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Abstract: In the past, very little guidance has been available for site characterization activities addressing the concentration and mass of energetic residues in military training range soils. Energetic residues are heterogeneously distributed over military training ranges as particles of various sizes, shapes, and compositions. Most energetic residues are deposited on the surface, and the highest concentrations exist at firing positions, near targets, and where demolition activities are performed. In the case of impact and demolition ranges the greatest quantities of residues are from rounds that fail to detonate as designed. To address the compositional and distributional heterogeneity associated with the distribution of particles and to obtain representative mean energetic residue soil concentrations, the sampling strategy must strive for the acquisition of samples that contain the constituents of concern in the same proportion to the bulk matrix as exists within the decision unit (sampled area, population, or exposure unit). This report summarizes the sampling strategies and designs that have been implemented for various types of military ranges, including hand grenade, antitank rocket, artillery, bombing, and demolition ranges. These protocols were developed during investigations on active ranges and primarily addressed potential surface source zones from which energetic residues could be migrating into surface and groundwater systems. A multi-increment sampling strategy was selected to accomplish this task after exposing the inadequacies of discrete sampling.

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Preface

This report was prepared by Alan D. Hewitt, Dr. Thomas F. Jenkins, and Marianne E. Walsh, all of the Environmental Sciences Branch (ESB), Cold Regions Research and Engineering Laboratory (CRREL), U.S. Army Engineer Research and Development Center (ERDC), Hanover, NH; Michael R. Walsh, Engineering Resources Branch, CRREL-ERDC; Susan R. Bigl, ESB, CRREL-ERDC; and Charles A. Ramsey, Envirostat, Inc., Fort Collins, CO.

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The Commander and Executive Director of ERDC is COL Richard B. Jenkins. The Director is Dr. James R. Houston.

Acronyms, Definitions, and Compositions

Composition A5	98% RDX, 2% wax
Composition B	60% RDX, 39% TNT, 1% wax (referred to as Comp B)
Composition C4	91% RDX, 9% oil (referred to as C4)
2ADNT	2-amino-4,6-dinitrotoluene
4ADNT	4-amino-2,6-dinitrotoluene
2,4-DNT	2,4-dinitrotoluene
CRREL	Cold Regions Research and Engineering Laboratory
CSM	Conceptual site model
DMM	Discarded military munitions
DoD	U.S. Department of Defense
DQO	Data quality objective
EOD	Explosive ordnance disposal
ERDC	Engineer Research and Development Center
ESTCP	Environmental Security Technology Certification Program
H6	RDX, TNT, aluminum
HC	Hexachlorane
HEP	High explosive plastic
HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HTRW	Hazardous, toxic, and radioactive waste
LAW	Light anti-armor weapon
MC	Munitions constituents
MEC	Munition and explosives of concern
NC	Nitrocellulose
NG	Nitroglycerin
NQ	Nitroguanidine

OB/OD	Open burning / open detonation
Octol	70% HMX, 30% TNT
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine
RSD	Relative standard deviation
SERDP	Strategic Environmental Research and Development Program
Tetryl	Methyl-2,4,6-trinitrophenyl nitramine
TNB	1,3,5-trinitrobenzene
TNT	2,4,6-trinitrotoluene
TPP	Technical project planning
Tritonal	80% TNT, 20% aluminum
USACE	U.S. Army Corps of Engineers
USAEC	U.S. Army Environmental Center
USEPA	U.S. Environmental Protection Agency
UXO	Unexploded ordnance
WP	White phosphorus

1 Introduction

Currently, characterization of energetic residues on firing ranges is heavily dependent on the sampling and analysis plans that have been adopted by different branches of the government. A growing concern within the U.S. Environmental Protection Agency (USEPA), the Department of Defense's Strategic Environmental Research and Development Program (SERDP) executive board, and the U. S. Army Engineer Research and Development Center, Cold Regions Research and Engineering Laboratory (ERDC-CRREL) is that many of these sampling and analysis plans fail to acquire the appropriate information needed to address potential risks to humans and the environment. For example, many firing range characterization studies have relied, and continue to rely, on discrete samples or a sample comprising five or fewer increments. In many cases, these samples are mixed and split in the field before being shipped to a laboratory. Because the current guidelines in Methods 8330 and 8095 do not specify that the entire sample be processed, laboratories often process and analyze only a small portion of this already split sample. These practices yield samples that can underestimate or fail to detect the energetic residues present and are not repeatable, i.e. they have a large amount of uncertainty (Jenkins et al. 2005a, b).

SERDP, the U. S. Army Environmental Center (USAEC), the U.S. Garrison Army Alaska, and the U.S. Army Corps of Engineers Distributed Source Program have supported research to investigate the mass loading and fate of energetic munitions constituents on military live-fire training and testing ranges. Specific goals of this research included the following:

- Identify the concentrations and distribution of energetic residues present in surface soils at various types of military live-fire training ranges;
- Evaluate the mass of residues deposited from live-fire, blow-in-place, and low-order detonations of munitions such as hand grenades, mortars, and artillery rounds;
- Evaluate sampling strategies for collecting representative surface soil samples to enable estimation of source zone concentrations and masses of common energetic munition constituents; and

- Evaluate sample processing and analysis protocols to enable accurate and precise laboratory determination of these constituents.

The subsequent knowledge gained from these activities is integral to the development of the conceptual site model (CSM) for training range characterization. This document is intended to help promulgate guidance for sampling activities associated with characterizing the surface loading of energetic residues on military training ranges.

This report summarizes the sampling strategies and designs that have been implemented for various types of military ranges, including hand grenade, antitank rocket, artillery, bombing, and demolition ranges. These protocols were developed during investigations on active ranges and primarily addressed potential surface source zones from which energetic residues could be migrating into surface and groundwater systems. A multi-increment sampling strategy was selected to accomplish this task after exposing the inadequacies of discrete sampling. This sampling guidance should complement existing Department of Defense (DoD) and USEPA programs challenged with determining if military training and testing facilities present risks to human health and the environment. More specifically, this information will aid in the development of data collection activities during technical project planning (TPP) involved with establishing the existence and amount of residual energetic munitions constituents (MCs) resulting from training and testing activities. Energetic MCs (energetic residues) can be a risk to human health and the environment and often are treated as other hazardous, toxic, and radioactive waste (HTRW). However, because energetic residues often are coincident with munitions and explosives of concern (MECs) that may exist at levels presenting immediate detonation or deflagration hazards, special precautions and protocols should be invoked during sampling.

This document recommends use of a multi-increment sampling strategy with a systematic random (random grid) sampling design to obtain a sample or replicate samples of approximately 1 kg mass to characterize the average concentration of MCs within a chosen decision unit. The entire sample should be thoroughly pulverized and mixed so as to minimize subsampling variability. This approach is dramatically different from the collection of discrete samples and the commonly used practice of field splitting or laboratory subsampling by removing only a portion of the sample received from the field for further processing. Moreover, collection of discrete samples failed to meet the objective of the Environmental Security Testing Certification Program (ESTCP) Environmental Restoration Project ER-0628, which is to establish an economical approach for providing scientifically defendable environmental characterization.

2 Purpose

This document is intended to assist with planning sampling activities during the technical project planning process for the characterization of MCs on operational and non-operational military training ranges, under the Sustainable Range Program and the Military Munitions Response Program. It is recognized that some of the ranges covered under these programs have been inactive for more than five decades and that, in many cases, the formulations of the munitions fillers have changed. In addition, most munitions can contain a variety of fillers: high explosives, smoke, incendiary materials, and inert materials. The guidance provided here was developed for high explosives. It is anticipated that the dispersion mechanisms, the areas most heavily influenced, and the relevant environmental media are most likely very similar for all MCs. For these reasons the descriptions of the ranges and the rationale provided for the sampling strategy and for the range specific sampling designs provided in this text should be considered when addressing the questions "Are energetic residues present?" and if so, "At what average concentration do they exist in areas that historically have been influenced by training activities?" In addition to this document, several others should also be considered when developing a sampling plan (e.g., USEPA 2002, 2006a, USACE 1998, 2003).

3 Background

General Sample Support

Energetic residues accumulate on DoD training ranges as particles of either pure compounds or mixtures of explosive compounds and as fibers and particles of propellants and rocket fuels. High concentrations of energetic residue particles are typically found at operational firing points, sites where munitions have undergone a low-order (partial) detonation or have ruptured (breached upon impact or by sympathetic detonations), where demolition activities have occurred frequently, and sometimes where unexploded ordnance (UXO) has been blown-in-place on impact ranges. Figure 1 shows unconsumed particles of 2,4,6-trinitrotoluene (TNT) following the blow-in-place detonation of a 155-mm howitzer round with a block of Composition C4 (C4) and fibers that accumulated on the snow in front of a gun where the M1 propellant was used to accelerate 105-mm howitzer projectiles.

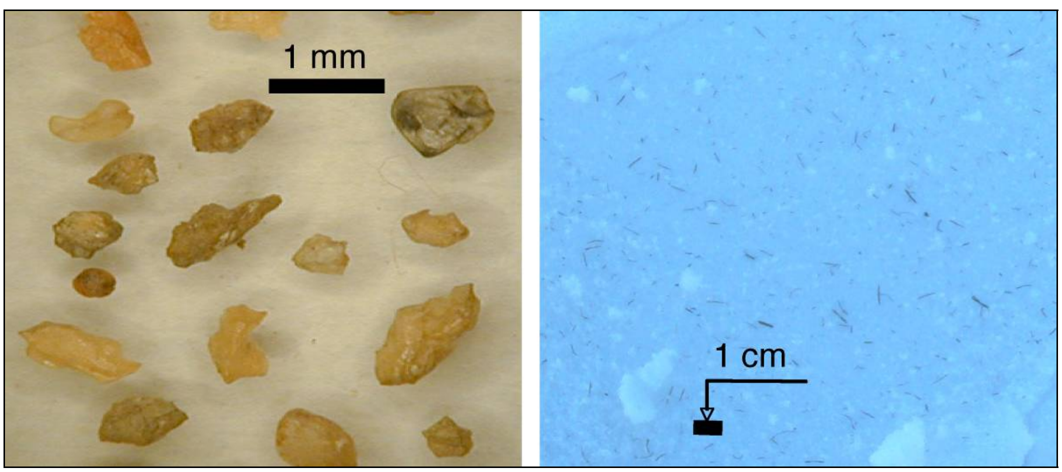


Figure 1. Examples of energetic material particles: TNT particles (<1 mm, fraction) from a blow-in-place detonation (left), 105-mm howitzer propellant fibers on a snow surface (right).

The chemicals in these energetic residue particles have low vapor pressures. Therefore, the principal mechanisms that determine the fate of these chemicals include dissolution and leaching, transformation, and, for some, chemical mineralization. Figure 2 shows concentration profiles of energetic residues obtained directly beneath chunks (> 2 cm) of explosives found on the surface. Concentrations of energetic residues in the surface soil sample (often discolored) immediately beneath the chunks were a consequence of small (< 1 mm) particles washed off or abraded from the surface. With increasing depth the concentration is due to migration of dissolved energetic analytes. The inherently lower concentrations of the subsurface samples result from a combination of limited solubility and limited volumetric soil moisture content. A large decrease in energetic residue concentrations with profile depth is also characteristic of firing point locations. Therefore, with the exception of ranges where the surface is physically moved and particles become buried, the highest concentrations are present near the ground surface on operational ranges (Jenkins et al. 2006a, Hewitt et al. 2005a). Generally, energetic residue particles are within the top 10 cm; in some cases, the vast majority is in the top 2.5 cm. Once the energetic residue particles have been completely dissolved, it is unlikely that they will remain detectable in surface soils for more than a couple years. That is, once energetic residues are no longer present in solid form, they degrade or migrate away from the original source area.

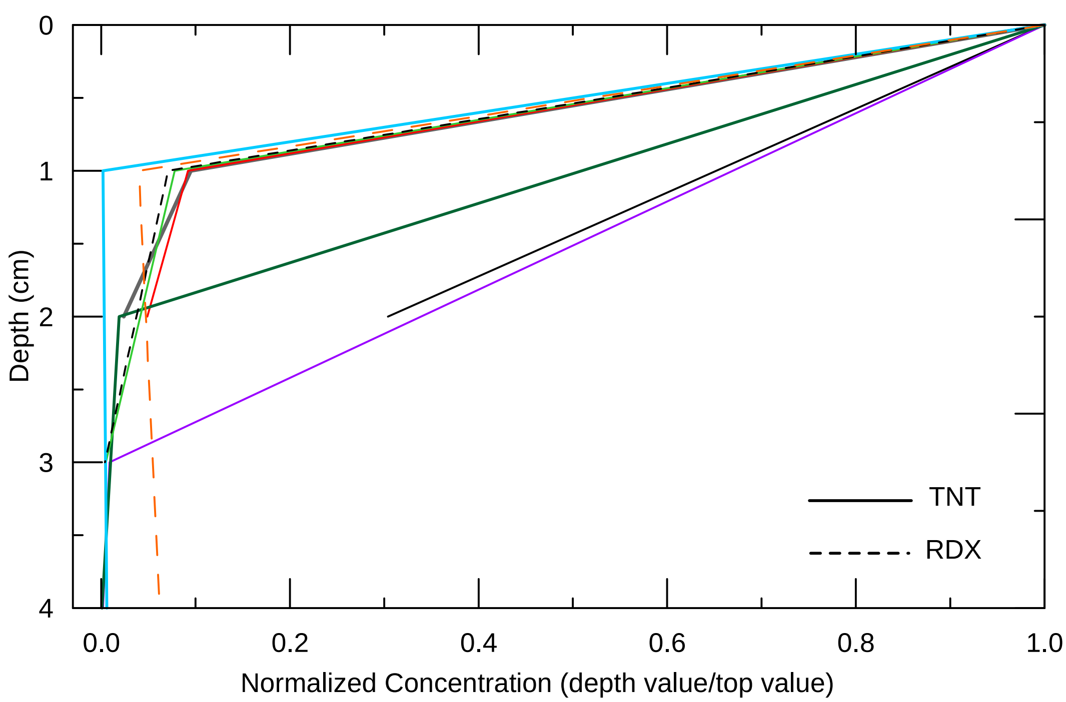


Figure 2. Normalized concentration profiles for TNT (solid lines) and RDX (broken lines). Profiles show a decreasing trend of these two energetic residues with depth directly beneath chunks (> 2 cm) of explosives found on the surface.

Because the greatest quantities of energetic residue particles exist near the ground surface, removal of surface vegetative cover (short grasses and mosses) is not recommended prior to sample collection on operational ranges. Figure 3 shows examples of vegetation present at a firing point and surrounding a crater formed by the low-order detonation of an 81-mm mortar on an artillery impact range. If vegetation is removed or patches of vegetation are avoided, any energetic residues trapped within this portion of the surface matrix will not be included in the sample, and the analyzed amount of energetic residue at a location is likely to be underestimated. The use of specially designed (Fig. 4) (Walsh, M.R. 2004) or commercially available coring tools at vegetated sites aid in collecting surface samples with minimal surface disturbance and human effort. Most importantly, the use of coring tools helps avoid biased sampling, i.e., sampling only the exposed soil surfaces. In addition, this type of sampling tool enhances the surface area and increment volume precision. With the exception of very thick vegetative mats, vegetation from the surface interface included with a soil sample typically makes up less than 1% of the total dry sample weight.

Figure 3. Examples of surface vegetation at a firing point (inset) and in and around a crater of an 81-mm mortar low-order detonation crater on an artillery impact range.

Figure 4. Coring tool designed specifically for collecting multi-increment cohesive soil samples.

Sampling Design

Sampling Theory

Representative sampling must be a major project objective (USEPA 2002, 2003, D.M. Crumbling, personal communication). To do so, the sampling strategy must address the compositional and distribution heterogeneity of the constituents of concern (Pitard 1993). Compositional heterogeneity occurs because not all soil-sized particles within the population have the same concentration of target analytes. This heterogeneity is at a maximum when a portion of the target analytes is present as discrete particles. The error caused by compositional heterogeneity is called the fundamental error and is inversely related to the sample mass. Distributional heterogeneity occurs because contaminant particles are scattered across the site unevenly, sometimes with a systematic component as well as a short-range random component. The error associated with distributional heterogeneity is inversely related to the number of individual increments used to build the sample. This type of error is at a maximum when a single discrete sample is used to estimate the mean for a larger decision unit. (Examples of larger decision units are populations, areas of concern, and ecological habitats.) To reduce the influence of distributional heterogeneity error sources in the estimate of the mean concentration for a decision unit, the collection of 30 or more evenly spaced increments to form an individual sample has been recommended (Jenkins et al. 2004a,b, 2005c, 2006a, Walsh, M.E. et al. 2005, Hewitt et al. 2005a). The objective of this multi-increment sampling strategy and systematic random design is to obtain an amount of energetic residue particles (<2 mm) of every composition (e.g. Tritonal, Composition B, H6) and shape (e.g. crystalline spheres or elongated fibers) that is proportional to what exists within the selected decision unit and not to oversample or miss any portion of the decision unit.

In the past, the estimate of mean concentration for a decision unit has often been derived from the collection and analysis of several discrete samples. Studies comparing both of these sampling strategies for the characterization of military training activities have shown that the distribution of data obtained from discrete samples is always non-Gaussian and positively skewed, whereas that from a multi-increment data set is often normally distributed (Jenkins et al. 2004a,b, 2005c, 2006a, Walsh, M.E. et al. 2005), a result consistent with the central limit theorem of statistics. Moreover, a single discrete sample or small set of discrete samples almost always results in a lower estimate of the mean concentration than the multi-increment sampling strategy. As the number of discrete samples collected approaches the number of increments in the multi-increment sample, the difference between the estimates of mean concentrations resulting from these two strategies merges, but the variability among values for the estimate of the mean for replicate multi-increment samples is always much smaller.

Uncertainty

The best way to estimate the total measurement error in the characterization process is to collect and analyze replicate field samples (Appendix A). The total measurement error calculated from these replicates includes contributions from sample collection, sample processing, and analytical determination. It must be emphasized that these are not field splits; rather, they are independently collected samples from within the exposure unit. We recommend that triplicate samples be collected for a percentage of the total multi-increment samples collected for a given characterization activity, the actual percentage being determined on a site-specific basis depending on the data quality objectives. The standard deviation (variance) computed from these triplicates often can be used to compute an upper 95% confidence limit for the mean concentration within a decision unit.

The ability to achieve low sampling error depends on the sampling strategy and the military training activity under investigation. In general, the more repetitious a given activity (e.g., projectiles fired or detonations occurring in the same general location), the more likely the distribution of energetic residues will become more pronounced (heavier accumulation) and uniformly distributed. As a consequence, sampling uncertainty is likely to be lower at sites such as a fixed firing position, near a direct line-of-sight target, and a demolition range than at sites around a target or former target on an indirect fire impact range. Studies at firing points and within impact ranges have supported this anticipated trend and have shown that analyte variability is much greater for a large set of discrete samples (n = 33) than for a small set (n = 3) of replicate 33-increment samples (Jenkins et al. 2004a,b, 2005c, 2006a, Walsh, M.E. et al. 2005). This is a common characteristic of analytes that are heterogeneously distributed as particles. For many environmental programs, this source of uncertainty (i.e., determining if the sampling design and strategy result in representative samples as inferred from the ability to reproduce the sampling results) has often been ignored. This is particularly alarming in light of studies showing sampling error to be the largest portion of the total characterization uncertainty for energetic residues on military training sites (Jenkins et al. 1997a,b, 1999). Therefore, both scientific (data quality) and economic advantages can be realized through the processing and analysis of multi-increment samples.

Sampling Decision Unit

In many cases the size of the sampling decision unit can correspond to the entire area where it is anticipated that the greatest amount of energetic residues have accumulated. The appropriateness of larger decision units is based on the dispersion of energetic residues around guns and live-fire and blow-in-place detonations (Hewitt et al. 2005b, Walsh, M.R. et al. 2005a,b,c, 2006). These studies determined that energetic residues are spread over large areas, typically on the order of hundreds of square meters. Additional considerations are the total size of the area influenced by the activity and what constitutes a manageable sample for field and laboratory operations, without compromising data quality. These parameters, coupled with range use records, range function and design, surface conditions, and the data quality objectives, should all be considered when deciding where to sample and the size of the decision unit. In some cases the area impacted by an activity is so large that it must be divided into multiple decision units.

Visual Observations and Field Screening

Additional considerations and special precautions should be invoked when sampling around low-order detonations and ruptured munitions, both of which often fall under the classification of MECs. First, the size of the decision unit should at least address the area covered with residues. This often is subjective, based solely on visual evidence. Because areas covered with visible pieces of energetic residues are likely to be over hot spots with high energetic residue soil concentrations, these areas are candidate source zones for surface and ground water migration pathways. Chunk residues (pieces of energetic materials > 2 cm) often are present within and around ruptured (low-ordered or breached) munitions and in areas that have been used for open burning / open detonation (OB/OD) of off-specification, obsolete, or excess energetic materials. Field analytical screening techniques should be used to identify chunks of energetic residues. Methods approved by the USEPA include colorimetric SW-846 Methods 8510 and 8515 and immunoassay Methods 4050 and 4051 (USEPA, 1996a,b,c, 2000). Other screening techniques, such as use of the Expray™ kit, may be used for identification purposes (Plexus Scientific, Silver Spring, MD) (Bjella 2005). Once identified, chunks of energetic materials should be gathered, weighed (if not adhering to a munitions casing), and removed by EOD personnel or UXO technicians prior to sampling. Additional information regarding residue identification and the safety concerns are presented in Method 8330B (USEPA 2006).

Systematic Random Sampling

We recommend using a systematic-random sampling design when collecting individual increments to build each sample (Hewitt et al. 2005b). This sampling design is analogous to systematic grid sampling (USEPA 2002), where the starting location is chosen randomly and the remaining sampling locations are laid out in a regular pattern (Cressie 1993). To use this approach, the sampler begins at a point on the edge of the area to be characterized and collects an increment of surface soil after a predetermined number of steps, while walking back and forth in a systematic manner across the area of interest. Figures 5 and 6 provide examples of the serpentine path a sampler would take using this sampling

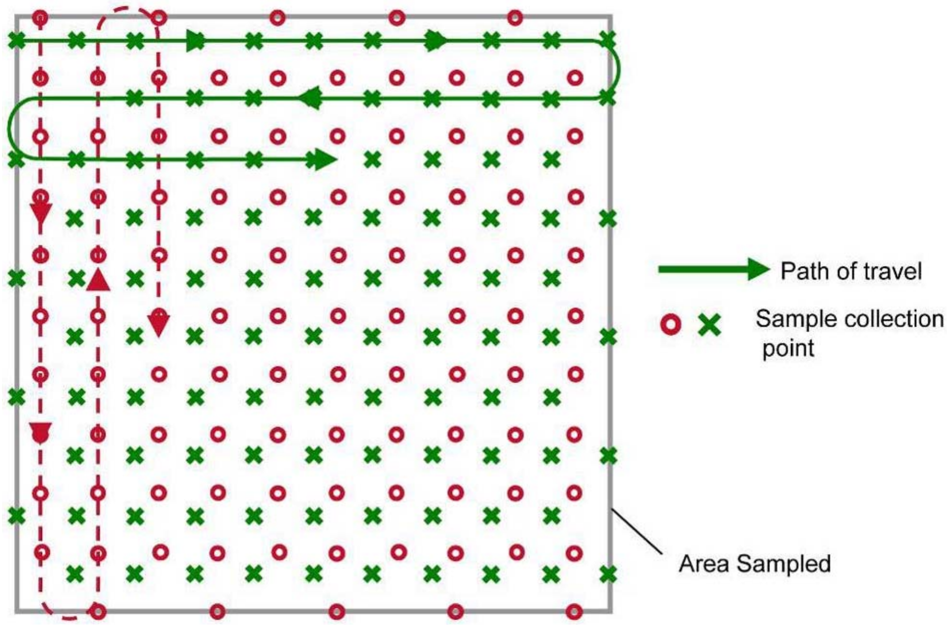


Figure 5. Systematic-random 100-increment sampling pattern used for collecting samples in grid areas.

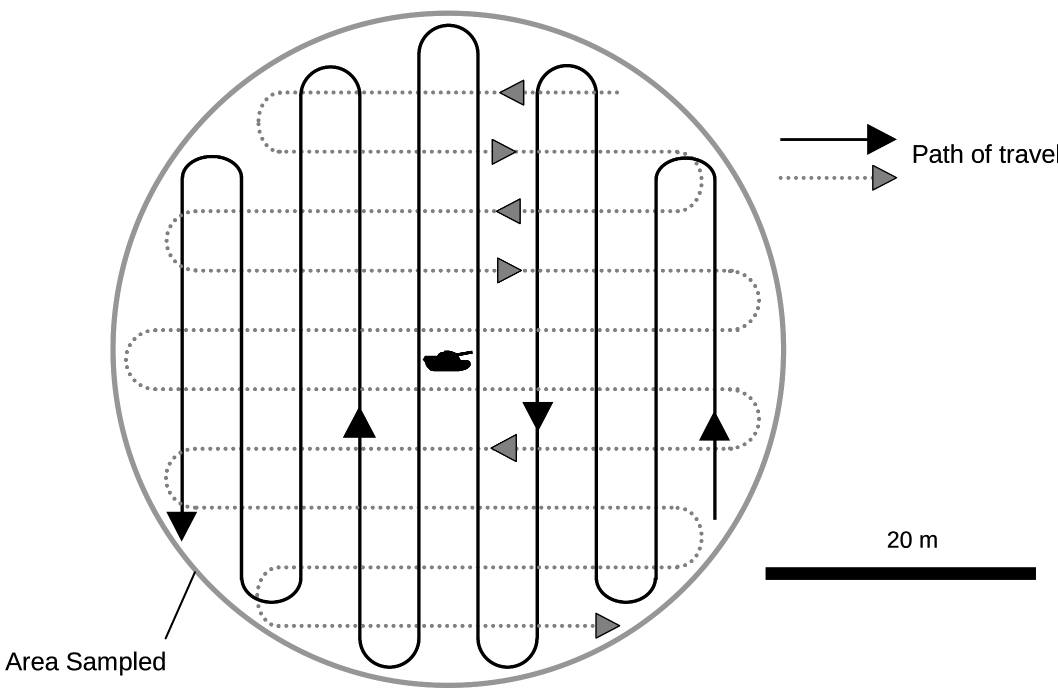


Figure 6. Systematic-random multi-increment sampling design surrounding a tank target at the impact area of an anti-tank range.

design and strategy for square and circular areas. The proper number of steps between locations where an increment is collected to obtain a representative (reproducible) sample is a function of the compositional and distributional heterogeneity. The number of increments and the size of the decision units cited for the range-sampling activities described below have often produced replicate samples with similar analyte concentrations. The assumption that the distribution of energetic residues is similar at other military facilities with ranges designed for the same activity is the basis for the recommended sizes of decision units and number of increments. Because increments are being combined to create a single sample, cleaning the sampling tool between collection increments of a given sample is unnecessary. A clean sampling tool is necessary for each new sample, including replicate samples. In addition, each replicate sample should be obtained starting from a different location and following a different serpentine path direction (Fig. 5 and 6). To be random, the increments obtained from evenly spaced locations through the decision unit in each replicate should not be co-located with the increments obtained for one of the other replicates. Additional guidance on the sampling strategy and design is provided in Appendix A.

Sample Processing

The typical weight of multi-increment samples collected with the sampling designs and strategies described above and in the following sections are 1 kg or greater. Recently, Method 8330 was revised and Method 8330B was published by the USEPA (USEPA 2006). This revised method provides laboratories with guidance on how to handle and process soil samples so that they can be representatively subsampled in preparation for analysis. Several studies cited in the revised method have shown that, to determine representative analyte concentrations in soils containing energetic residues, laboratories must either grind the samples mechanically prior to subsampling or extract the entire sample. Following the guidance in Method 8330B, the results for laboratory replicate subsamples have been shown to be both reproducible and experimentally accurate (method established accuracy), since in a few cases, the remaining sample was extracted and analyzed to produce a known concentration.

4 General Guidelines

Multi-increment Sampling

A multi-increment sampling strategy and a systematic-random sampling design are recommended for all the military training ranges addressed in this document. In addition, collecting triplicate multi-increment samples is strongly recommended for at least one decision unit on each type of training range under investigation. To aid in collecting multi-increment samples with a targeted weight of approximately 1 kg, special sampling tools may need to be acquired so as to obtain the appropriate incremental mass relative to the recommended number of increments and sampling depth (Appendix A). These coring tools, shown in Figure 4, are made with 2- and 3-cm inner diameters to help meet these needs; although they are not currently commercially available, they may be in the near future. Oakfield corers or similar push tube devices are soil sampling tools available in several core barrel widths and lengths. These soil-coring tools are easy to operate in cohesive soils. However, they are not practical for some cobbled and non-cohesive soils. Metal or hardened plastic scoops and trowels are more suited for use in cobble-rich and non-cohesive (sandy) soils. Both of these soil-sampling tools are available from equipment vendors such as Forestry Suppliers, Inc. (www.forestry-suppliers.com), EnviroTech (www.envirotechonline.com), and Ben Meadows Company (www.benmeadows.com).

It also should be noted that the guidance provided here also applies to the surfaces of other ranges that are not specifically addressed in this document but are operationally similar. For example, on direct-line-of-sight ranges, the areas anticipated to have the highest accumulation of munitions constituents would be at the firing point and around targets.

Health and Safety

Sample-collecting activities must occur only in the presence of military EOD personnel or qualified UXO technicians. Clearance provided by EOD personnel or UXO technicians is mandatory for areas where UXO and discarded military munitions (DMMs) are present or may exist. Safety clearance procedures often differ based on the sampling activity, local range rules, and range activity. At firing points, often only a visual inspection of the surface is necessary prior to granting clearance for near-surface sampling. All of the areas where surface sampling is conducted on impact and demolition ranges should have the top 30–45 cm of surface profile screened for metallic anomalies (i.e., potential UXO/DMMs) using a hand-held analog or digitally recording magnetometer, electromagnetic induction (EM) sensor, or metal geophysical detector (http://www.itrcweb.org/Documents/UXO-4.pdf). Moreover, all surface UXOs and near-surface potential UXO/DMMs should be marked for avoidance. For smaller decision units, the magnetometer is swept over the entire sampling area; for larger decision units, often the increment collection points are cleared during sample collection. Where profile sampling is performed, a geophysical sensor should be used to clear below the surface at 20-cm depth intervals. At demolition ranges, sampling to much greater depths may be necessary to completely define the potential source region.

5 Hand Grenade Ranges

Conceptual Site Model

Hand grenade ranges are only a few hectares or smaller in size and are sometimes divided into several grenade courts, or throwing bays. The surface of grenade ranges is poorly vegetated and very heavily cratered from the large number of individual detonations that occur during troop training exercises. Grenades are thrown from a bay that is behind a well-fortified shoulder-height earthen or manmade wall to shield personnel from the casing fragments that disburse on detonation. The highest energetic residue concentrations are typically in the most heavily impacted area, often located between 5 and 40 m from the throwing bay. Depending on the range management practices, craters in the impact area of the range may or may not be periodically filled by grading the surface.

The grenade most often used today at these ranges is the M67 fragmentation grenade. This grenade contains 185 g of Composition B explosive that is 60% military-grade RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine), 39% military-grade TNT, and 1% wax. Military-grade RDX contains about 10% HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine), and thus the energetic compounds most often found in soils at hand grenade ranges are RDX, TNT, and HMX. TNT is subject to microbial, chemical, and photochemical reactions yielding several transformation products, including 2-amino-4,6-dinitrotoluene (2ADNT), 4-amino-2,6-dinitrotoluene (4ADNT), and 1,3,5-trinitrobenzene (TNB); these compounds are sometimes detectable in soils at hand grenade ranges (Jenkins et al. 2005c).

Concentrations of RDX, TNT, and HMX in surface soils at hand grenade ranges have varied from the low µg/kg to the low mg/kg levels. In experiments that were conducted to estimate the mass of energetic residues deposited when a single M67 hand grenade detonates as designed (a high-order detonation), RDX was the only energetic compound detected, with an average deposited mass of 25 µg (Hewitt et al. 2005b). The loading rate based on this mass and information from training records is insufficient to explain the RDX and HMX concentrations found at several of the hand grenade ranges studied and doesn’t account for the presence of TNT and its breakdown products (Jenkins et al. 2005c). For these reasons, the major source of contamination appears to be grenades that either undergo a low-order (partial) detonation or are duds (UXOs) and are blown in place by explosives ordnance technicians using C4 explosive (91% RDX).

Occasionally ruptured hand grenades have been found on the surface within and around the impact range. Often visible energetic residues were adhering to the interior grenade surfaces. The absence of observed energetic residue particles near grenades that have undergone low-order detonations has been attributed to subsequent detonations that spread these residues across the range.

Residue deposition is predominantly at the surface. However, because repeated detonations occur in one place, craters often become enlarged and are filled in subsequently during range management operations. This mixes residues deeper into the soil profile than is the case at most other ranges (Jenkins et al. 2005c). Some profile sampling should be performed to establish if residue particles are present at greater depths.

Hand Grenade Range – Recommended Sampling Protocols

The area from approximately 5 m in front of the throwing bay to a distance of 40 m and the width of the impact zone should be sampled. For grenade ranges where grenade courts are not separated by barriers, the distance between throwing bays is typically small enough to allow the entire impact range to be characterized as a single decision unit. When walls or other features separate the impact zone into several distinct areas, at least one sample should be taken for each impact zone.

Individual increments for multi-increment samples should be collected from the soil surface to a depth of 10 cm. If the surface area to be characterized is less than 100 m^2, the sample collected should include 30 or more increments. For larger areas, we recommend samples consisting of 100 increments. In both cases, the sample collection pattern should be as shown in Figure 5.

Profile sampling is recommended for these ranges. Within the area with the highest crater density, at least five depth profiles should be collected in 10-cm intervals down to a depth of at least 30 cm. Sample increments from the same 10-cm depth interval (0–10 cm, 10–20 cm, and 20–30 cm) should be combined to produce a single five-increment sample (Fig. 7). Because of the limited number of increments, this sampling strategy is best suited for determining the depth to which residues have been mixed into the soil profile and not to estimate the average concentration for a subsurface layer over a large horizontal cross-sectional area. To achieve this second objective, 30–100 increments should be collected. For depths below 30 cm, a surface geophysical survey may not be sensitive enough to detect grenades; therefore, down-hole clearance should be performed.

If a ruptured grenade with energetic residues on its interior surfaces or a grenade surrounded by chunk residues is encountered, an area that encompasses the visibly affected surface should be sampled as a separate decision unit after all visible pieces of energetic residues (i.e., energetic residues present as MECs) are removed. A 30-increment sample should be collected from the decision unit.

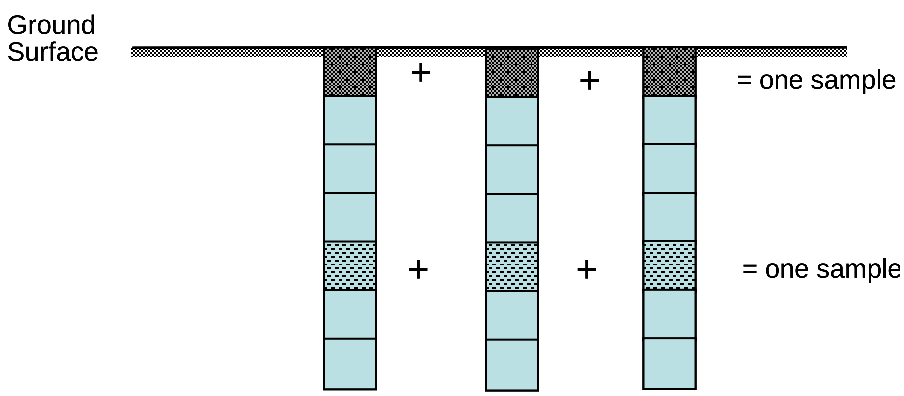


Figure 7. Schematic of procedure to collect multiple-increment profile samples where transport and deposition of energetic materials are suspected.

6 Anti-tank Rocket Ranges

Conceptual Site Model

At anti-tank rocket ranges, projectiles are fired from shoulder-mounted tubes. These ranges are generally several hundred hectares in size and covered by low-growing vegetation because of the necessity of maintaining a direct line of sight between the firing points and targets. Often the targets are derelict vehicles placed downrange at distances of 100 m or more from a firing line. On ranges used only for firing practice rounds (sub-caliber rounds), targets are often made of wood. When the rockets are launched from shoulder-mounted tubes, propellant residues eject from both ends. The highest concentrations of energetic residues have been found around targets and behind the firing line.

The weapon fired to the greatest extent in the last couple of decades was the 66-mm M72 light anti-armor weapon (LAW) rocket. More recently, the AT-4 rocket has been fired at these ranges. The warhead of the LAW rocket contains 0.3 kg of the melt-cast explosive octol with either a tetryl (methyl-2,4,6-trinitrophenyl nitramine) or RDX booster. Octol is composed of 70% HMX and 30% TNT. The warhead of the AT-4 also contains octol. The double-base M7 propellant for the LAW rocket contains 54.6% nitrocellulose (NC), 35.5% nitroglycerin (NG), 7.8% potassium perchlorate, 0.9% ethyl centralite, and 1.2% carbon black. Practice rounds contain propellant but do not contain a high-explosive-containing warhead. We have been unable to locate information regarding the proprietary composition of the AT-4 propellant.

Recent studies at a number of anti-tank rocket ranges have consistently determined HMX to be the major energetic residue in surface soils near targets. In several cases, HMX concentrations in surface soils near targets have exceeded 1000 mg/kg. TNT, RDX, 4ADNT, and 2ADNT are also detectable. However, concentrations of these analytes are two or more orders of magnitude lower than HMX. The concentrations of energetic residues in surface soils decrease with distance from the target (Jenkins et al. 2005c). The major source of energetic residues at these ranges results from M72 rockets that shear open on impact without detonation, thereby depositing crystalline explosives over the surface (Jenkins et al. 1997b, Thiboutot et al. 1998).

Occasionally dud and partially ruptured LAW rockets with intact warheads have been encountered on impact ranges around targets. However, visible chunks of octol (0.5 cm in diameter) have been observed only rarely (an exposed warhead or on the surface). For safety reasons, sampling should not be performed near intact or ruptured anti-tank rockets because the fuze may be armed. Similar to grenade ranges, it is believed that octol residues from ruptured rounds become disbursed by subsequent detonations.

NG is present in surface soils in front of and behind the firing line and around targets at anti-tank rocket ranges. Between the firing line and the targets, concentrations are generally in the high µg/kg to the low mg/kg range. Along the rocket flight path, concentrations were found to be higher just in front of the firing line and at the targets. NG presence around the targets is due to detonation and dispersal of unconsumed rocket fuel. Behind the firing line, NG concentrations are often thousands of mg/kg (Jenkins et al. 2005c). Moreover, concentrations as high as 100 mg/kg have been detected as far as 25 m behind the firing line. Profile samples taken in front and behind the firing line have shown that NG can migrate more than 50 cm below the surface (Pennington et al. 2005).

Near anti-tank weapon firing lines, NC is also present at concentrations probably several times higher than that of NG, but the lack of a validated analytical method for NC in soils has prevented evaluation of the actual quantity except in a few instances (Jenkins et al. 2007). Perchlorate has not been detected in soil samples from these firing point areas, even though it is a component of the propellant formulation.

Anti-tank Rocket Range Targets – Recommended Sampling Protocols

Several studies at anti-tank rocket range impact areas have indicated that most of the residues are within a 25-m radius of targets (Jenkins et al. 1997b, 2004b, Thiboutot et al. 1998). To estimate the mass of residues on these ranges, multi-increment samples collected within a 25-m radius around each target is recommended (Fig. 6). Because the area to be represented by each sample will be about 2000 m^2, we recommend that the soil sample be built from 100 increments of the top 5 cm.

If a more detailed characterization is required, we recommend a segmented halo design (Jenkins et al. 2004b, Pennington et al. 2004). In this design, concentric rings are established at distances of 5, 15, and 25 m from the target, the rings are segmented, and multi-increment samples are collected within each segment (Fig. 8). Because the surface area within a segment is relatively small, each sample should be built from 30 increments.

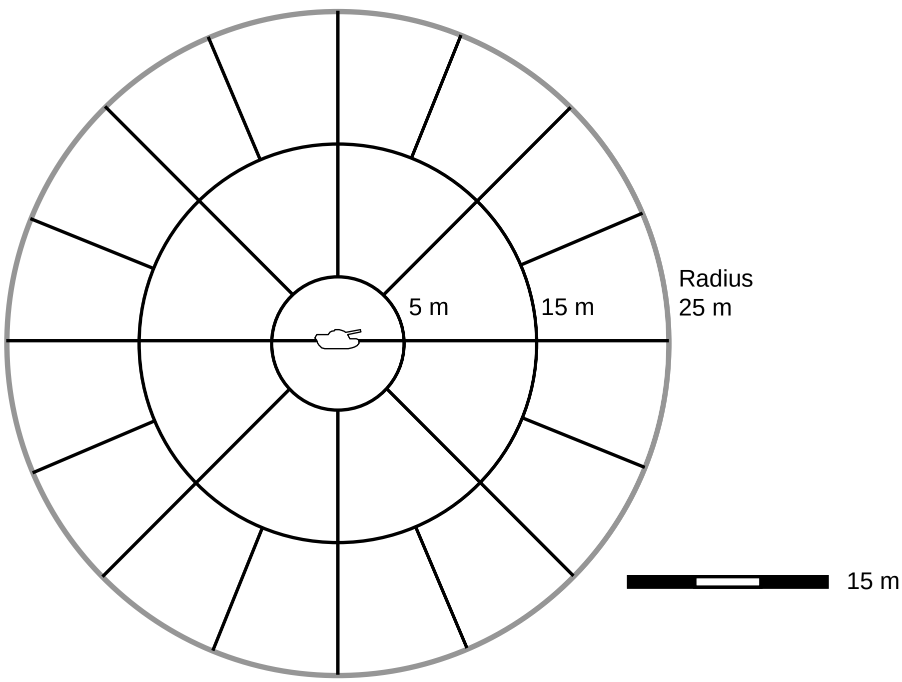


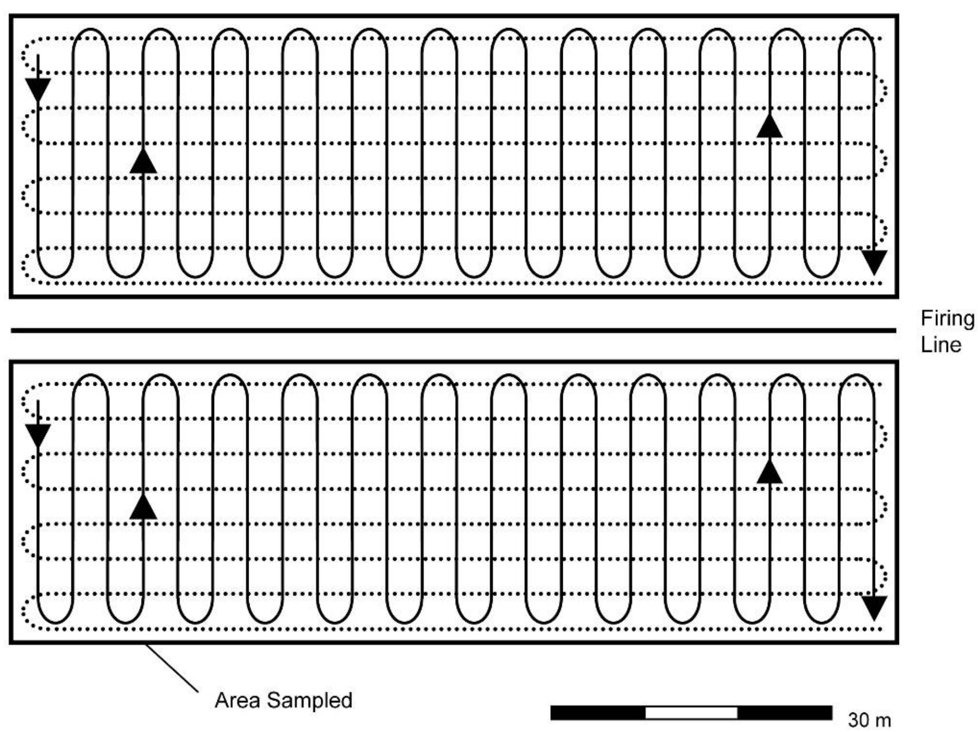
Figure 8. Segmented halo sampling pattern surrounding a tank target at a live-fire bombing range impact area.

To assess any subsurface migration of dissolved energetic residues, the same strategy as presented for the hand grenade range is recommended (Fig. 7). Sampling locations should be near the heaviest impacted target, where it is anticipated that the surface concentrations will be very high. A surface geophysical survey may not be sensitive enough to detect dud rockets at depths below 30 cm. Therefore, down-hole clearance should be performed.

Anti-tank Rocket Range Firing Points – Recommended Sampling Protocols

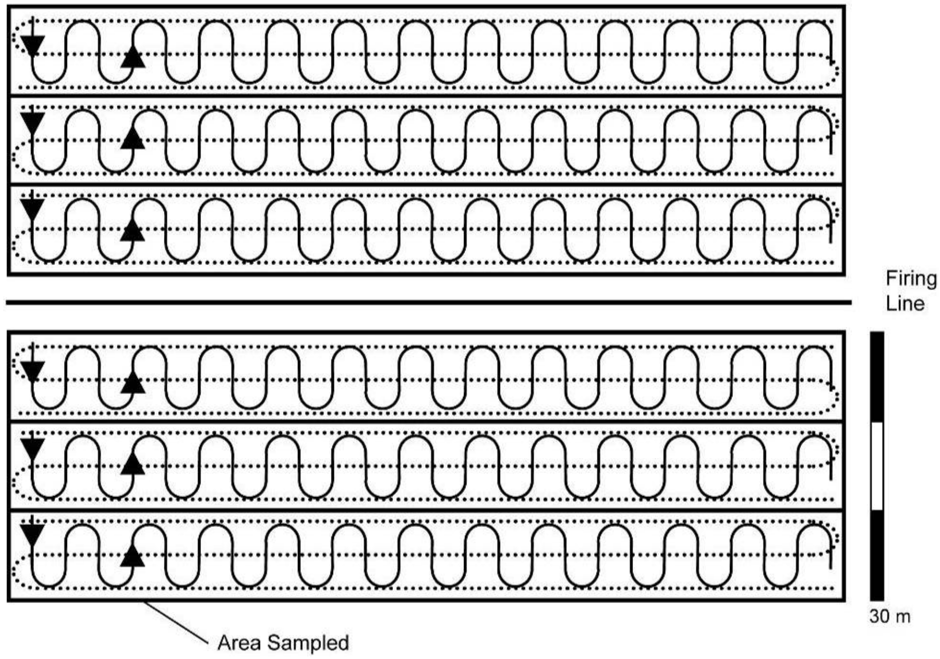
The highest concentration of the propellant residues at these ranges is behind the firing line. If it is desired to estimate the total mass of residue in this area, a single 100-increment sample collected in a rectangle 30 m wide and running the entire length of the firing line is recommended (Fig. 9a). This same design and strategy can also be used just in front of the firing line. If a more detailed characterization is desired, we recommend dividing the area behind and in front of the firing line into three 10-m-wide rectangles along the entire length of the firing line and collecting a 30-increment sample within each area (Fig. 9b). Because residues are deposited at the surface and little surface disruption occurs, we recommend that firing point samples be taken from the top 2.5 cm.

To assess whether subsurface accumulation of energetic residues has occurred, the same strategy as presented in the hand grenade range is recommended. Sampling locations should be 5–10 m behind or in front of the firing line at the firing position used most heavily. If possible, samples should be collected from depths greater than 30 cm. However, the area behind the firing line has often been covered with gravel fill, making it difficult to acquire deep profiles.



a. Pattern to collect one multi-increment sample in a single 30-m wide decision unit.

Figure 9. Strategies for collecting multi-increment samples in rectangular decision units behind or in front of a firing line.



b. Pattern to collect multi-increment samples in three 10-m-wide decision units.

Figure 9 (cont). Strategies for collecting multi-increment samples in rectangular decision units behind or in front of a firing line.

7 Artillery Ranges

Conceptual Site Model

Artillery ranges are the largest training ranges in the Army inventory, generally covering hundreds of square kilometers. Firing positions are often arranged around the circumference of the range with firing fans extending into the main impact zone, which generally is positioned near the center of the range (Fig. 10). Once fired, most artillery and mortar rounds and rockets travel several kilometers before detonating upon impact in the general vicinity of the targets. The flight path takes these rounds over an area referred to as the range safety fan, the large area between the firing point and the target and/or a large area surrounding the target that is off limits to personnel during training activities. Generally, only a very few misdirected or defective rounds land within the main impact zone outside of target areas.

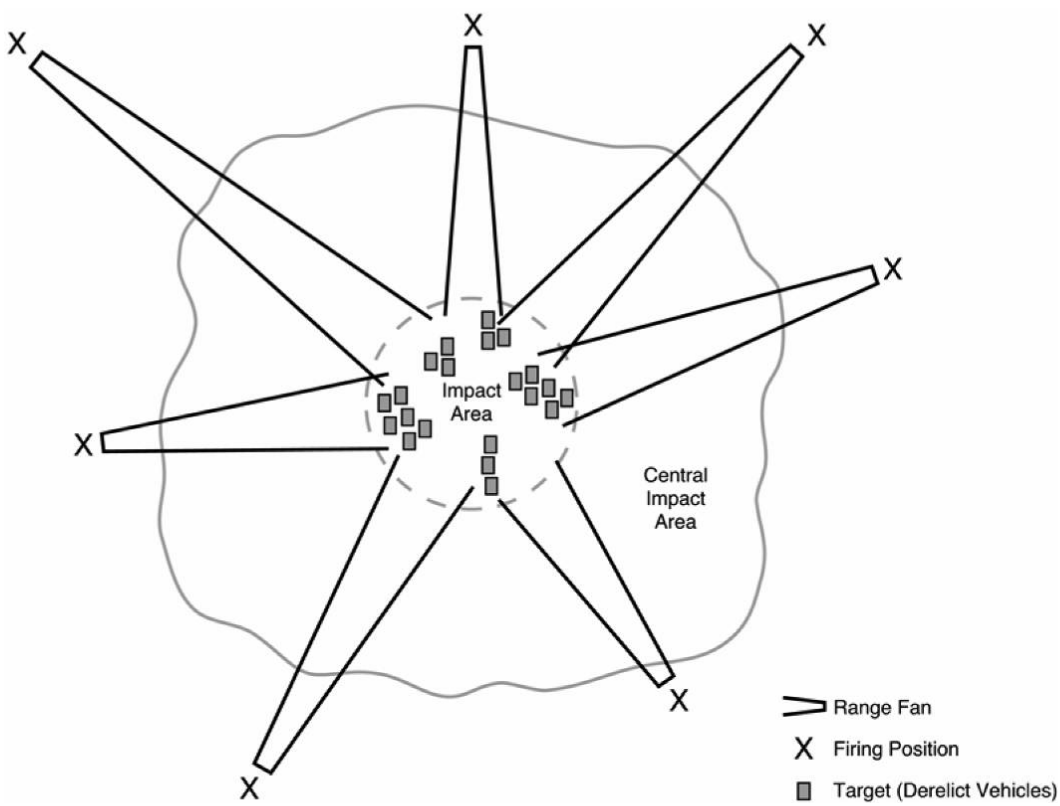


Figure 10. Schematic diagram of an artillery range showing firing points, range safety fan, and impact areas.

Munitions fired into these ranges are artillery and mortar guns, although various rockets, missiles, and Air Force and Navy bombs have been used on many of these ranges in the past. Weapons currently fired in the greatest quantities are 155-mm howitzers and 105-mm artillery projectiles, 120-mm tank projectiles, and 81-mm, 60-mm, and 120-mm mortar rounds. However, a wide variety of other munitions have been (and some continue to be) periodically fired into these areas, including 90-mm recoilless rifle rounds, 4.2-in. mortar rounds, 8-in. artillery projectiles, bombs of various sizes, 40-mm grenades, 106-mm high-explosive plastic (HEP) rounds, 2.75-in. rockets, LAW rockets, and TOW missiles. The high explosives used in artillery and mortar warheads are generally either TNT or Composition B (RDX and TNT), although some older rounds also contained tetryl. Some smoke-generating munitions contain metal nitrates, hexachloroethane (HC), and potassium perchlorate, and spotting charges contain white phosphorus (WP) and black powder. Bombs that have been dropped in some of these ranges contain TNT, tritonal (TNT and aluminum) or H6 (RDX, TNT, and aluminum), some 40-mm grenades contain Composition A5 (RDX), and LAW rockets contain octol (HMX and TNT).

When rounds perform as designed, the detonation often forms a crater in the soil, the size of which is a function of the type of munitions, the physical properties of the soil, the type of fuze, and the fuze setting. Therefore, impact areas can be identified by the presence of targets, debris from past targets, and areas with a large number of craters (crater fields). Old crater fields and target areas can often be identified from high-resolution aerial photography, LIDAR, and range maps. Experiments have been conducted to estimate the mass of energetic residues deposited when various mortar and artillery rounds detonate as designed (Jenkins et al. 2002, Hewitt et al. 2003, 2005b, Walsh, M.R. et al. 2005a,b,c, 2006). Overall, high-order detonations consume the energetic compounds in the warhead very efficiently, depositing only microgram to milligram quantities per round over hundreds of square meters of surface area. As a consequence, almost all surface soil samples collected from individual craters and from heavily cratered areas (absent of rounds that have undergone low-order detonation) contained residue concentrations below 0.1 mg/kg (Jenkins et al. 2001, Hewitt et al. 2005a).

Figure 11. Example of an 155-mm artillery round that has undergone a low-order detonation. Explosive fill is still present in and around the casing.

Occasional rounds that impact without detonating result in either surface or subsurface UXOs. On ranges with rocky or very hard soil, many of these UXOs can be seen on the surface. In a relatively small number of cases, a round will partially detonate upon impact, resulting in a low-order detonation. In these cases, only a portion of the explosive filler is consumed, sometimes leaving a substantial fraction of the explosive in or near the ruptured casing (Fig. 11). Sometimes a nearby high-order detonation will rupture a UXO or cause it to undergo a low-order detonation. Here again, a substantial portion of the explosive fill will remain. These low-order detonations and ruptured rounds result in the largest source of energetic residues at artillery ranges (Jenkins et al. 2001, 2004a, Hewitt et al. 2005a, Walsh, M.R. et al. 2005c). Typically these events are rare for most munitions, so their distribution is both isolated and random. Therefore, the results of low-order detonations often exist as distributed point sources of very high concentrations of residues, the sizes of which can vary considerably (5–1000 m^2). Moreover, unlike anti-tank target areas, there is generally no well-defined gradient of energetic residues around artillery and mortar targets. This random spatial array in the occurrence of low-order detonations has been attributed to indirect fire and the large distance between the firing position and the target. Currently, delineating the area impacted by a low-order detonation is based on judgement (defined by visual identification) and can easily be confounded by vegetation and deterioration of the residues. The size of this area and a more thorough analysis of their occurrence and spatial distribution need additional study. Surface soil concentrations of energetic residues in areas where rounds have undergone low-order detonations often reach into the hundreds of mg/kg and may present a risk to humans and the environment. The major residues found in impact areas from these low-order detonations are TNT, RDX, and HMX (Jenkins et al. 2001, 2004a, Hewitt et al. 2005a). Moreover, since these samples are likely to be much higher in energetic residue concentration, they should be isolated from all other samples during shipping and laboratory processing (USEPA 2006b).

Mortars, howitzers, and rockets are fired from firing points and open firing areas. Open firing areas have become more common with the development of mobile artillery, which often employ a "shoot and scoot" strategy. At firing points and areas, propellant residues are deposited downrange of the guns and mostly behind firing positions for the rockets. The amount of propellant residues deposited is highly dependent on the different weapons systems and their individual propellant formulations and configurations. These munitions are delivered using single-, double-, or triple-base gun propellants and rocket and missile propellants. Single-base gun propellants are composed of nitrocellulose (NC) and 2,4-dinitrotoluene (2,4-DNT); double-base gun propellants are composed of NC and nitroglycerin (NG); and triple-base gun propellants are composed of NC, NG, and nitroguanidine (NQ). At heavily used firing points and areas, energetic residue concentrations often range between low to tens of mg/kg. However, because mortar and howitzer rounds are frequently fired with less than a full load of available propellant, if any excess propellant is burned near the firing point, it is likely to create areas with even higher concentrations (burn points).

Away from Firing Points and Targeted Areas – Recommended Sampling Protocols

Sampling studies performed in the region 100 m from an established firing position to within 500 m of targets or heavily cratered areas have generally not found any measurable concentrations of energetic compounds (Ampleman et al. 2003, Thiboutot et al. 2003, 2004, USACHPPM 2001, 2003, 2004, in press, Walsh, M.E. et al. 2001). If it is decided that this area needs to be sampled, a square decision unit of 50- × 50-m or larger should be chosen if no surface anomalies are observed, and a 100-increment sample should be collected from the top 5 cm. Alternatively, if the sampling plan requests that a qualitative reconnaissance (visual inspection) be performed in this area, it is recommend that a multi-increment sampling strategy with widely distributed collection points accompany this activity. When sampling large areas (> 10,000 m^2), global positioning systems could be used to help locate evenly spaced positions where individual increments will be collected. This is particularly important in adverse terrain with large changes in elevation and/or dense vegetation.

Impact Areas – Recommended Sampling Protocols

For areas with a defined target (or target debris), a 50- × 50-m square grid is recommended, centered on each target, and a 100-increment sample should be collected from the top 5 cm using the systematic-random design (Fig. 5). If rounds have undergone low-order detonation or chunks of energetic residues are visible and identified by field screening methods, a 10- × 10-m grid or smaller decision unit centered on each of these areas should be marked (Fig. 12). Then, qualified personnel should remove all visible pieces of MEC. In some cases, a UXO that cannot be moved for safety reasons may also be present in the decision unit. This item and any other magnetic anomalies should be marked for avoidance. Once these tasks have been completed, a 30-increment sample should be collected within these areas from the top 5 cm.

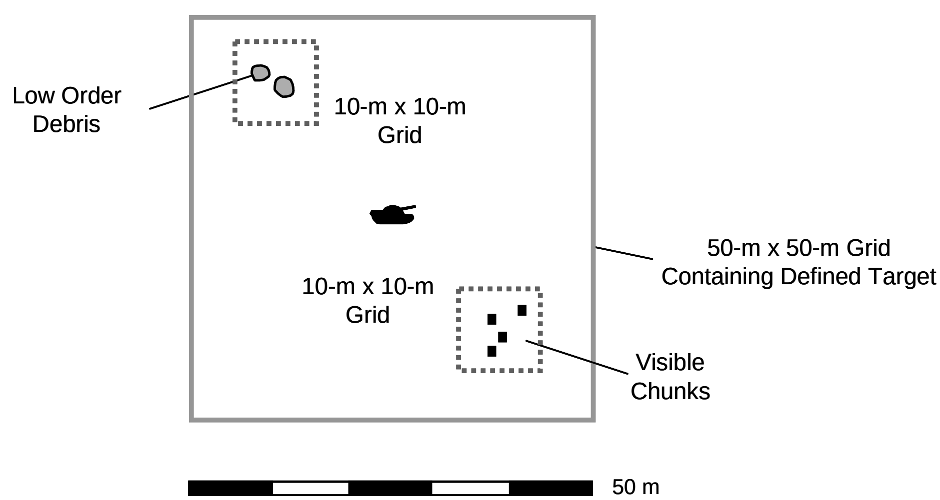


Figure 12. Decision unit for collecting multi-increment sample surrounding a defined target at the impact area of an artillery range.

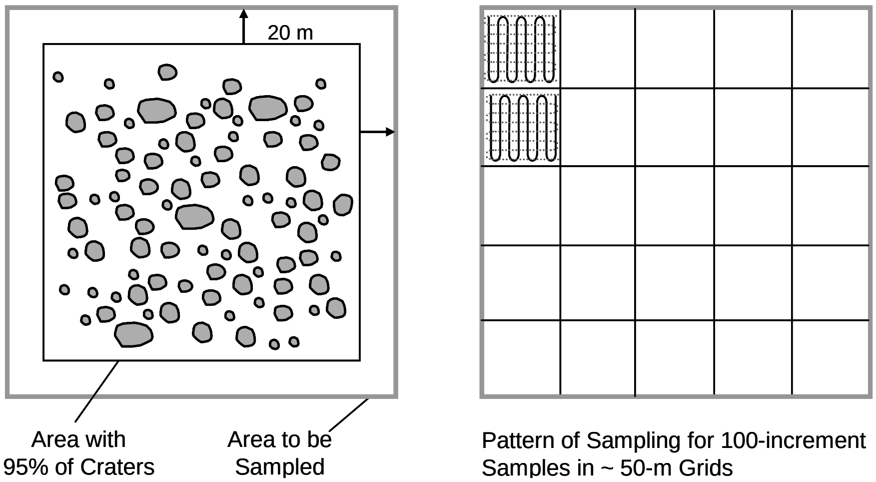


Figure 13. Example of sampling strategy at a crater field section of an artillery-mortar range impact area.

For heavily cratered areas, the area of concern should encompass at least 95% of the craters and a 20-m buffer zone (Fig. 13). These areas can be very large, depending on several factors such as placement of targets, training objectives, and age of the training facility. The recommended size of sampling units within this area is 50 × 50 m (or smaller) and a 100-increment sample from the top 5 cm should be collected in each unit. If chunk explosive or a round that has undergone low-order detonation is encountered, a 10- × 10-m or smaller sampling grid is established and sampled as discussed above.

Profile sampling is recommended only in areas where low-order detonations have been found. As before, we recommend collecting at least five profile samples, then combining the individual depth intervals (0–10 cm, 10–20 cm, and 20–30 cm) to form a single five-increment sample for each of these depths (Fig. 7). The intent of this sampling strategy is to establish the depth to which residues have been mixed into the soil profile, not to determine the average concentration for a subsurface layer over a large area. To achieve this second objective, 30–100 increments are needed. For depths below 30 cm, a surface geophysical survey may not be sensitive enough to detect UXOs, so down-hole clearance should be performed.

Firing Point Areas on Artillery-Mortar Ranges – Recommended Sampling Protocols

Most of the residue deposition at mortar or artillery firing locations occurs in front of the gun tube. However, residue can accumulate on the surface at detectable levels up to 100 m downrange (Pennington et al. 2002, Walsh, M.R. et al. 2006). Within firing areas where a variety of gun arrays are used, gradients become obscured but may exist downrange from the edge of the firing area. Within the firing area, decision units of 50 × 50 m or smaller can be used for collecting 100 increments from the top 2.5 cm (Walsh, M.E. et al. 2004, 2005).

At an established firing line or along the perimeter of the firing area, samples can be collected in rectangular decision units to assess the downrange gradient parallel with the direction of fire. For each rectangular decision unit, a 30-increment sample of the top 2.5 cm should be collected (Fig. 9b).

When a location that has been used to burn excess propellant is distinguishable, this area should be treated as a separate decision unit. A 30-increment sample from the top 5 cm should be collected within a 10- × 10-m or smaller area centered on the location.

Profile sampling would only be recommended at a heavily used fixed firing point or directly beneath a location where propellant was burned on the ground surface. At a fixed firing point, profile sampling should be performed using our recommended strategy within 5 m of a mortar firing point and within 10 m of a howitzer firing point.

8 Bombing Ranges

Conceptual Site Model

Air Force ranges are very large, generally hundreds of square kilometers. In the past, bombs often landed up to a kilometer away from the intended targets; however, with the development of precision guided systems, the area impacted is becoming much smaller, generally only tens of hectares. The Air Force periodically conducts range clearance activities—duds are blown in place, chunks (larger than golf-ball size) of high explosive compounds observed on the surface are gathered up and destroyed by detonating them with C4, and craters are often filled.

The high explosive present in U.S. and Canadian Air Force bombs is usually either tritonal (TNT, aluminum powder) or H-6 (TNT, RDX, aluminum powder). Some older bombs contained solely TNT. Although experiments documenting the residue deposited when a bomb detonates as designed have not been conducted, experimental results for large artillery rounds indicate that large-mass HE detonations are very efficient, dispersing only microgram-to-milligram quantities of residue when they detonate at high order (Hewitt et al. 2005b, Walsh, M.R. et al. 2005 a,b,c, 2006). As with other ordnance items, low-order detonations or duds, ruptured by impact or subsequent detonations, are thought to be the major source of residues on bombing ranges. Both ranges that we have sampled had isolated areas within the impact zones where a bomb had undergone low-order detonation. In these areas, chunks of high explosive were observed on the surface, and high mg/kg concentrations of energetic residues were determined in the <2-mm size fraction of the soil collected. However, most of the rest of the heavily impacted area had residue concentrations of less than 1 mg/kg (Pennington et al. 2004, Jenkins et al. 2006b).

Bombing Ranges– Recommended Sampling Protocols

We conducted surface sampling studies on two bombing ranges. At one range, we sampled around a fixed target position, and at the other range, we sampled in a large (tens of hectares) crater field. Based on these preliminary findings the sampling designs and strategy recommendations for an artillery impact range would also apply here. LIDAR, high-resolution orthophotography, and range maps can be evaluated as forensic evidence to locate targets and craters from historical range usage.

9 Demolition Ranges

Conceptual Site Model

Duds that are safe to move and outdated munitions are destroyed on demolition ranges by military explosive ordnance disposal (EOD) technicians. In addition, sometimes chunks of high-explosive, unused propellants and items found by law enforcement within the area served by the EOD unit stationed at that facility are also destroyed at these ranges, either by demolition or open burning. Demolition ranges are generally only a few hectares, and the active areas are sparsely vegetated. Often several active demolition craters and burn pits are present on a demolition range. These craters and pits are used many times and sometimes are subsequently filled in. Because of this common range practice, high concentrations of energetic residues can be detected deeper in the soil profile at these ranges than at other range types. Consolidated detonations of buried multiple rounds may result in a source area up to approximately 4 m deep at demolition ranges.

The common practice today is to place one or more blocks of C4 explosive on the item to be detonated. The C4 donor charge often is initiated using a blasting cap. This practice has been used for the disposal of UXOs and bags of propellants, as well as for cutting metal. At some demolition ranges, for example, C4 explosive is used to make holes in practice bombs to ensure that they contain no high explosives before these items are recycled (demilitarization of items). C4 is composed of 91% military-grade RDX that has an impurity of HMX at about 10%. Research studies indicate that substantial residues of energetic compounds can sometimes be deposited during demolition events, particularly if they result in a low-order detonation of the item being destroyed, or if the C4 doesn’t detonate completely and becomes scattered across the site (Pennington et al. 2004). Even in cases where only practice bombs are breached, the residues from the C4 demolition explosive accumulate on these ranges (Jenkins et al. 2006b). The C4 demolition explosive is unconfined, and this may lead to lower destruction efficiencies than for detonation of confined charges (Pennington et al. 2004).

Surface soil sampling has almost always resulted in the detection of RDX, HMX, 2,4-DNT, and NG; at several ranges, substantial pieces of C4 were found on the surface. Concentrations of 2,4-DNT and NG result from attempts to improperly detonate surplus propellants or from kick-out during open burning activities. Within the demolition range, there are often areas with concentrations of one or more energetic residues in the tens to hundreds of mg/kg.

Demolition Ranges– Recommended Sampling Protocols

The portion of the range where demolition or open burning is performed should be identified and divided into 10- × 10-m grids (Fig. 14). A 30-increment sample from the top 10 cm of depth should be collected in each decision unit. Profile samples should also be collected in areas where the surface has been discolored or where demolition craters had been located in the past. Depth increments from at least five profile samples should be combined in a manner similar to that recommended for other ranges. In this case, however, the sampling depth should extend below 4 m and perhaps continue to the groundwater table. For depths below 30 cm, down-hole clearance should be performed at 20-cm intervals.

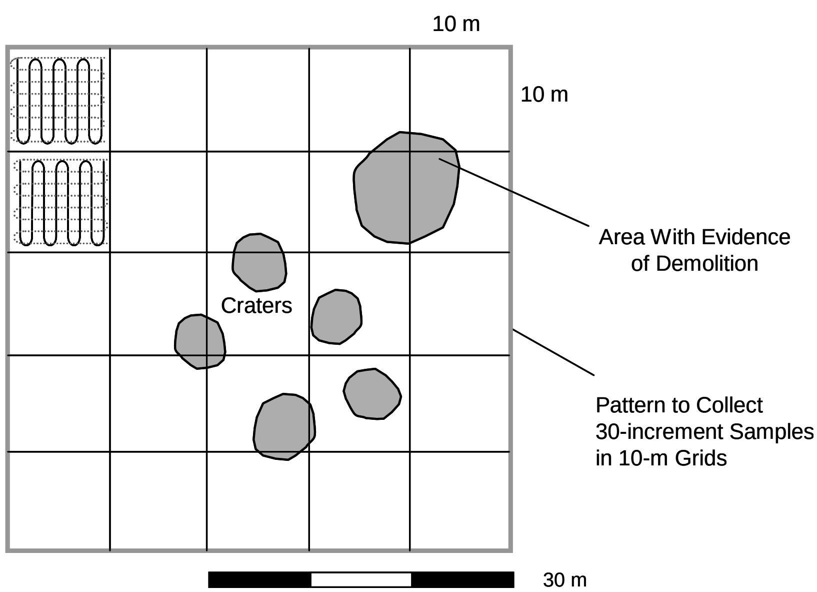


Figure 14. Recommended sampling strategy for collecting multi-increment samples at a demolition range.

10 Lessons Learned

The most important aspect of an environment characterization program is the sample collection activity. Failure to collect the appropriate type and number of samples cannot be compensated for by subsequent laboratory activities. In contrast, laboratory shortfalls can often be corrected, allowing the data to be validated. For this reason, the sampling activity should receive the greatest amount of oversight, and the sampling strategy and design should be conservative, i.e., additional samples should be collected, and the number of increments obtained to build multi-increment samples should be maximized rather than minimized. Of equal importance is the collection of field triplicate samples to assess the uncertainty in the sampling strategy and design for a given activity. It is also important on active ranges not to remove surface vegetation (mosses, leaf debris, and short grasses) prior to collection. When the extent of the area influenced by an activity is unknown or in dispute, more decision units should be added to the sampling plan, and these areas should be sampled. Adoption of this philosophy will reduce the number of times the field sampling team is deployed for a given investigation.

With respect to the processing and analysis of field samples, it is imperative that either the entire field sample be pulverized and properly subsampled, or that the entire field sample should be extracted. This laboratory activity must be scrutinized visually, and triplicate subsamples should be taken at an established interval to assess the uncertainty associated with this activity. As a rule of thumb, a program should strive to achieve a field sampling variance of less than 50% relative standard deviation (RSD), and preferably 30% RSD, and laboratory subsampling variance should be less than 20% RSD, and preferably 10% RSD.

This approach was used at Hill Air Force Base to characterize the surface loading of energetic residues on a large demolition range. Of particular interest was the concentration and distribution of HMX and perchlorate. The sampling plan developed to characterize the surface of this range used 100- × 100-m contiguous sampling grids within the area of concern. From each grid, one or triplicate 100-increment samples were collected using the systemic-random sampling design. The total characterization variance for the field triplicates has routinely been below 20% RSD and often below 10% RSD for both of these analytes. More about this program can be found at www.sesincusa.com/em/KNieman/CY2006_Final_ADV_Soil_Sampling_Report.pdf.

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Appendix A: Standard Operating Procedure for the Multi-increment Sampling Strategy and Systematic-Random Sampling Design

The intent behind this strategy and design is to obtain soil sample increments positioned at collection points that are distributed relatively evenly throughout the sampling area (decision unit). The final sample weight and total number of increments collected to make a single representative sample depends on the compositional and depositional heterogeneity of the constituent of concern. Replicate multi-increment samples from the two example decision unit sizes described below—100 m^2 (10 × 10 m) and 2500 m^2 (50 × 50 m)—have resulted in reproducible concentrations for energetic residues at the ranges addressed in the body of this report. The increment number and sample weight, however, do not need to be exactly the recommended target values. For example, the sample weight can vary from 20% below to 300% above the 1-kg target weight. The target number of increments can also vary but should always be at least 30. The decision unit size and shape will likely depend on data quality objectives and terrain features. It may be square, rectangular, or circular, or it may fit the outline of an area established by terrain features (either man-made or natural). The objective is to collect increments so that the entire area is sampled evenly and is represented in the sample.

Select an appropriately sized decision unit for the activity and the study objective. Sizes recommended for military training ranges typically range from 10 × 10 m (100 m^2) to 50 × 50 m (2500 m^2).

Position boundary limit flags at each corner of the selected area. Along two opposite sides, place nine flags at even intervals (e.g., 1-m or 5-m intervals) to define 10 lanes.

Select the number and size of increments to be collected and the sampling depth. Typically, 100 increments are collected in a 50- × 50-m area, and 30 increments are collected in a 10- × 10-m area. The recommended sampling depths are 2.5, 5, or 10 cm, depending on the expected depth distribution of the analytes. Set or mark the sampling tool for the appropriate depth. For greater depths and larger number of increments, use a smaller diameter sampling tool so as not to build samples that weigh much more than 1 kg. Given that soil density is typically around 1.7 g/cc, the following sizes of sampling tools are recommended for different sampling depths and number of increments. For a depth of 2.5 cm, 2- and 3-cm-diameter coring tools (or scoop/trowel) would be appropriate for a 100- or 30-increment sample, respectively. For a depth of 5 cm, 1.75- and 2-cm-diameter coring tools would be appropriate for a 100- or 30-increment sample, respectively. For a depth of 10 cm, 1.25- and 1.75-cm-diameter tools would be appropriate for 100- and 30-increment samples, respectively.

Sampling works well as a two-person activity: one person collects the increments and the other holds the sample container (clean polyethylene bag) and keeps track of the number of increments. Using the flags to visualize the 100 sub-units, start in one corner of the sampling area and acquire an increment near the middle of the sub-grid and every third one thereafter for a 33-increment sample, and every other for a 50-increment sample. This should appear as a serpentine sampling pattern ending at the opposite corner of the decision unit from where sampling was started (see Fig. 5).

When replicate field samples are taken, flags should be positioned at the appropriate intervals on all four sides of the sampling area, creating a visual sub-grid pattern. The replicate samples should be collected starting at a sub-grid offset from the original position, or if every sub-grid is a collection point, then a random position should be selected within that sub-grid and repeated throughout the decision unit. Random predetermined locations within a sub-grid can be generated by rolling dice, or by paying close attention to where the previous increments were collected, and offsetting increments from the same sub grid. If dice or some other random number generator is used, replicate samples can be collected during a single pass through the decision unit using multiple bags.

When decision units are rectangular, the conversions for the spacing (steps) between increment collection points are fairly straightforward to calculate. However, with other shapes, it is recommended that the perimeter be marked and flags be pre-positioned at an estimated interval across the middle of the decision unit in two perpendicular lines. Then a trial run (no sample collection) is performed to quickly establish the distance between increment collection points to achieve the desired number of increments, while using the flags inside the decision unit as guides. The spacing between these flags should provide grid markers to assist with judging where the increments are to be collected.

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14. ABSTRACT
In the past, very little guidance has been available for site characterization activities addressing the concentration and mass of energetic residues in military training range soils. Energetic residues are heterogeneously distributed over military training ranges as particles of various sizes, shapes, and compositions. Most energetic residues are deposited on the surface, and the highest concentrations exist at firing positions, near targets, and where demolition activities are performed. In the case of impact and demolition ranges the greatest quantities of residues are from rounds that fail to detonate as designed. To address the compositional and distributional heterogeneity associated with the distribution of particles and to obtain representative mean energetic residue soil concentrations, the sampling strategy must strive for the acquisition of samples that contain the constituents of concern in the same proportion to the bulk matrix as exists within the decision unit (sampled area, population, or exposure unit). This report summarizes the sampling strategies and designs that have been implemented for various types of military ranges, including hand grenade, antitank rocket, artillery, bombing, and demolition ranges. These protocols were developed during investigations on active ranges and primarily addressed potential surface source zones from which energetic residues could be migrating into surface and groundwater systems. A multi-increment sampling strategy was selected to accomplish this task after exposing the inadequacies of discrete sampling.

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