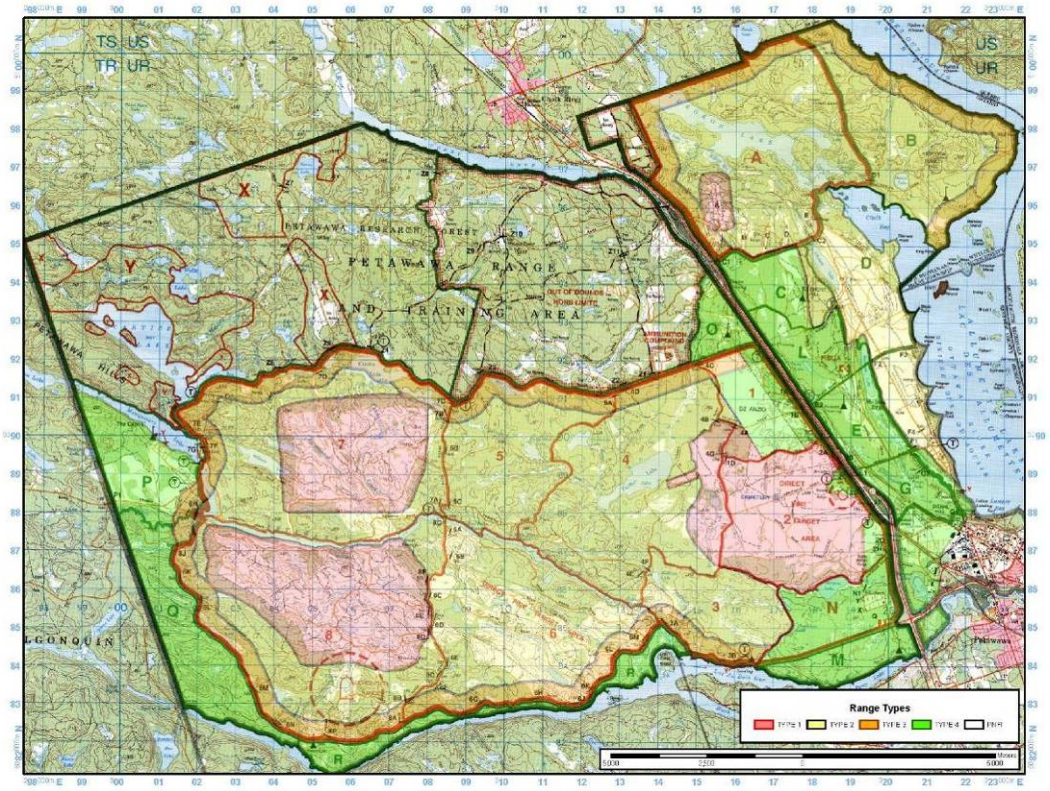


Figure 7. Layout of Training Ranges at CFB Petawawa.

5.3 CONTAMINANT DISTRIBUTION

Pre-demonstration sampling at Fort Richardson performed in August 2006 led to the following range being selected: 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 nitroglycerin (NG) concentrations between 1 and 30 mg/kg (Walsh et al., 2007). At the demolition range, concentrations of Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX), Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), 2,4,6-trinitrotoluene (TNT), and 2,4-dinitrotoluene (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 (Walsh et al., in preparation).

Pre-demonstration sampling at CFB Petawawa was conducted in October of 2005 and 2006. The ranges selected for sampling during this demonstration study were FP Juliet Tower and a hand grenade range. At this firing point, 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).

6.0 TEST DESIGN

6.1 CONCEPTUAL EXPERIEMENTAL DESIGN

This demonstration compared the level of uncertainty in data obtained when applying an MIS strategy and whole sample processing protocol described in Method 8330B with that resulting from discrete sampling and 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 was compared by calculation of the UCL of the mean with 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 was to show that MIS strategy and whole sample processing is the most cost effective way to meet acceptance criteria necessary for estimating the mean concentration of energetic residues on military training ranges.

6.2 BASELINE CHARACTERIZATION

See Section 4.3.

6.3 TREATABILITY OR LABORTORY STUDY RESULTS

N/A

6.4 FIELD TESTING

See Section 4.3.

6.5 SAMPLING METHODS

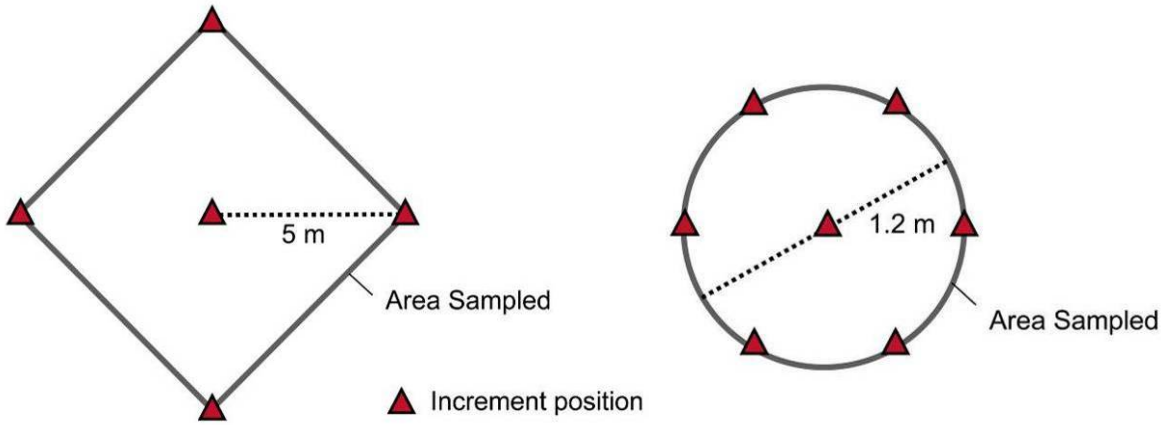
Sample collection and handling protocols followed the guidelines provided in Method 8330B.

Table 2 lists the number and types of samples collected at the five sampling areas. A constant sampling depth of 2.5 cm was used in all sampling activities. Five box and wheel samples were collected within each sampling area. For the box sampling design, five increments are combined to form a sample. The increments were collected at the center and at 5-m distances from this point moving in the four cardinal directions (Figure 8a). 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 8b). Ten *MULTI INCREMENT* samples were collected, each composed of 100 increments collected at evenly spaced intervals within the decision unit (Figure 9). This number of increments (100) was chosen to maximize precision among the field sample replicates. Ideally, the number of increments collected within a sampling area would be addressed during the development of a project's data quality objectives. It should be noted that increasing the number of increments

from 30 to 100 has impact on the sampling effort and often leads to larger samples, both of which impact field and analytical costs.

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
Hand Grenade Range	100	5	5	10
Firing point Juliet Tower	100	5	5	12



a. Five-increment 50-m^2 box **b. Seven-increment 1.2-m diameter wheel**

Figure 8. Conventional Sampling Designs.

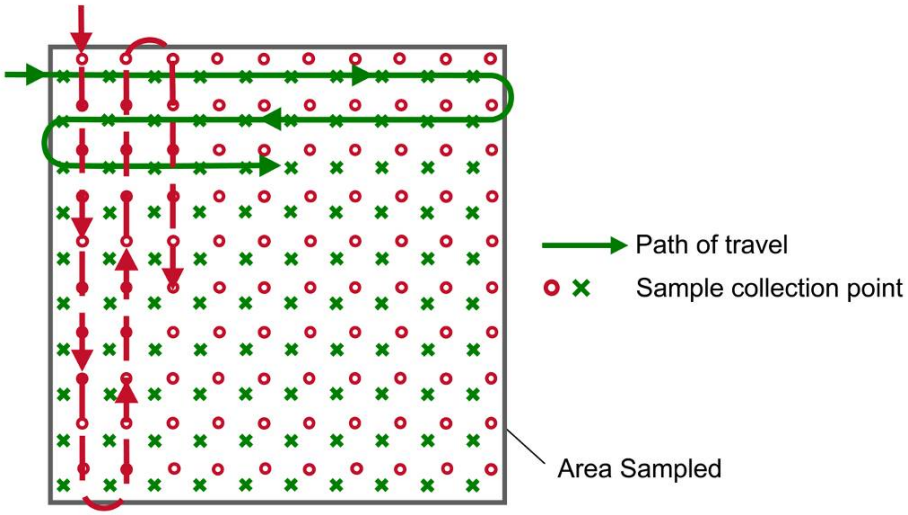


Figure 9. Illustration of the MIS Designs for the Collection of Two Separate Samples.

Lastly, either 100 or 200 discrete samples were collected within decision unit at either totally randomly selected positions or at evenly spaced positions after randomly establishing a starting position (i.e., same location in each 100 subgrids of the selected decision unit).

6.6 SAMPLING RESULTS

As noted in Section 3.1, 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 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.

6.6.1 Sample Extract Preparation and Analysis

Among the 15 samples selected, there were 32 sets of triplicate analyte determinations. Overall, the 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 1.

6.6.2 Subsampling Error

Both the error associated with removing subsamples from sample jars prior to processing the entire sample and after processing the entire sample were assessed. In all there were potentially 37 sets of estimated analyte concentrations for the triplicate subsamples, removed from unprocessed samples. Among the 31 triplicate sets with complete analytical results above the estimated reporting limit (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 nondetections, i.e., false negatives, and the large and erratic variance among triplicate subsamples established a level of uncertainty that is much greater than the target threshold of 10% RSD for laboratory subsampling (Table 1).

Concentration estimates were also obtained for each entire sample (subsamples 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). Figure 10 illustrates the distribution and degree of bias observed for subsamples removed (scooped off the top) from unprocessed samples.

The results for the triplicate subsamples removed from the processed *MULTI INCREMENT* samples, showed that analytes were detected in all the replicates that were collected after mechanical pulverization of the entire sieved (<2-mm) fraction. 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 (Table 1) and is a vast improvement over the commonly employed scoop-off-the-top method.

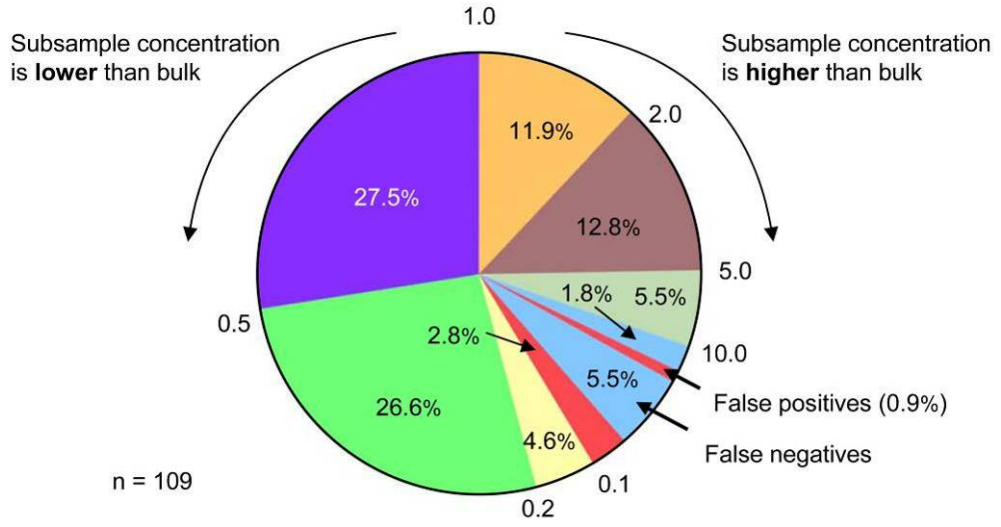


Figure 10. Distribution of the Ratios of the Subsample Concentration to the Total Sample Estimated Concentration for 109 Samples Analyzed.

To further 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. The RPD between the mean for the concentration estimates of the triplicate subsamples and bulk sample ranged from 1.4 to 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.

6.6.3 Field Sample Splitting Error

The random error associated with splitting a sample collected using the box or wheel sampling design in the field was evaluated by removing several splits from a single sample. Among 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%. The uncertainty associated with this practice far exceeds the target threshold of 10% for field splitting (Table 1).

Concentration estimate were also 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 11). This comparison indicates that the majority (76%) of sample splits contained analyte concentrations that were within a factor of two of the “bulk concentration estimate” for the sample collected in the field.

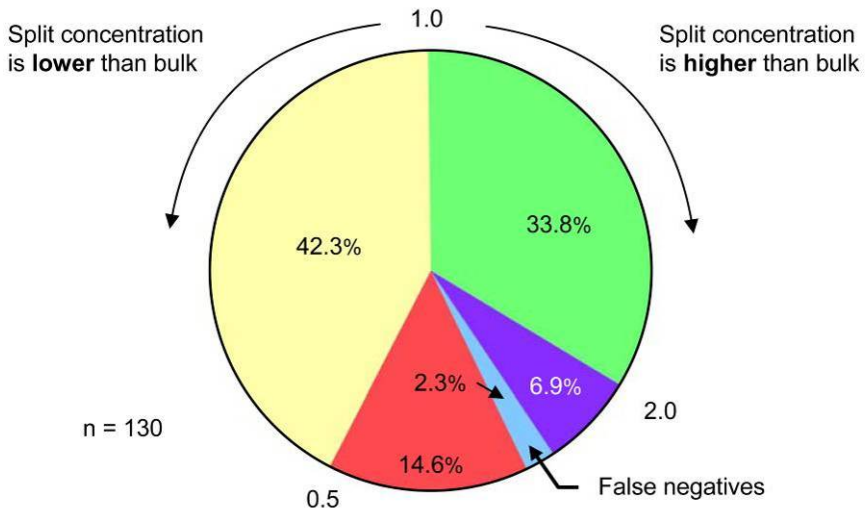


Figure 11. Distribution of the Ratios of the Sample Split Concentration to the Total Sample Estimated Concentration for 130 Samples Analyzed.

6.6.4 Uncertainty in the Mean and the UCL of the Mean for a Single Population

The 95% UCL of the mean were estimated using the Student's t-test for data sets with three values, and with ProUCL version 4.0 when more than three values were available (USEPA, 1992, 2007). This evaluation only addresses samples that were adequately processed prior to subsampling or samples extracted in their entirety, and therefore were not subject to 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 95% UCL were tabulated for each sampling strategy independent of establishing a normal distribution of values. Therefore, in some cases these values serve only as qualitative inferences of uncertainty.

Firing Point Fox. Within the 40 × 40-m area chosen for this study, NG was detected in 88% of the 100 randomly located discrete samples. Its concentration ranged over four orders of magnitude, from less than the ERL of 0.065 to 196 mg/kg. NG was detected in all the *MULTI INCREMENT* samples. Table 3 reports the mean, median, variance, and %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 nondetects.

Table 4 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 nondetects were estimated using lognormal regression on order statistics (ROS).

To further explore the range of possible 95% UCLs of the mean that could be obtained from these data sets, five replicates composed of three and five randomly selected *MULTI INCREMENT* samples and groups of five and 30 randomly selected discrete samples were evaluated using the Student's t-test and ProUCL (Table 5). Estimating 95% UCLs with the Student's t-test was necessary since ProUCL requires four values. This exercise used a random number generator and did not allow the same sample to be selected twice within a single group; values for the

nondetects were estimated with lognormal regression on order statistics. 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 3. Summary of Sampling Results at Firing Point Fox.

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

* Results with outlier removed

Table 4. Estimates of the 95% 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 Nonparametric 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 5. Estimates of the 95% 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 in parentheses.
** Student's t-test, all others 95% UCLs were estimated with ProUCL

The 95% UCLs of the mean based on five 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 three or five *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 et al., 2005). In one case this highest value represents only half of the ProUCL predicted 95% UCL of the mean.

Combining the mass of NG and that of the soils processed with the 100 discrete and nine *MULTI INCREMENT* samples (18.1 kg) establishes a grand mean concentration of 5.70 mg NG/kg for the area sampled. The UCL derived from the nine *MULTI INCREMENT* samples (absent of the outlier) is the best UCL for the grand mean concentration of the population at this site. Moreover, UCL values derived from three or five *MULTI INCREMENT* samples were reasonable and varied by less than a factor of two, 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.

Demolition Range #3. The 30-m × 30-m area selected for sampling the 100 randomly located discrete samples confirmed the presence of all the previously detected compounds at this range. 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-dintrotoluene (2,6-DNT), 2-amino-4,6-dinitrotoluene (2AmDNT), and 4-amino-2,6-dinitrotolune (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. Table 6 reports the mean, median, variance, and %RSD values for HMX, RDX, and 2,4-DNT for the two sampling protocols used at this site. To estimate the mean and variance for 2,4-DNT in the discrete samples, a value half of the ERL was substituted for the nondetects.

Among these data sets, only the 2,4-DNT concentration estimates for the *MULTI INCREMENT* samples are normally distributed (Figure 12). The large range of values for HMX and RDX among the *MULTI INCREMENT* samples was the consequence of one elevated sample, suggesting presence of a "nugget effect" that is, small (<2-mm) C4 pieces were at one or more locations within the chosen sampling area (Table 6). 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 for the mean concentration of 2,4-DNT.

Table 6. Summary of Sampling Results at Demolition Range #3.
(Analyte concentrations are presented in mg/kg.)

Statistic	a. Discrete Samples			b. *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%

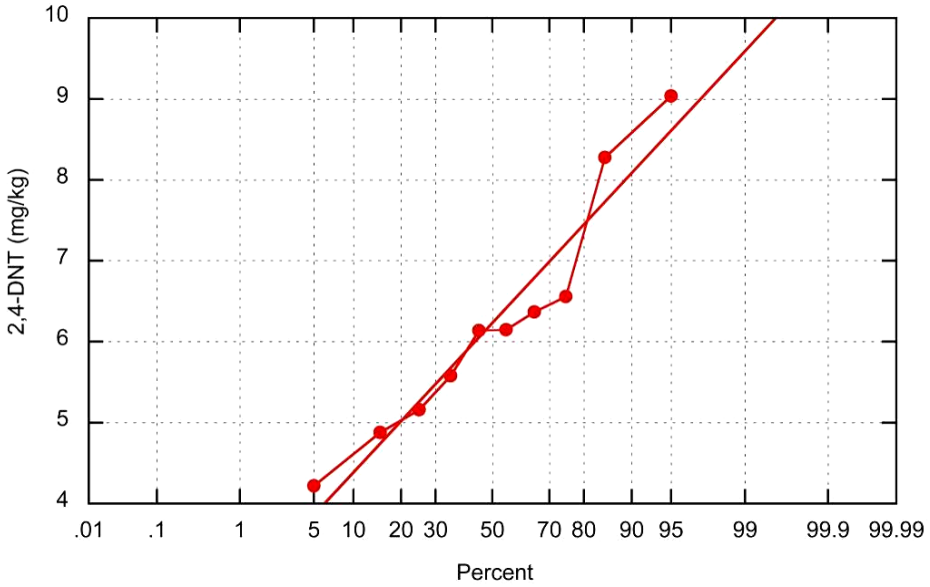


Figure 12. Probability Plot of the 2,4-DNT Concentration Estimates for 10 *MULTI INCREMENT* samples Collected at Demolition Range #3.

Table 7 lists the ProUCL estimates of the 95% UCL of the mean for 2,4-DNT derived from the discrete and the MIS strategies (USEPA, 2007). In the case of the discrete samples, values for the nondetects were estimated using gamma ROS.

Table 7. Estimates of the 95% 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 Nonparametric 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

To further 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 five and 30 randomly selected discrete samples were evaluated using the Student's t-test and ProUCL (Table 8). This exercise used the same logic as described previously.

Table 8. Estimates of the 95% 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 other 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 five 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 three and five *MULTI INCREMENT* samples. Combining the mass of 2,4-DNT and soils processed among the 100 discrete and ten *MULTI INCREMENT* samples (28.1 kg) yields an estimated grand mean concentration of 7.05 mg 2,4-DNT/kg for area sampled. The UCL of the mean based on ten *MULTI INCREMENT* samples and random sets of five *MULTI INCREMENT* samples were both very reasonable and consistent relative to the grand mean of this population. Indeed, the 95% UCLs based on three *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 five, 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 for 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 doesn't appear to be same as for HMX and RDX since 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 nondetects 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. The %RSDs for the *MULTI INCREMENT* samples was below the targeted threshold of 30% listed in Table 1 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 fewer of these samples.

Impact Range Low Order #3. The 20 × 20-m area sampled in this demonstration encompassed a crater formed by a low-order detonation. Among 200 discrete samples, HMX was the most frequently detected compound, at 41%. 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. 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 range of values and the median are reported; the minimum and median are displayed as ERL (Table 9). For the *MULTI INCREMENT* samples, Table 9 reports the mean, median, variance, and %RSD values for HMX, RDX, and TNT.

Table 9. Summary of Sampling Results at Low Order #3.
(Analyte concentrations are presented in mg/kg)

Statistic	a. Discrete Samples			b. *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 10 lists the estimates of the 95% UCLs of the respective means of RDX derived from the discrete and the MIS strategies (USEPA, 1992, 2007). Nondetect values for the discrete samples were estimated using gamma ROS.

Table 10. Estimates of the 95% 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 Nonparametric Computational Method
200 Discrete – systematic/random	153 – 95% KM (Chebyshev) UCL *
10 *MULTI INCREMENT* – systematic/random	44.2 – 95% Approximate Gamma UCL **

* nonparametric
** parametric

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 11). Larger numbers of discrete samples were selected in this case because of the large number of nondetects. Aside from the number of discrete samples, this exercise used the same logic as presented previously.

Table 11. Estimates of the 95% 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 other 95% UCLs were estimated with ProUCL

The possible 95% UCL of the mean based on 30 and 50 discrete samples, and three or five *MULTI INCREMENT* samples were erratic. Likewise, there was a large spread in values for the individual 100-increment samples. Combining the mass of RDX and soils processed among the 200 discrete and 10 *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 yield 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 that was within a factor of 6 of the grand mean; also, the 95% UCLs based on three or five *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 four.

Hand Grenade Range. The 20 × 20-m area selected for sampling was in the middle of the impact range, 5 m from the front wall of the grenade castle. HMX and RDX were present in concentrations above their ERLs in 93% of the discrete samples. TNT and its two breakdown products were only detectable in a couple of discrete samples. All the *MULTI INCREMENT* samples had detectable levels of HMX and RDX; only one had detectable TNT. The mean, median, variance, and percent RSD values for HMX and RDX are reported in Table 12. To estimate the mean and variance for HMX and RDX for the discrete samples, a value half of the ERL was substituted for the nondetects.

Table 12. 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%

HMX concentrations among the *MULTI INCREMENT* samples appear 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 13 lists the ProUCL estimates of the 95% UCLs of the mean for RDX derived for the discrete and MIS strategies (USEPA, 2007). Nondetect values for the discrete samples were estimated using gamma ROS.

Table 13. Estimates of the 95% 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 Nonparametric 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

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 14). This exercise used logic as described previously.

Table 14. 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

The UCL of the means based on five or 30 discrete samples were generally higher and more variable than those based on the five *MULTI INCREMENT* samples. In comparison to the UCL intervals based on three *MULTI INCREMENT* samples and the individual values for the 100-increment samples, the UCLs based on five discrete values were much more variable, whereas, 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 the area sampled. The UCL derived from the 10 *MULTI INCREMENT* samples is the best upper confidence limit for the grand mean concentration of the population at this site; however; those derived from five *MULTI INCREMENT* samples were very reasonable. The UCLs of the mean based on three *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 five, 30, and 100 discrete samples.

FP Juliet Tower. Within the 30 × 30-m area sampled, NG was detected in all of the discrete samples; 2,4-DNT was detected in 94%. Both energetic residues were detected in all the *MULTI INCREMENT* samples. Table 15 reports the mean, median, variance, and %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 half of the ERL was substituted for the nondetects.

Table 15. Summary of Sampling Results at FP Juliet Tower.
(Analyte concentrations are presented in mg/kg)

Statistic	Discrete		*MULTI INCREMENT*	
	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%

Among these data sets, the 2,4-DNT and NG concentration estimates for the *MULTI INCREMENT* samples are normally distributed (Figure 13). Table 16 lists the ProUCL estimates for the 95% UCL intervals of the means for 2,4-DNT and NG derived for the discrete and MIS strategies (USEPA, 2007). In the case of the discrete samples, the nondetect values for 2,4-DNT were estimated using lognormal ROS.

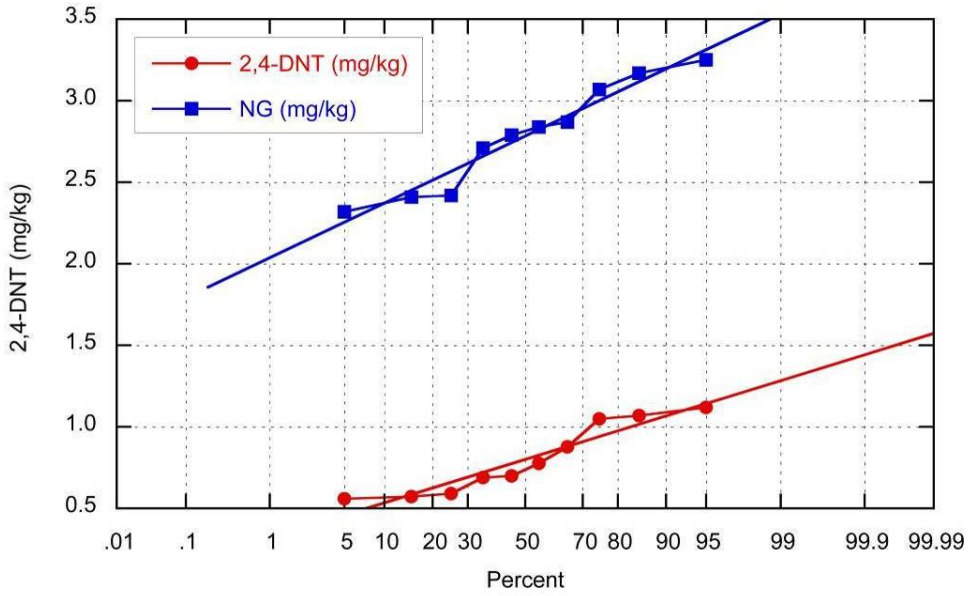


Figure 13. Normal Probability Plot of Estimates of NG and 2,4-DNT in *MULTI INCREMENT* Samples at FP Juliet Tower.

Table 16. 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 Nonparametric 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

To further explore the range of possible 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 17). This exercise used the same logic as described previously.

Table 17. 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

The ProUCLs of the mean based on five and 30 discrete samples were generally higher and more variable than the individual means of the 100-increment samples and the UCLs based on five *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) yields 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 three and five *MULTI INCREMENT* samples were both very reasonable and had consistent upper confidence intervals for this population. Indeed, each value for the 100-increment samples was a more reasonable estimator of the grand mean concentrations for 2,4-DNT and NG than the UCLs of the mean based on five, 30, or 100 discrete samples. The %RSDs for the *MULTI INCREMENT* samples was below the targeted threshold of 30% listed in Table 1 for both 2,4-DNT and NG at this sampling location, and very reasonable and reproducible UCL intervals of the mean concentration of these two energetic residues can be obtained based on five or fewer of these samples.

7.0 PERFORMANCE ASSESSMENT

A great deal 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 was 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, 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 subsample that was extracted and analyzed and do 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 subsampling error, this method recommends that the entire sample be air-dried and sieved, the less than 2-mm fraction be thoroughly pulverized (mechanically ground) and subsequently mixed, and a *MULTI INCREMENT* subsample be collected for extraction and analysis. In the majority of cases, uncertainty among triplicate subsamples removed from soil samples over 1,200 g in mass was less than 10% RSD. 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 and 34 kg of soil.

The central limit theorem of statistics states that distribution 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 common among the discrete sample data sets in 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: 1) individual 100-increment sample values, and 2) 95% UCLs of the mean based on three or five *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 five *MULTI INCREMENT* samples, and greater than the variation among the values based on 10 individual 100-increment samples. In all cases, tighter tolerances were obtained for five *MULTI INCREMENT* samples as compared to 30 discrete samples. Estimates of the 95% UCL of the mean based on only five 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).

8.0 COST ASSESSMENT

The major future cost differences between the recommended protocols in Method 8330B and those currently in practice will be a fewer number of actions associated with unnecessary management of cleanup activities due to a lower false positive error. Conversely, Method 8330B has a more reliable detection rate, thus avoiding false negatives. Presently a lot of attention is being given to cost associated with 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 defendable environmental data." It is anticipated that this will guide better stewardship of military training ranges, limiting the amount of costly remediation programs that would be incurred if residues migrate off base.

8.1 COST MODEL

Table 18 presents a cost estimate comparison between the expenses for 30 and 100 discrete samples, along with those for one, three, and five *MULTI INCREMENT* samples using only the categories with anticipated differences, and basing the costs for shipping/disposal and for quality control (QC)/QA to be at a minimum of $300. The cost of sample processing and analysis being the simplest to predict is based on $150 for a discrete sample and $300 for a *MULTI INCREMENT* sample. The other two variables have a fixed initial fee, then increase based on either mass and the number of analyses. This simple analysis shows the potential for a 50% to >90% cost savings when using *MULTI INCREMENT* samples, depending on the DQOs. For this comparison both sampling strategies were held to the same DQOs. Therefore, for a low level of uncertainty, 30 or more discrete samples would be needed as compared to three or more 100-increment samples. It has been our experience that when a large number of discrete samples is required, the times to locate, record, decontaminate equipment, and complete the chain-of-custody forms for each sampling location negates the time and labor associated with increasing the number of increments from 30 to 100, for replicate *MULTI INCREMENT* samples. In contrast, for less stringent DQOs, the number of increments needed for each *MULTI INCREMENT* sample can be decreased as can the number of discrete samples. It should be recognized that the savings will be smaller on a project scale since, in many cases, the total cost associated with sampling and logistics, i.e., the first three subcategories in Table 19, are likely to rival or be greater than that associated with processing and analysis.

Table 18. Cost Tracking.

Procedure	Cost				
	30 Discrete	**100 Discrete**	**1 *MULTI INCREMENT***	**3 *MULTI INCREMENT***	**5 *MULTI INCREMENT***
Shipment / disposal	$300	$900	$300	$300	$600
Sample processing/ analysis	$4,500	$15,000	$300	$900	$1,500
Quality control/ assurance	$300	$750	$300	$300	$300
Total	$5,100	$16,650	$900	$1,500	$2,400

Table 19. Estimated Cost Comparison Between *MULTI INCREMENT* and Discrete Sampling Methods.

Cost Category	Subcategory	Ratio of Expense (*MULTI INCREMENT* vs. Discrete)
Field sampling	Gaining site assess	1
	EOD* personnel	1
	Travel and labor	1
Samples	Shipment/disposal**	$300/5 kg
	Sample processing/analysis	2
	QA/QC***	$300 or $150/batch

*EOD = explosive ordnance disposal
** Adjusted to mass—discrete 150 g; MULTI INCREMENT 1500 g, increasing at 0.5 kg increments
*** Based on $150 / batch or every 20 samples

8.2 COST DRIVERS

Following the publication of Method 8330B, the DoD EDQW issued guidance (see Section 2.1.4). This document presently requires that a blank be ground between each sample, and that each blank be analyzed. Requirements were also established for taking performance evaluation (PE) material through the sample handling protocols. Both these requirements and others are currently being debated between the EDQW and commercial laboratories as to their usefulness and practicality. The outcome of this debate could weigh heavily on the cost of performing the processing of samples for analysis by Method 8330B.

Several projects that had already received funds switched from the collection of discrete or wheel samples to *MULTI INCREMENT* samples. Since funding in many cases had already been established, there was little room for reallocations. To meet the budget constraints, typically half as many *MULTI INCREMENT* samples were collected as the originally planned number of wheel samples. Therefore, with no additional cost the area sampled and therefore also visually inspected increased by several orders of magnitude. An example of how the sampling plans transitioned between using a wheel and MIS strategies and designs is covered in a letter from USEPA Region 6 (available in Appendix F of the ER-0628 Final Report).

8.3 COST ANALYSIS

The purpose of this program was to demonstrate methods that produce scientifically defendable environmental data. To accomplish this goal, the entire sample must be either adequately processed or extracted in its entirety. To estimate a mean concentration or a 95% UCL of the mean for 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 this study. The expenses associated with gaining site access and engaging EOD support, travel, and labor are anticipated to be equivalent for these two sampling procedures (Table 19). Given that each discrete sample weighs 150 g and each *MULTI INCREMENT* sample weighs 1,500 g, it will mostly likely cost approximately twice as much to ship and dispose of the *MULTI INCREMENT* samples. 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 involves removing only 20 to 30 g from a field sample to perform this task. Indeed, costs may be

higher if performing whole discrete sample (150 g) extraction on a routine basis is required. To dry, sieve, and adequately pulverize each 1,500-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; therefore, 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 the guidance issued by the DoD EDQW (Guide for Implementing EPA SW-846 Method 8330B, Section 2.1.4), the QA/QC costs associated with a batch of 20 samples might increase twofold for Method 8330B. Currently, this guide 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 predetermined frequency.

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9.0 IMPLEMENTATION ISSUES

ERDC-CRREL has an open door policy to providing information and meeting the concerns of DoD agencies. Presently, we are engaged in an effort to support the ITRC in developing guidance for the implementation of MIS 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.

We believe that military, government 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 Air Force Base, MMR, and at Ravenna Army Depot. The states of Alaska and Hawaii have mandated it be used on several projects, and USEPA Region 6 has also made strong recommendations for its implementation on MMRP FUDS and other related projects.

The success of implementation of the MIS strategy for the energetic residues (Method 8330B) and other constituents has often gone hand-in-hand with more in-depth planning during the early stages of a project. In the case of developing sampling plans for some MMRP FUDS, the MIS strategy was selected over the collection of a few discrete samples based on the acknowledgement that it provided better site coverage and, therefore, a better opportunity to establish if energetic residues still existed. For projects that are in the remedial investigation phase, our experience has been that all parties must first reach a consensus on two aspects: 1) that the critical statistical parameter is the mean concentration and 2) how the sampling area is to be stratified based on the conceptual site model. Both these parameters often are in compliance with risk-based criterion. After these two parameters have been resolved for a remedial investigation, the only remaining variables are the number of increments per sample and the number and frequency of field replicate samples, which relates to the level of uncertainty that is desired. Therefore, going through the process of evaluating if the MIS strategy is appropriate for a given project has helped to reinforce the technical project planning and DQO process. However, there remains a lot of resistance to adopting the strategy since many of the concepts are new and therefore require broad distribution of information. As a consequence, in addition to the guidance now available from two state agencies (Hawaii and Alaska), the following agencies are in the process of developing additional MIS guidance: the U.S. Army Corp of Engineer Environmental and Munitions Center of Expertise, U.S. Department of the Navy, U.S. Army Environmental Command, and the ITRC.

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APPENDIX A

POINTS OF CONTACT

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APPENDIX B

ITRC PROJECT PROPOSAL: Multi-Increment Sampling (MIS)

PROPOSAL DATE: June 27, 2008

Please use brief statements or bullet items to input the requested information.

Proposal Contacts:

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Problem Statement (why is this project necessary?)

Quality control and reproducibility (i.e. scientific defensibility) are lacking in the sampling designs, strategies, and sample handling and processing protocols for environmental soil and sediment studies. Sampling theory and evidence indicate that the greatest bias and uncertainty in soil and sediment data quality is attributable to field sampling and laboratory sub-sampling, not laboratory instrumental error. Historically, quality control measures have focused on improving laboratory analytical procedures, resulting in very good reproducibility and QC of laboratory analyses. Quality control measures in field sampling design, strategy, and laboratory sub-sampling procedures have been neglected. The quality and information value of soil sampling data depends not only on laboratory analytical precision, but perhaps even to a greater degree on appropriate field sampling design and methodology to obtain scientifically defensible data that are relevant to the sampling objectives. Without the development and adoption of innovative sampling strategies that improve data reliability, the capabilities of modern laboratory measurement are underutilized, and progress toward cost-effective improvements in the reliability of site cleanup decisions is unlikely to be realized.

Conventional soil investigations typically rely on the collection of discrete surface soil samples from a site. Each sample is analyzed separately. The range of variability is often high and the reproducibility of duplicate samples notoriously poor due to the heterogeneous nature of soil (compositional heterogeneity) and the uneven distribution of contaminants across a site (distributional heterogeneity). Quite often, the results are compared to risk-based or regulatory values based on mean analyte concentration over a specific area (exposure unit).
A large number of discrete analyses (typically more than 30), and corresponding large cost, can address distributional heterogeneity to obtain a statistically useful data set and estimate mean concentrations, but discrete samples do not address error due to compositional heterogeneity. In an effort to minimize costs, the number of samples all too often is reduced below the number required to demonstrate good statistical reproducibility. Statistical uncertainty (e.g. UCL95) is unnecessarily high. Additionally, statements of sampling objectives and sampling plans often do not specify requirements that ensure proper sample coverage, collection and processing procedures, or relevance of the data to inform project decisions. Together these factors contribute to high cost, decision uncertainty, and can lead to missed contamination or unnecessary cleanup.

The recently published EPA Method 8330B for explosive compounds, based largely on results of SERDP and ESTCP-funded programs, recommends the use of Multi-increment Sampling (MIS) procedures in both the field sample collection and laboratory sub-sampling to address the aforementioned problems. The remarkably improved reproducibility of results demonstrated by the development and proper application of MIS, and the precedent of recommended field sampling procedures for an EPA SW846 Method, has spurred interest in the approach throughout the environmental community. The laboratory procedures of 8330B were developed for the evaluation of explosive and propellant compounds. The field sampling objectives address range sustainability issues at active military firing ranges. Thus, the specific protocols developed are not necessarily directly transferable to studies having other objectives or other analytes; however, the underlying principles are critical to the scientific defensibility of all environmental studies. Although the need for scientifically defensible (reproducible) soil and sediment data that meet sampling objectives at minimum cost is universal to soil investigations, guidance on the concepts underlying successful MIS design is not readily accessible.

Implementation of MIS has outpaced development of guidance and understanding of the underlying principles and concepts by the practitioners. A dozen or more state regulatory agencies are now requesting MIS for many DoD and other soil investigations, but only a handful have developed even limited guidance. Many MIS investigations and sampling plans are flawed and the data mis-directed or even misleading because of inadequate understanding of the fundamental requirements for correct application of the methodology. In some states, all composite sampling is perceived as diluting out “hot spots” or missing contamination. Some regulators have voiced concern that MIS loses spatial information.

Because of previous ill-conceived, uninformed, or mis-applied composite sampling, explicit regulatory barriers to its use exist. Deeply ingrained prejudice, misconceptions, and blanket prohibitions to "composite" sampling need to be overcome before MIS can be widely accepted. These barriers, biases, and misconceptions severely limit the use of one of the most cost-effective ways to improve environmental data quality.

Solution / Impact (how will the project impact the environmental marketplace?)

Multi-increment sampling (MIS) is a structured composite sample collection and processing approach designed to obtain a laboratory sub-sample for extraction having constituents in the same proportion as a specified area (volume) of soil or sediment about which a decision is to be made (i.e. the decision unit). The objective is to obtain a single sample for analysis that has a mean concentration

representative of the decision unit. Mean concentration underlies the basis of many environmental decision criteria (risk-based screening levels, soil concentrations protective of groundwater, etc.) and is the most appropriate value on which to base most environmental decisions. The decision unit must be purposefully delineated so that the mean analyte concentrations obtained are directly relevant to well defined and explicitly articulated sampling objectives, including those investigating the spatial distribution of contaminants. Using MIS, reliable estimates of mean concentration for a specified area of virtually any size can be obtained from analysis of a single sample. Reproducibility between properly collected replicate MI samples is good (RSD/RPD <30 percent have been achieved). Data distribution of replicate samples tends to be normal (as opposed to positively skewed for discrete samples). Fewer non-detect results are obtained, thus mitigating the problems caused by censored data sets and lessening the chance of missing significant contamination (as defined in project DQOs). Levels of statistical confidence (UCL95) and decision uncertainty that would require a large number of discrete analyses often can be obtained with a few MI samples.

By developing guidance for the appropriate implementation of MIS to a wide range of sampling objectives, analytes, and circumstances, the project will improve data quality and reduce characterization costs at many sites where soil and sediment data are collected. The project will provide users with a practical working knowledge of the concepts and principles of the methodology, emphasize the critical importance of clearly articulated sampling objectives, and provide the sound basis for adapting the MIS approach to meet project goals and site-specific objectives. Sound guidance will help avoid misapplication and pitfalls of the approach, and correct misperceptions to overcome existing regulatory barriers and bias against well-conceived and properly applied composite sampling.

The project will be sharply focused on the MIS sampling methodology. It will capitalize on the successful demonstration/validation studies conducted by USACE/CRREL by developing broad guidance for implementation of MIS to a full range of analytes and a broad range of sampling objectives, and will parallel rapidly growing capability of commercial laboratories to meet the expanding use of the method.

Success Measures (how you determine the project impact to the market place)

- State concurrence with the Technical and Regulatory Guidance document will be an important measure of success. MIS is being applied in at least ten states, yet only two (AK and HI) are known have published even limited guidance. Because of the nearly universal need for improved soil and sediment data at reduced cost, the guidance is highly relevant. The number of states using MIS is growing rapidly, little guidance exists, and thus a high level of concurrence with sound ITRC guidance might be anticipated. The challenge of overcoming regulatory barriers to composite sampling should not be underestimated by the team, however an increasing number of states are requesting, and even requiring the use of MIS in some instances.
- Because soil and sediment sampling is a fundamental component of so many environmental investigations, participation in a long-running internet training, which is easily tracked, can be expected to be high.
- Case studies showing demonstrated cost savings by improved systematic planning a use of MIS of soil investigations to obtain soil data supporting project decisions will be tracked to the degree possible.
- Use of ITRC MIS Technical and Regulatory Guidance in conjunction with, or in lieu of state or other guidance will be tracked to the extent possible. DoD representation and established networking of team members within the community of practice should provide success stories on DoD facilities.

Summary of Deliverables (primary project outputs)

- To begin addressing the need for sound information on MIS, example posters and fact sheets developed for presentations and meetings by team members working on MIS over the last few years will be adapted for ITRC use, and made available on the web through the team page.
- "An Overview of Multi-increment Sampling: pitfalls, and potential" will be made available as soon as possible to fill the pressing need for information on MIS recognized throughout the community of practice. Existing case studies, both good examples and "lessons learned", will be presented. Cost information is available for some. The overview will include comprehensive references and be resource for information on the existing body of knowledge. If timing permits, results of a survey on the regulatory status and extent of use of MIS will be included.
- The primary product will be the ITRC "Technical and Regulatory Guidance for the Implementation and Use of Multi-Increment Soil Sampling". The document will begin with discussion of heterogeneity – the primary source of error (variability) in soil sampling data, and the ways in which MIS effectively mitigates the difficulties posed by heterogeneity. This will be followed by guidance on formulating quantitative sampling objectives, focusing on sample "effectiveness" and data relevance to the objectives, and the use of MIS to meet those objectives. It will include critical discussion on the underlying basis of common decision criteria against which soil data are evaluated, and development of explicit DQOs not just for laboratory QC, but also for end use of the data and quantifying ultimate decision uncertainty. Considerations for three-dimensional applications (sub-surface soil) will be included, and analyte and method-specific collection, handling, and laboratory processing and sub-sampling requirements also will be addressed. I short, all of the considerations necessary to guide sampling design and execution for a successful MIS project will be covered.
- Internet-based training based on the Tech-Reg and of the same title will be developed concurrently with the Tech-Reg document.

Presentations and training on MIS as applied to munitions constituents at DoD sites, already are being given at meetings and conferences by team members. These will continue and expand to include other constituents and the full spectrum of objectives and analytes as work progresses.

Project Schedule

Because the scope of the project is well defined, and because key participants and subject matter experts already developing MIS techniques and guidance are willing to participate, a delivery time frame of 24 months to the implementation phase of the project life cycle is anticipated.

2009

- Early 2009 – 1st team meeting/kickoff. Consolidate team knowledge base. Many identified team members already have substantial knowledge and materials on the subject. Because MIS is relatively new, research is ongoing (some by team members), and new information continues to be gained at a rapid rate, team knowledge will continue to increase throughout the project life-cycle. Develop detailed project work plan; begin tracking and progress reports to Board Team Leader Liaison.
- Early 2009 – Adapt existing posters and fact sheets to ITRC format and audience for posting on ITRC team web site. Design state regulator survey.
- Early to mid 2009 – Conduct survey of the state regulatory status and concerns that might prohibit or impede the use of properly designed MIS for appropriate objectives. Regulatory barriers and impediments, and their origin and basis, will be particularly addressed in team documents. Collect and review case studies.
- Early to Mid-2009 – 2nd team meeting (May?) Organize and analyze results of survey. Develop outline and assign portions of the overview document to various co-authors.
- Late 2009 – Fall meeting (3rd team meeting) – Draft final overview document ready for courtesy review and start work on the Tech-Reg.
- Dec 2009 or Jan 2010 – Deliver final overview document.

2010

- Early to mid 2010 - Continue Tech-Reg guidance document and outline IBT.
- 4th team meeting (Feb?) – continue work on Tech-Reg. Begin slides for IBT.
- Mid 2010 – 5th team meeting (May/June?) – finalize Draft Tech-Reg for POC / DoD review
- Mid to late- 2010 Train-the-trainer internet training practice
- Late 2010 – Fall Meeting (6th team meeting)
- End 2010 - Deliver Final Tech- Reg document for publication and POC internet training dry run.

2011

- Implementation phase. Close-out and implementation strategy meeting 7th team meeting. Actively pursue concurrence from states and evaluate success of project. Continue internet training as demand warrants.

Target Audience

The primary target audience is twofold:

- state and federal regulators, project managers, and consultant personnel who are responsible for and/or directly involved in identifying soil and sediment sampling objectives and methodologies. This includes policy makers and data end users (e.g. risk assessors). The work products will be useful for developing technical and regulatory decision processes without specialized technical skill sets.

- personnel directly responsible for developing, implementing, and overseeing specific sampling strategies and field sampling plans to meet those objectives. Specific examples and case studies will be provided.

Secondary audiences will be

- field personnel who should have an understanding of the basic concepts underlying activities they are performing to recognize potential problems that may arise during sampling, and

- decision makers/upper management personnel and stakeholders who should have an understanding of the nature and quality of the soil and sediment data upon which environmental decisions are based.

Resources Required

Personnel: Note: A team leader from a state regulatory agency has not yet been identified.

State Representatives

Email confirmation of intent to participate, and management support from:

- Roger Brewer, Hawaii Dept. of Health
- Earl Crapps, Alaska Dept of Environmental Conservation
- Jeffrey E. Patterson, Texas Commission on Environmental Quality
- Jeanene Hanley, Arizona Dept of Environmental Quality

Verbal expression of interest in participation and likely management support:

- Matt Thomas, Alabama Dept of Environmental Management
- Craig Kleinhenz, Ohio EPA, Criminal Investigations

Industry Affiliates

Email confirmation of interest in participation and willingness to join IAP:

- Larry Penfold, Federal Program Quality Manger, TestAmerica, Inc. – Denver
- Mark Bruce, Technical Director, TestAmerica, Inc. – North Canton. OH

DoD

Email confirmation of intent to participate from:

- Alan D. Hewitt, USACE/CRREL
- Edward J. Brown, Environmental Chemist, AFCEE
- Keith Hoddinott, Risk Assessor, US Army CHPPM

Intent for Navy participation from:

- Ed Hartzog, Director, Laboratory Quality and Accreditation Office NAVSEA Programs; Chairperson, Tri-Services Environmental Data Quality Workgroup (EDQW)
- Jordan Adelson, NAVSEA Laboratory Quality and Accreditation Office
- Ed Corl, Chemist, EDQW

EPA

Verbal confirmation of participation and management support:

- John Warren, statistician, USEPA (HQ), Quality Staff, Office of Environmental Information

Current members of the ITRC Risk Assessment Team, UXO Team, and Sampling, Characterization and Monitoring Team have expressed interest and may be expected to in participate on this proposed project if it is approved.

Financial Resources:

Year	Team Travel	Contractor Support	Printing/Calls	Total
2009	$ 22,000	$ 60,000	$ 12,000	$ 90,000
2010	$ 22,000	$ 60,000	$ 12,000	$ 90,000
2011	$ 9,000	$ 12,000	$ 1,000	$ 31,000
Total	$ 53,000	$ 132,000	$ 25,000	**$ 210,000**

Related Work:

The MI sampling approach is relevant to projects and work products of the ITRC Risk Assessment Team; UXO Team; and Contaminated Sediments Team. MIS products would be integrated or referenced by each of these teams. The broad range of environmental applications for MIS, its recent and rapid emergence, and the in-depth treatment and expertise that comprehensive guidance will require, have been beyond the scope of the individual teams. Several current members of those teams have expressed interest in participating and contributing to guidance development from their perspective for MIS.

Other identified ITRC priorities for 2009 related to MIS include:

- Remediation Risk Management Team —Risk-based decision making for land re-use.
- Soils Risk Management – upland soil contamination and risk based clean up objectives.

The concurrent ITRC proposal “A Guide to Munitions Constituents Regulatory Status, Sampling, and Treatment” has some overlap with this proposal. Proponents have discussed coordination on subject matter common to these complementary projects, and possibly having team members in common to maximize benefit from both team efforts. The full range of applications for multi-increment sampling has broader requirements, and warrants more in-depth treatment than the overview discussion now planned for the munitions constituents guidance.

Current ESTCP proposals 09 E-ER1-009 "Demonstration of the Attributes of Multi-Increment Sampling and Proper Sample Processing Protocols for the Characterization of Metals on DoD Facilities" (Alan D. Hewitt).
Note: Proponent intends to participate on this ITRC project if it goes forward.

The proposed MIS project will build directly on work successfully performed under ESTCP Project ER-0628 “Characterization of Energetic Residues on Military Training Ranges” (Alan D. Hewitt), and SERDP Projects ER-1155 and 1481.

The Tri-Services Environmental Data Quality Workgroup is developing “Guide for Implementing EPA SW-846 Method 8330B”. Document discusses considerations in the use of 8330B and application of MIS to other contaminants and applications. EDQW (Navy) intends to participate on this ITRC project if it goes forward.

In response to requests and requirements by a growing number of state regulatory agencies to implement MIS, the Army Corps of Engineers is currently developing an interim guidance for the implementation of MIS in at Formerly Used Defense Sites (FUDS).

Update of the EPA Handbook on the Management of Munitions Response Action EPA 505-B-01-001, May 2005, (interim final) has just begun. Among the updates is inclusion of MIS methodology based on the USACE/CRREL work, but it will not involve in-depth discussion of MIS principles and concepts necessary to adapt the technique to a broad spectrum of environmental investigations. Proposed team members are involved and EPA Federal Facilities personnel may become involved.

A draft EPA Federal Facilities Forum Issue Paper tentatively titled “Sampling Design and Analysis of Munitions Residues on Ranges and Open Burn/Open Detonation Units” has been in development since 2006.

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