STANDARDS RELATED DOCUMENT

AJEPP-6.1

MANUAL FOR ENVIRONMENTAL SAMPLING PROTOCOLS

EDITION B Version 1

APRIL 2022

NORTH ATLANTIC TREATY ORGANIZATION

Published by the
NATO STANDARDIZATION OFFICE (NSO)

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NORTH ATLANTIC TREATY ORGANIZATION (NATO)

NATO STANDARDIZATION OFFICE (NSO)

NATO LETTER OF PROMULGATION

12 April 2022

1. The enclosed Standards Related Document, AJEPP-6.1, Edition B, Version 1, MANUAL FOR ENVIRONMENTAL SAMPLING PROTOCOLS, which has been approved in conjunction with AJEPP-6 by the nations in the MCJSB, is promulgated herewith.

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Dimitrios SIGOULAKIS
Major General, GRC (A)
Director, NATO Standardization Office

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CHAPTER 1 GENERAL INFORMATION

1.1. **INTRODUCTION**

Military readiness is critical to NATO and the Partnership for Peace (PfP) countries. Responsible environmental management of ranges and field deployment sites, essential to maintaining the land assets necessary for military readiness training, requires effective documentation. Environmental documentation supporting NATO field camp site selection, site management during the lifetime of the field camp, and site handover is necessary to protect human health and safety and reduce potential legal liability. Environmental monitoring of live fire training events and training ranges is key to long-term to success and sustainability of NATO operations. Characterizing and managing contamination during field camp and live-fire training operations requires credible, defensible, and reproducible methods for environmental sampling.

A key component of field camp environmental documentation will be the collection and analysis of soil and/or water samples that are both representative and reproducible to determine the baseline conditions prior to, during, and following the occupation of a site. Data derived from sampling will support the Environmental Baseline Survey (EBS), Environmental Health Site Assessment (EHSA), Environmental Closeout Study (ECS), Environmental Impact Assessment (EIA), Environmental Condition Report (ECR), hazardous material record management, and environmental handover certificate.

To prepare for combat operations and achieve a high level of readiness, live-fire training with a wide variety of munitions is required. Munitions used during live-fire training contain energetic materials and other constituents such as heavy metals that have the potential to contaminate the environment. Many of the constituents are toxic to human health and the environment. Therefore, it is important to ensure that the accumulation of contaminants on training ranges does not occur at levels that will result in adverse effects to the environment or human health. In order to control these risks, it is important to know what kind of residues are released from different types of munitions, how the residues behave in the environment, and how they are distributed and move through the environment.

The sampling results obtained through the implementation of the protocols detailed in this guidance document can be used as a decision-making tool for the management of field camps and ranges to avoid the accumulation of toxic and hazardous compounds to harmful levels. This knowledge will help minimize and manage environmental contamination during NATO operations. Furthermore, science-based characterization of the land and management of the environmental impacts of live firing will lead to sustainable training and will protect governments from future environmental liabilities, the closure of important facilities, and the loss of critical training assets.

1.2. PURPOSE

The purpose of this publication is to serve as a standards related document that will assist in the environmentally responsible management of military field camps and ranges as well as the mitigation of contaminants on these sites. The ability to perform proper risk assessments is critical in this respect. If the risk for human health and/or the environment is overestimated, more money than needed may be spent on remediation, unintended damage to the environment may occur, or unnecessary restrictions may be placed on land use. If the risk is underestimated, human health and the environment can be harmed, and remediation will be more-costly because the extent of the contamination will expand and intensify over time, which can lead to range restrictions or closures and high liability costs. Precise risk assessments imply more investment in research and data acquisition. However, because different ranges share many of the same properties, much of the data and experience can be shared between sites.

The following chapters provide guidance on soil and water sampling as well as laboratory and documentation guidance that will support both NATO field camp and live-fire operations.

CHAPTER 2 GENERAL SAMPLING DOCUMENTATION

2.1. FIELD DOCUMENTATION

Proper documentation of all site activities is an integral part of the field investigation. Standard practices and procedures, as outlined below, should be used when documenting sampling activities. Documentation requirements include procedures required for field documentation, sample labelling, and the maintenance of chain of custody (COC). Proper completion of all documentation with indelible ink is necessary to support the use of these records in any potential legal actions. Corrections to documentation should not obliterate data entries; rather place a single line through an incorrect entry, note the corrected information, add the recorder's initials, and record the date that the correction was made. Maintaining sample integrity through proper documentation is essential. Following site activities, all project documentation becomes part of the project file and is the basis of the field report.

2.2. FIELD NOTEBOOKS

The field notebook should contain sufficient information to enable the sampling activity to be reconstructed without relying on the collector's memory. Field notebooks should be bound and have numbered water-resistant pages. The project name shall be recorded on the inside front cover of the notebook. All pertinent information regarding the site and sampling procedures must be documented as near to real-time as possible. At the conclusion of each day, the person maintaining the notebook should sign and date the day's documentation entries. Notations should be made in logbook fashion, noting the time and date of all entries. Notebooks should be kept in the member's possession or in a secure place during field work. Following site activities or if the notebook is completely filled, the notebook becomes a part of the project file as noted above. Suggested topics to include in the field notebook are:

- Name and exact location of site of investigation or interest;
- Name and title of person maintaining the notebook (author);
- Date and time of arrival at and departure from site location;
- Purpose of site visit or sampling activity;
- Name and address of field contact;
- Names and responsibilities of key persons at site location;
- Level of personal protective equipment worn at the site and reasoning;
- Weather conditions on the day of sampling, and any additional environmental conditions or observations pertinent to field activities;
- Sample type, e.g. soil, water;

- Sample collection method, e.g. grab, composite, or multi-increment for soils, cross-sectional multi-depth for surface water, and pumped aliquots for groundwater;
- Sample collection tools;
- Sample handling procedures;
- Sample location, e.g. clearly document the location or area of samples taken;
- Dimensional sketch of the general surroundings of the site to be occupied, and support with other forms of documentation (e.g. photographic log). Sample identification numbers should correspond directly with sample locations;
- Sample numbers, volumes, and containers (number, size, type) used for each sample collected. Note the date and time of each sample, identify any associated quality control samples, or any factors that may affect the quality;
- Any field measurements, field screening/analytical results generated, calibration methods used, field results, and quality control information;
- Appropriate references to maps and photographic logs of the sampling site; and
- Number of shipping coolers packed, COC numbers (or attach a copy of the COC), and record of the mode of transport and applicable tracking numbers.

2.3. **DOCUMENTING SAMPLING POINTS AND DECISION UNITS**

The exact locations of sampling points (discrete samples) and/or decision units (DUs) shall be documented. Sampling points and outlines of decision units should be determined as accurately as possible in the field and precisely located on a map. Positioning is especially important if the sites are to be re-sampled at a later date.

GPS grid coordinates should be in the military grid reference system (MGRS) and have at least a 10-figure grid reference (1 m accuracy). Accuracy and latest calibration of the instrument should be noted. It may be necessary to create a monument (stationary reference point). If a building or other stationary structure exists, its corner may act as a reference point at each site to act as a stationary reference point from which all sampling points can be referenced (e.g. 10 m from the headquarters flagpole on a bearing of 345°). The methodology for sample numbering should include the following as a minimum:

- The site-specific identifier (e.g. NA11, CA1, FOB1);
- Type of sample;
- Date taken;
- Initials of sampler;
- Numeric identifier;
- GPS coordinate for grab or composite sample points for soil and water;

- Number of increments and boundary of the DU for multi-increment sampling;
- Replicate number (if applicable).

2.4. PHOTOGRAPHIC DOCUMENTATION

All sampling points or areas should be documented using digital photography with a date-time indication on the photograph. A photographic record of a sampling event allows positive identification of the sampling point or area. In some cases, a photograph of the actual sample collected may also be required.

Photographs are the most accurate and convenient record of field personnel observations. Photographs taken to document sampling points should include two or more reference points to facilitate relocating the point at a later date (e.g. sample in relation to a flagpole). Keeping a record of photographs taken is crucial to their validity as a representation of an existing situation. Photographic documentation is invaluable if the sampling and subsequent analytical data end in legal actions or formal investigations. It should be noted that in some instances digital photography might not be considered a legal photograph, and conventional photography must be used. In addition to photographs, video coverage of a sampling episode can be equally as valuable as, or even more valuable than, photographs, because it can be used to prove that samples were taken properly as well as to verify the location at which they were taken. Video coverage can be used as a record of site conditions and can give those who have not been on site an idea of the circumstances. For each photograph taken, the following items should be noted in the field notebook:

- Date;
- Time;
- Photographer (name and signature);
- Name of site;
- General direction faced and description of the subject; and
- Sequential number of the photograph and the roll number if conventional.

Special attention should be given to the appropriate storage of digital photographs and videos to guarantee both their eventual confidentiality and their availability for later consultation along with other (digital and non-digital) field documentation.

2.5. DOCUMENTATION QUALITY CONTROL

- Corrections to Documentation. All original data recorded in field notebooks and on sample labels, COC records, and receipt-for-samples forms must be written in waterproof ink. If an error is made on an accountable document, corrections should be made by crossing out the error and entering the correct information.

The erroneous information should not be obliterated. Any error discovered on a document should be corrected by the person who made the entry. All corrections must be initialed and dated.

- Photographs. The photographer should review the photographs and compare them with the photographic log to confirm that the log and photographs match.
- Sample Labeling. Although most sample labeling tags are made with water-resistant paper and are filled out using waterproof ink, inclement weather and general field conditions can affect the legibility of sample labels. It is recommended that after sample label tags are filled out and affixed to the sample container, the tag should be covered with wide clear tape. This will preserve the labeling and keep it from becoming illegible. The sample bags may also be labeled as a redundancy safeguard in the event that the sample label tag is lost. In addition to label protection, COC and analysis request forms should be protected when samples are shipped in iced coolers. Typically, these forms should be placed inside a ziplock bag or similar waterproof protection and taped to the inside lid of the secured shipping container with the samples. Copies of all sample documentation should be made and stored separately prior to shipment.

CHAPTER 3 SOIL SAMPLING

3.1. GENERAL

Soil samples are collected to determine the presence of potential contaminants of concern from previous site use (EBS) and NATO site use (ECS). Contaminants of concern may include one or more of the following: Volatile Organic Compounds (VOC), Poly-Aromatic Hydrocarbons (PAH), Total Petroleum Hydrocarbons (TPH), metals, energetics, pyrotechnics, and pesticides. Samples are typically taken from defined areas within the area of interest, which is the NATO occupied land. The areas defined within the area of interest are established through the site planning process. These areas are typically referred to as decision units (DUs), Exposure Units (EUs), or sampling areas. Sampling of these areas is based on planning processes such as the data quality objective (DQO) process used by the US EPA, which establishes statistical limits to be applied to the analytical data obtained from the samples (Ref. d). This section of the document provides guidelines for the collection and shipment of samples from a DU that will help meet the DQOs for the contaminants of concern in soils.

3.2. SITE SURVEY

Site surveys are qualitative assessments used to physically characterize a site for future planning purposes. Two copies of the site plan (or a rough map of the general site layout indicating major landmarks or reference points) for each location should be available for mark-up. On the map, indicate the following, ensuring that accurate MGRS locations are indicated as well as the location of photographic evidence:

- General site layout – indicate accommodation, parking, administration, workshop, maintenance, POL, and medical areas or facilities;
- Surface characteristics – indicate areas of soil types/vegetation cover, surface water drainage pathways; visible staining of surface soil and/or vegetation stress; and
- Locations where NATO may potentially have a negative impact on the environment (e.g. POL storage and handling locations).
- A site survey must also take into consideration previous area use if relevant and other possible sources of contaminants natural or manmade, and decide what reference samples should be taken.

3.3. DECISION UNITS (SAMPLING AREAS)

Samples will be collected within the perimeter of a NATO Camp as detailed below. The decision units and the quantity of samples to be collected will be determined based on site usage and a general risk assessment.

Decision units are the area/volume of a site from which samples will be collected and a decision made. In most cases, the DU covers the likely extent of contamination expected from an activity or point source and its boundaries derived from the site survey and/or conceptual site model (CSM), a model of the conditions and the physical, chemical and biological processes that control the transport, migration and potential impacts of contamination to human and/or ecological receptors. In other cases, the DU boundaries are defined by the risk presented to a particular receptor (e.g., grazing area of a particular at-risk species) and are also referred to as an Exposure Unit (EU). DU selection is an integral component of the systematic planning process and should be tailored to the site in order to address the study question (i.e., DQO).

A given site can have multiple DUs to cover multiple suspected contamination activities or sources. If a DU is overly large in area or complex, it may be subdivided into sampling units (SUs) to be sampled separately and together inform the decision.

The area boundaries of a DU should be determined by the CSM and informed by sources such as the site survey, historical site records, local knowledge, and satellite/aerial imagery. The nature of the suspected contaminant and site topography/hydrology should dictate expansion of the DU, and/or addition of separate DU/SU, to include areas where the contaminant may have mobilized. Care should be taken to avoid over-estimating the DU boundaries, as expansion into clean areas will effectively underestimate the mean contaminant concentration.

The vertical extent of soil DUs strongly depend on the nature of the contaminant and the site soil type. Contaminants released as a liquid (e.g., diesel fuel, PCBs, firefighting foam) will likely migrate deeper into soil than contaminants released as solids (e.g. explosives, propellants, small-arms metals). Contaminant-specific solubility and interaction with soil also govern vertical migration and extent. Organic-rich soil tends to bind hydrophobic contaminants (e.g., TNT), clay-rich soil tends to bind cationic contaminants, and certain minerals complex some contaminants (e.g., Pb). Also, the age of the contamination will influence its extent. As time progresses, contaminants will have more time to dissolve, oxidize, metabolize and migrate further and deeper from its source. Finally, site use changes can affect burial depth, such as construction, paving, and explosive training (e.g., cratering). All of these factors should be considered when selecting soil sampling depth, for example just collecting surface soil (e.g., 0 to 6 cm depth) vs. deep soil coring (e.g., 0 to 1 m or deeper). The aim is to go as deep as the contamination goes until the last sample is clean which is important for the clean-up method selection as well as the cost estimation.

Table 3.1. Example target analytes for a generic NATO camp or base.

Site/Activity	Analyte/Potential Contaminant
Fuel Point	TPH
Garage / Motor Pool	TPH, Hydraulic Fluid, Lubricants
Power Generation	TPH, Hydraulic Fluid, Lubricants, PCBs
Firefighting Training Area	PFAS, PAH
Disposal Site	TPH, VOCs, SVOCs, PCBs, Energetics, Metals
Firing Point	Energetics, metals
Target Berm	Energetics, metals
Landfill	TPH, VOCs, SVOCs, PCBs, Energetics, Metals

The decision to perform remedial action is often made through comparison with a reference or acceptable concentration. For this comparison, the 95% upper confidence level from many discrete samples or the mean contaminant concentration from MI samples are commonly used. In cases where naturally occurring or pre-existing contamination may be present, background sampling should be performed. The DUs for background samples should similarly follow the CSM and same sampling procedures to capture contaminant concentrations in undisturbed areas.

For planning purposes, the following DUs should be established and sampled:

- POL and maintenance facilities;
- power generation location(s);
- accommodations;
- kitchen/ablution/mess locations;
- wastewater plant location;
- any industrial building located within the site; and
- locations that appear to be stained or contain other evidence of possible contamination, such as scrap metal or munition residues (e.g. firing ranges, locations where firefighting activities have taken place);
- At locations that have potential liquid contaminants (such as POLs and VOCs) and/or water soluble contaminants (such as phosphorus and nitrates), the DU or an additional DU should include additional area down gradient of the site.

3.4. SAMPLING METHODS

Sampling methods vary according to the information that can be derived as well as the quality of the data. Table 3.2 below describes the three most common sampling methods used to sample soils, descriptions of the methods, their applicability to various sampling situations, and a comparison of the characteristics for the various methods.

It will be the responsibility of the military engineer or NATO environmental officer to determine how much data is required, what the data will be used for, and the quality of that data, to decide which sampling method or methods are to be used.

Table 3.2 - Sampling methods for characterization of a NATO operations site

Sample Method	**Sampling Method Description**	**Applicability**	**Characteristics**
Grab (discrete)	A grab sample is a discrete sample that is collected at a specific location at a certain point in time.	Grab samples can be used to collect preliminary information at a specific point within a contaminated location to help determine the presence of a contaminant. **Best used for recent spillage of contaminants such as VOCs, POLs, and PAHs prior to soil excavation.**	Grab samples can only provide a "snapshot" of information at a particular point. Grab samples represent only the material at the point of sampling, typically not greater than 1 square meter. If the distribution of a contaminant in an area varies spatially, a single grab sample is not representative of that area. It is not recommended to take grab samples from any soil that may have been excavated prior.
Composite	Composite samples consist of multiple increments (>25) taken at different locations within a decision unit. Composite samples have a minimum mass (>500g). **Reference ISO 10381-1 through 5.**	Composite samples can be used to provide a rough estimate of contaminant concentrations within a DU. **Can be used in limited areas containing evenly-distributed solid (metals, energetics) or liquid (VOCs, POLs, pesticides) contaminants.**	A composite sample is more representative of a decision unit than a single grab sample but is not as effective as a multi-increment sample in estimating mean concentration levels throughout the decision unit. Composite samples are not effective for delineating extent of contamination in an area of interest.
Multi-Increment	Multi-increment (MI) samples consist of a combination of many (>100) individual soil increments of the same geometry and mass taken from evenly distributed locations throughout the decision unit to form one representative sample. MI samples have a minimum mass (>1 kg). **EPA SW846**	MI sampling is used to provide reproducible estimates of mean contaminant concentrations within a decision unit. **Best used for contaminants that are in particulate form, such as energetics and metals, but may be used for liquid contaminants (although not advised for VOC's). This is especially**	MI sampling will represent a DU more accurately than a composite sample, because the number of increments that makes up the sample reduces the distributional error of the contaminant. Data from replicates obtained within a DU using MI sampling are more normally distributed and typically meet data quality objectives. MI sampling can be used to delineate the extent of contamination in an area of interest.

	Method 8330B. AVT-197, AVT-244.	**relevant to excavated soil.**	Incremental Sampling Methodology (ISM) is a structured composite sampling protocol having specific elements designed to reduce data variability and increase sample representativeness. Properly executed, ISM provides unbiased, reproducible estimates of the mean concentration of analytes in the specified volume of soil (Einax and Kraft, 2002)[1]

3.5. IN FIELD TESTING METHODS

An array of in-field soil tests are commercially available that may provide qualitative or quantitative information to guide further sampling for lab analysis or to even make decisions. Such tests may particularly be useful in cases where the site history and the type and extent of contamination are unknown. Some of the field test results should be verified by laboratory analysis, for quality control.

Field tests and field measurements could also be used to help with the characterization of the extent of a pollution and thus for determining the DU/SU's (e.g., PID-measurement of fuel related VOC's for a fuel pollution). A key consideration in deploying these in-field tests and evaluating their results is the scale of the measurement relative to the decision unit. Many devices and kits have small areas of interrogation (e.g., X-Ray Fluorescence [XRF] is influenced by variables like soil moisture and soil texture. In field sample preparations may improve accuracy. One particularly useful application of in-field testing is identifying point sources of contamination, for example pieces of suspected explosives or bullets, and heterogeny distribution of contaminants. Common field tests include XRF and Laser Induced Breakdown Spectroscopy for metals, colorimetric assay and Raman Spectroscopy for explosives, immunoassays and turbidimetry for petroleum hydrocarbons. In general, field tests have higher detection limits and are more susceptible to interferences than laboratory tests, which should be considered in result interpretation.

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[1] Einax JW1, Kraft J., Small-scale variability of metals in soil and composite sampling, Environ Sci. Pollut. Res. Int. 2002, 9 (4) : pp. 257-61.
Hewitt. A, Ramsey, C. Bigl, S., Multi-increment TCE vadose-Z one Investigation, Remediation Journal, December 2008, Wiley Interscience publications, doi.org/10.1002/rem.20196, pp. 125-140.
Walsh, M. E., Ramsey, C. A , Collins, C. M., Hewitt, A.D., Walsh, M. R., Bjella, K. L., Lambert, D. J., Perron, N. M., Collection Methods and Laboratory Processing of Samples from Donnelly Training Area Firing Points, Alaska, 2003, ERDC/CRREL TR-05-6, March 2005, pp. 1-74.
The Interstate Technology & Regulatory Council, Incremental Sampling Methodology February 2012, https://www.itrcweb.org/ism-1/pdfs/ISM-1_021512_Final.pdf (consulted December 2018).

3.6. INFLUENCE OF SOIL TYPES ON SAMPLING METHODS

Tools for surface soil sampling should collect to an accurate depth and retain all soil particles within the removal volume. For MI sampling, where many increments comprise the entire sample, it is essential to maintain consistent soil removals with each increment. In cohesive soils, easy and consistent increments of the soil surface (up to 6-cm depth) can be collected with the CRREL Multi-Increment Sampling (CMIST) tool (Walsh, 2004 & 2009). This tool has variable stainless steel core heads and a variable sampling depth that can collect increments at a near walking pace. In less cohesive soils, coring tools such as the CMIST may not retain all soil particles and can be substituted with a manual tool, ideally with a flat bottom to retain equal depth throughout the sample/increment area.

Deeper soil sampling (i.e., > 10 cm) can be achieved with a driven coring system (e.g., slide-hammer, direct push, and vibracore), auger tool, or even excavator bucket. Complete recovery of soil from a given depth interval using these different systems depend highly on the system and the soil type, and likely require field validation prior to sampling.

3.7. VOC SAMPLING

Volatile organic compounds (VOCs) are a class of organic compounds that contain carbon and are emitted into the air from a variety of processes or as a product of a chemical reaction. Depending on their identify, they can be toxic, carcinogenic, and have short- and/or long-term health effects. VOCs are often present in groundwater and soils and represent a major environmental public health issue for many contaminated sites. As the name suggests, certain organic compounds possess high volatility and low boiling points (typically characterized by less than or equal to 250°C), which makes them especially susceptible to partitioning from solution to gaseous phase. Commonly found VOCs in groundwater include trihalomethanes (THMs) like chloroform, tetrachloroethylene (PCE), trichloroethylene (TCE), toluene, benzene, vinyl chloride, and methyl tert-butyl ether (MtBE). Common sources of VOCs in the environment are derived from paint, cleaning products, solvents, and fuels (gasoline, diesel, petroleum, etc.) [2]

Analyzing for VOCs in groundwater, water, or soil matrices typically consists of a preconcentration step since most VOCs are present at dilute levels. Sampling for VOCs can be challenging in the field due to the low concentrations and the propensity for VOCs to partition to gases and vapors at ambient air conditions necessitating the

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[2] Zogorski J.S., Carter J.M., Ivahnenko T., Lapham W.W., Moran M.J., Rowe B.L., et al. The Quality of Our Nation’s Waters—Volatile Organic Compounds in the Nation’s Ground Water and Drinking-Water Supply Wells. Circular 1292. Reston, VA: U.S. Geological Survey; 2006.

sampler to containerize the sample quickly to prevent evaporation[3]. For the purposes of this review, sampling for VOCs will primarily focus on groundwater and soil matrices. This is a general guide for sampling for VOCs in non-well environments and specific VOCs should be confirmed with the analytical testing method as to the appropriate sample collection/pre-concentration protocols and whether there are any interferences with other analytes that may be present in the samples. It is important to characterize each field site and sampling location prior to sample collection and gather as much information as possible about (1) what types of contaminants may be present, (2) in what concentration ranges, and (3) any other site information, i.e., historical information, pH, oxidation-reduction environment. This type of information plays a role in the overall sample collection best practices and which preservation agent to employ, etc.

Step-by-step guide for sampling VOCs in groundwater:

1. Sampler puts on nitrile gloves
2. Do not open sample bottle until it is ready to be filled and be sure to minimize opening the sample bottle near sources of VOCs (vehicle exhaust, heavy equipment, generator, etc.).
3. Typical sample bottles consist of a 40 mL amber purge-and-trap vials that are filled leaving a positive meniscus at the top and free of air bubbles, as shown in Figure 1. Depending on the type of VOC sampled for and whether the source is chlorinated or unchlorinated (mostly for drinking water samples) there may be additional preservation agents added prior to filling the vial with the groundwater sample, including ascorbic acid, hydrochloric acid, maleic acid, and sodium thiosulfate.
4. Sampler fills the vial completely, caps the vial, and inverts to determine if there are any air bubbles present. If there is an air bubble, discard sample and fill a replacement until there are no bubbles present if there is no preservative in use. If there is a preservative in the bottle then add more sample until a positive meniscus is present and re-cap, checking for air bubbles until none are present.
5. Store at less than or equal to 4°C until analyses, do not freeze the sample. Sample hold times are approximately 14 days and instrument time should be checked and/or shipping methods to an analytical laboratory should be

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[3] Hewitt A. D. Losses in trichloroethylene from soil during sample collection, storage and laboratory handling; U.S. Army Cold Regions Research and Engineering Lab, Hanover, NH; 1994; SR94-8.

previously established before collecting the groundwater sample for VOCs as the shelf-life is relatively short.

Step-by-step guide for sampling VOCs in soil:

1. Sampler puts on nitrile gloves
2. Soil samples for VOCs characterization are typically collected in wide-mouth amber jars or corers with acrylic liners. Soil sample preservation agents include methanol or sodium bisulfate depending on how dilute the VOCs are expected to be in the soil. Some core samplers have a sealed chamber and do not require preservation agents like methanol, etc.
3. Soil samples for VOCs require knowing the mass of the soil, typically 10 to 25 grams (non-core sample). If collecting soils using a jar (non-core sample), collect a known amount of soil (25 grams, for example) in a wide-mouth amber jar.
4. Cap the sample collection container (bottle, corer, etc.) as soon as possible after soil removal as volatilization of VOCs occurs within seconds of exposure to ambient conditions.
5. Store the soils at 4°C for up to 14 days prior to analyses.

3.8. **SAMPLE COLLECTION**

The following equipment will be required to collect surface soil samples:

- Field notebook;
- Camera and GPS;
- Nitrile gloves;
- Shovel and pickaxe (depending on soil composition);
- Soil sample tools;

- Sample containers (varies depending on analysis required);
- Deionized water;
- Cooler and ice packs; and
- COC form and tape.
- Face masks;
- Plastic bags for waste collection;
- Equipment for hands washing after soil collection.

Sample collection requires adherence to sampling protocols to prevent cross-contamination and to demonstrate the validity of the laboratory analysis. The following key elements must be followed and are explained in more detail in the subsequent sections:

- Wear nitrile gloves at all times when collecting samples;
- Wear face mask when (suspected) presence of soil borne diseases (e.g. cocciidioidomycosis) or contaminants (e.g. asbestos) that could be inhaled;
- clean sample equipment with deionized water between sample locations (eventually use a mild detergent for cleaning in-between sample sites);
- keep samples cool during sample collection and transportation; and
- take surface soil samples to a minimum depth of 3 cm to 10 cm;
- take a quality control (QC) sample at a non-suspected area in order to have a representation of the initial soil quality.

3.9. PERSONAL PROTECTIVE MEASURES FOR THE SAMPLING TEAM

When undertaking soil sampling it is important to verify suitable and adequate personal protective equipment is in place prior to undertaking field sampling. When selecting protective equipment there a variety of variables to consider, such as:

- The terrain and location of the site to be sampled;
- Weather at the time of sampling;
- Other hazards that may occur during sampling.

Specific hazards may be associated with the actual contaminant in the soil or surrounding environment, e.g. vapors, gases, and particulates from hazardous substances but also hazards related to eventual use of preservatives and/or reactants should be considered. For this reason, personnel undertaking sampling may be exposed to hazardous substance response type activities. Therefore, personnel must wear appropriate personal protective clothing and equipment whenever they are in and near the sampling site. Also, the more that is known about the hazards at the sampling site prior to the actual sampling, the easier it becomes to select appropriate personal protective equipment. Typically, national Occupational Health & Safety regulations will apply to personal protective measures.

The US Environmental Protection Agency has four levels of personal protective equipment and provide a good illustration of protection of personnel:

- **Level A protection** is required when there is greatest potential for skin, ingestion, inhalation or eye exposure to hazards, and therefore requires highest level of skin, respiratory, and eye protection.
- **Level B protection** is for circumstances requiring the highest level of respiratory protection, with lesser level of skin protection. At most abandoned outdoor hazardous waste sites, ambient atmospheric vapors or gas levels have not approached sufficiently high concentrations to warrant level A protection.
- **Level C protection** is required when the concentration and type of airborne substances is known and the criteria for using air purifying respirators is met.
- **Level D protection** is the minimum protection required. Level D protection may be sufficient when no contaminants are present or work operations preclude splashes, immersion, or the potential for unexpected inhalation or contact with hazardous levels of chemicals.

Levels A to D are general guidelines for protection and Table 3.3 summarises typical equipment to be used when exposed to these hazardous substance response activities. However, other combinations of protective equipment may be more appropriate, depending upon specific site characteristics. In addition, the completion of pollutant hazard risk assessment prior to work is essential to ensure Safety Data Sheets (SDS) are obtained for all known and potential pollutants (and other chemicals used during the field sampling) as level of protection needs to be for the highest risk contaminant.

Table 3.3 Summarises suggested protection for hazardous substance response activities, including soil sampling according to the US EPA (source: https://www.epa.gov/emergency-response/personal-protective-equipment)

Protection Level	Suggested personnel protective equipment for hazardous substance response activities
A	• Positive pressure, full face-piece self-contained breathing apparatus (SCBA); • Totally encapsulated chemical- and vapor-protective suit; • Inner and outer chemical-resistant gloves; • Disposable protective suit, gloves, and boots.
B	• Positive pressure, full face-piece self-contained breathing apparatus (SCBA) or positive pressure supplied air respirator with escape SCBA; • Inner and outer chemical-resistant gloves; • Face shield; • Hooded chemical resistant clothing; • Coveralls; • Outer chemical-resistant boots.
C	• Full-face air purifying respirators; • Inner and outer chemical-resistant gloves;

	• Hard hat; • Escape mask; • Disposable chemical-resistant outer boots.
D	• Gloves; • Coveralls; • Safety glasses; • Face shield; • Chemical-resistant, steel-toe boots or shoes.

3.10. SAMPLE LABELING

For each sample, the following information should be annotated on the sample tag and in the sample record (See Sections 2.3, 2.4):

- The site-specific identifier (Decision unit, grab sample point, or sampling area designation);
- sample method;
- date taken;
- initials of sampler;
- numeric identifier;
- GPS coordinate for grab or composite sample increment points;
- number of increments for multi-increment sampling;
- replicate number (if applicable).
- use of preservatives, if applicable; and
- any pertinent details related to the specific sample location.

3.11. REPLICATE SAMPLES

Replicate samples are taken to determine if DQOs have been met. Specifically, replicates demonstrate whether or not the samples taken within a specific DU are reproducible. Replicate samples are NOT the same as field splitting of a sample. A replicate sample is a unique sample within a DU that is taken in the same manner as the original sample. Replicates should be taken on a regular basis during EBS and ECS sampling. The number of replicates in a specific DU should be at least three and care should be taken to not co-locate replicate increments. The number of replicates and the number of DUs requiring replicate sampling is a function of the DQOs and the judgment of the environmental officer. Replicate sampling allows statistical analysis of the results and will reinforce the data in the EBS and ECS, making the analytical results more defensible. Replicate sampling is most effective with MI sampling (typically MI sampling requires 2 replicates additional to the original sample), should be done with composite sampling (typically 1 replicate), and are of limited value (but should be done) when taking grab samples (typically 1 replicate).

3.12. COOPERATION FROM THE LAB

While planning for fieldwork, a good dialog with the lab performing the processing and analyses of your samples is essential. To obtain reproducible analytical results, the samples that reach the lab must have the right quality, have sufficient mass, and be stored in the right type of containers. Also, obtain a list of available analytical options, their detection limits, necessary sample containers, preservation, time limitations, and other relevant factors. The lab may also need a notification before samples are shipped. This is important for your sampling strategy and quality of results. It may not be possible to replace inadequate samples. Do also check if there are local lab facilities.

3.12.1. Sample Processing Request

Samples taken in the field should mirror the areas from which they are taken. As such, samples taken from DUs with heterogeneously distributed contaminants will have contaminants heterogeneously distributed within the sample. This section applies to samples containing non-volatile (e.g. metals) and semi-volatile (e.g. energetic) contaminants. Prior to analysis, the contaminant must be extracted from the soil matrix of the sample. There are two methods to reproducibly obtain a representative extract: Whole sample extraction (slurry extraction) and subsampling of a properly ground sample.

Slurry extraction requires large amounts of solvent but does not require any specialized equipment. A shaker is sometimes used to facilitate the process. Care must be exercised to ensure the slurry does not heat up enough to vaporize volatile analytes during the agitation process. Extracts for analysis are typically filtered and finished processed prior to injection in analytical instrumentation.

Subsampling prior to extraction requires proper grinding of the samples to reduce the particle size of the matrix and contaminant (comminution) and mix the contaminant more uniformly throughout the matrix (blending). A properly ground sample significantly reduces the error associated with subsampling, allowing reproducible subsamples to be taken from the sample. Sample grinding requires specialized a specialized piece of commercial grinding equipment, a puck grinder, but generates much less hazardous waste in the extraction process and tends to be much quicker.

Samples should never be subsampled by scooping a small mass from the unprocessed sample. Samples should never be field split or split prior to grinding. A processing request should be included in the sample shipment specifying the type of processing needed to ensure error reduction on the subsampling process.

3.12.2. Analytical Request

Grab samples will be collected and analysed for VOCs. Samples collected at locations with potential POL contamination should be analysed for TPHs. Other samples that are collected based on a perceived hazard (i.e. significant staining/vapours) should have an analytical request for the contaminant of concern (i.e. solvent, metals, etc.).

3.13. SHIPPING CHECKLIST

Samples shall be placed into a sturdy container that will protect the samples during transport. Transport all samples in accordance with guidance from the receiving laboratory. The following general procedures apply to the packaging of all environmental samples:

a. Verify that each sample is securely sealed. For samples in jars or bottles, ensure the cap or lid is secured on the bottle. Place the jar in a clean plastic bag and seal for shipping before placing in the shipping container. For samples in bags, double bag the original sample bag. Label the bag and attach a tag containing sample identification written on the sample bag. Evidence tape or custody seals may be placed over the sample lid and container or over the seal of the bag for additional security. If using numbered seals, record each seal number in the notebook and if more than one shipping container is used, note on which container the seals are.
b. Prepare the shipping container for use. For a commercial cooler, this includes taping the drain plug shut inside and out. Place up to 10 cm of inert packing material in the bottom of the cooler. Add inert packing material as necessary to ensure separation of samples.
c. All environmental samples should be shipped to the laboratory on ice and chilled to max 4°C. If max/min thermometers are available, they should be place inside the cooler to determine if samples have been kept cool.
d. Place the paperwork (COC, processing request, and sample analysis request) being sent to the laboratory inside a plastic bag and tape it to the inside of the cooler lid. Close the cooler and seal it with strapping tape. Write your initials across the edge of the tape. Place at least two custody seals on the outside of the cooler (one on the front and one on the back). More custody seals may be used at the discretion of the sampler. Place appropriate labelling on exterior of the cooler. Place a copy of the import permit on exterior of the cooler. Keep the original waybills for your records.

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CHAPTER 4 WATER SAMPLING

4.1. GENERAL

Water sampling may be conducted as part of the EBS process. The water sampling could consist of representative sampling of surface water sources or existing groundwater sources (wells). The field procedures (sample handling, storage, analysis, documentation and shipment) are similar to those outlined above. This section will deal specifically with the field protocols for the capturing of water samples. In addition to the equipment identified above as required for soil sampling, the following equipment may be required for surface or groundwater samples:

- Surface water sampling device (isokinetic suspended sediment sampler);
- Peristaltic pump and power source;
- Polyethylene tubing; and
- 0.45 mm filters.

4.2. SURFACE WATER SAMPLING

Surface water samples will be taken from surface water sources on or near the proposed location. This may include streams, rivers, or ponds. The samples will be collected by dipping the collection bottle directly into the surface water source if the site permits. For a stream, a cross section of the water channel should be sampled. For non-moving waters, such as ponds and lakes, a profile of the water column must be obtained to address possible stratification of contaminants. An alternate method is to use a peristaltic pump to collect the surface water sample. Again, a cross section or a profile of the water body must be sampled. If a filtered sample is required due to high levels of sediment, use a 0.45-mm filter in conjunction with a peristaltic pump and polyethylene tubing. If VOC analysis is required, the sample will be decanted from a glass bottle. When sampling water for metals, the sample is usually filtered prior to the addition of the preservative (nitric or sulfuric acid) prior to shipment.

4.3. GROUND WATER SAMPLING

Groundwater samples will be taken from existing onsite wells. Avoid taking sample from newly assembled ground water well; if possible, allow to settle for 1 – 2 weeks before the first sampling. Sampling will normally be conducted using a bailer, an inertia pump, a submersible pump, a peristaltic pump or a bladder pump (if water level in the well is deeper than 8 m). The three first methods are considered high flow and a volume of groundwater comprises between 3 to 5 volumes of the standing water in the tubing of the well and the sand pack around the screen must be removed from the well before taking the groundwater sample. A more accurate method (Puls and Barcelona, 1995) involves the use of a peristaltic pump or a bladder pump at a low flow rate (100 to 500 mL/min) connected to a flow through cell where the field parameters (temperature, pH, electrical conductivity, dissolved oxygen, oxidation-reduction potential) are measured continuously with a multi-parameter probe in the purge water up to stabilization. These flow rates in combination with a maximum drawdown of 10-15 cm are allowed into the well to avoid bringing particles that are not part of natural groundwater flow. The groundwater sample is taken only after the variations of less than 0.1 pH unit, 3% on electrical conductivity and 10% on dissolved oxygen are recorded into the purge water. The water intake is usually placed at depth into the well in the middle of the screen or its upper part. The composition of the samplers must be compatible with the parameters that need to be analyzed. Teflon, viton and stainless steel components are preferred for organics analysis in groundwater whereas high density polyethylene (HDPE) can be good for metals. A dedicated sampling device (e.g. Teflon and viton tubing) is preferred as one that need to be washed between each sampling location (well). The procedure for the storage and shipment of samples will follow the same protocols as indicted above. The decontamination procedure implies the use of an acid (HCl 10%) to remove trace of metals, distilled water to rinse the acid, acetone to remove trace of organics (e.g. energetic material) and distilled water to remove the solvent. This procedure is require for all equipment that comes in contact with groundwater in the well such as the sampler and the water level measurement tape. All water samples must be kept cool (<4°C). It is important to consult the appropriate environmental specialist or the laboratory conducting the analysis to determine the quantity and type of bottles and preservative required.

4.4. REPLICATES

It is important to follow the protocols for the collection of field replicates for analytical purposes. Usually, 10% of the groundwater samples should be duplicates. If the resources exist, the preparation of field blanks should also take place during sampling.

CHAPTER 5 ADDITIONAL INFORMATION

5.1. IMPORT PERMITS

National policies may require a soil or water import permit in order to import any soil or water samples back for analysis. Ensure this is obtained prior to conducting any sampling.

5.2. CHAIN OF CUSTODY FORMS

The COC form will be used between the sampling team and the laboratory conducting the analysis. The form will be used as a quality assurance measure in sample handling and transport. The key components of the COC form will include the laboratory analysis requested, specific preservative requirements and the sample identification. There is no NATO standard COC form; therefore, nations will use national COC forms for sample handling and transport.

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CHAPTER 6 REFERENCES

The following references were used in developing this Standards Related Document:

a. Einax JW1, Kraft J., Small-scale variability of metals in soil and composite sampling, Environ Sci. Pollut. Res. Int. 2002, 9 (4): pp. 257-61.

b. Hewitt. A, Ramsey, C. Bigl, S., Multi-increment TCE vadose-Z one Investigation, Remediation Journal, December 2008, Wiley Interscience publications, doi.org/10.1002/rem.20196, pp. 125-140.

c. Hewitt A. D. Losses in trichloroethylene from soil during sample collection, storage and laboratory handling; U.S. Army Cold Regions Research and Engineering Lab, Hanover, NH; 1994; SR94-8.

d. The Interstate Technology & Regulatory Council, Incremental Sampling Methodology February 2012, https://www.itrcweb.org/ism-1/pdfs/ISM-1_021512_Final.pdf (consulted December 2018).

e. ISO 10381-1 through 5. Soil Quality-Sampling. https://www.ISO.org

f. Ramsey, CA, and C Wagner, (2015) Sample Quality Criteria. Journal of AOAC International. Vol. 98, no. 2, pg 265-268

g. Rasemann, W. (2000) Industrial waste dumps, sampling and analysis. In: Encyclopedia of Analytical Chemistry (Meyers, R.A., Ed.) John Wiley and Sons, Ltd, Hoboken, NJ USA.

h. Walsh, MR, and ØA Voie, eds (2016) Munitions-Related Contamination – Source Characterization, Fate, and Transport. NATO STO Technical Report TR-AVT-197 [AC/323(AVT-197)TP/668]. NATO Science and Technology Office, Neuilly sur Seine, France.

i. Walsh, MR, and ØA Voie, eds (2015) Munitions-Related Contamination. NATO STO Meeting Proceedings MP-AVT-197 [AC/323(AVT-244) TP/659]. NATO Science and Technology Office, Neuilly sur Seine, France.

j. Walsh, MR (2009) User's Manual for the CRREL Multi-increment Sampling Tool. ERDC/CRREL Special Report SR-01-1. USA CRREL, Hanover, NH USA.

k. US EPA (2006) Guidance on Systematic Planning Using the Data Quality Objectives Process QA/G-4. EPA/240/B-06/001. Washington, DC: US Environmental Protection Agency. See: https://www.epa.gov/sites/production/files/documents/guidance_systematic_planning_dqo_process.pdf (Accessed 18 APR 2018).

l. US EPA Method 8330B (SW-8346). Nitroaromatics, Nitramines, and Nitrate Esters by High Performance Liquid Chromatography. Revision 2, USEPA 2006 Washington, DC: US Environmental Protection Agency.

m. Walsh, M. E., Ramsey, C. A, Collins, C. M., Hewitt, A.D., Walsh, M. R., Bjella, K. L., Lambert, D. J., Perron, N. M., Collection Methods and Laboratory

Processing of Samples from Donnelly Training Area Firing Points, Alaska, 2003, ERDC/CRREL TR-05-6, March 2005, pp. 1-74.

n. Zogorski J.S., Carter J.M., Ivahnenko T., Lapham W.W., Moran M.J., Rowe B.L., et al. The Quality of Our Nation's Waters—Volatile Organic Compounds in the Nation's Ground Water and Drinking-Water Supply Wells. Circular 1292. Reston, VA: U.S. Geological Survey; 2006.

ANNEX A SAMPLING METHODS

A.1 INTRODUCTION

The objective of Annex A is to familiarize the reader with the purposes for sampling, how to reduce error in samples, and how to sample using the methods outlined in Table 3.1. This Annex will present basic information and procedures. For more detailed information on the subject matter, the reader is directed to the references at the end on the Annex.

A.2 PURPOSES FOR SAMPLING

The basic objective for sampling in the context of environmental characterization and protection is to obtain reproducible, robust data about contamination for a specific point or a delineated land area or water feature. There are two related needs for this data: to protect the environment and to protect NATO from liabilities associated with use and possible contamination of an operations site. In both cases, the data needs to be scientifically based and legally defensible to avoid unnecessary remediation or to ensure that no remediation is necessary. To be defensible, the error associated with the sampling must be controlled to meet data objectives and the sample and analytical data must be reproducible.

A.3 SAMPLING ERROR

Data quality is a function of the cumulative error associated with the characterization process. Error sources are many, but the main sources are associated with sample acquisition, sample processing, and sample analysis. Sample processing will be discussed in this Annex as it is closely related to activities that could occur in the field.

Rasemann, in his study on error associated with characterizing contaminated material, states that sampling is the single largest source of error in an environmental characterization project (Table A3.1). It is obvious from this table where the greatest potential source for error lies. It is also obvious where the greatest effort needs to be expended to reduce characterization error. However, for most projects, the quality assurance (QA) efforts are concentrated on the analytical phase of the process, after the major errors have already progressed through the process.

Table A3.1 - Three common sources of measurement error

Activity	Error (% of true value)
Sampling	1000%
Sample preparation	100—300%
Analytical measurement	2—20%

There are two essential sources of error when sampling. The first, fundamental sampling error (FSE), is caused by the non-uniform composition of each particle in the DU or sample. To correct for FSE, sufficient mass must be collected to ensure that the contaminant is properly represented within each sample. The second, Grouping and Segregation Error (GSE), is caused by the non-homogeneous distribution of the contaminant within a volume of material and inconsistent particle sizes within that volume. GSE is addressed by taking a sufficient number of increments within the characterization area to reduce the distributional error and reducing the particle size distribution (comminution).

Another significant source of error during sample collection, materialization error, is caused by the tools used to collect the sample. Figure A3.1 illustrates the sample geometry resulting from the use of three different sampling tool types. The samples geometry shown in A3.1 (a) is the preferred configuration because it equally samples material throughout the depth of the sample. The geometry Illustrated in A3.1 (b) is less representative because the volume of the material collected decreases with depth, thus biasing the sample towards surface contamination. The geometry illustrated in A3.1 (c) only represents the surface contamination and does not consider material at depth. Therefore, a tool that obtains a cylindrical core-shaped increment, such as shown in Figure A.3.2, is preferred and should reduce materialization and thus sampling error. However, the type of tool that can be used will depend on the soil type, e.g. sand vs. organic soil vs. hard packed clay.

Figure A3.1 - Increment shapes: (a) core, (b) trowel, and (c) spoon.

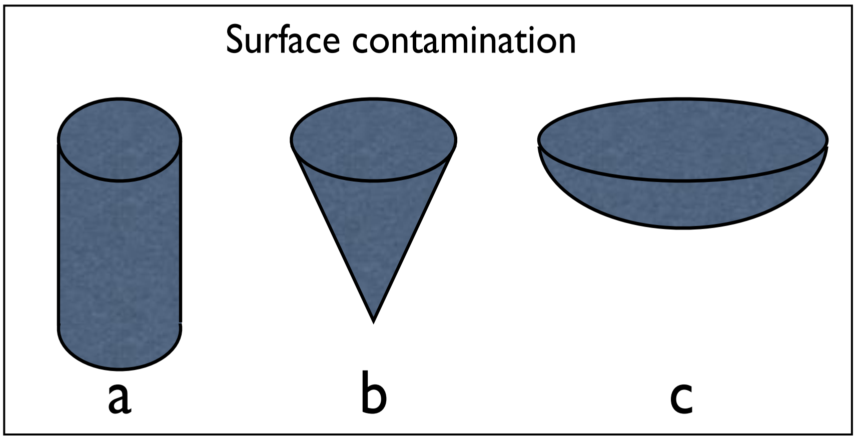


Figure A3.2. - An example of a sampling tool for obtaining uniform soil increments (CRREL).

A.4 SAMPLING METHODS

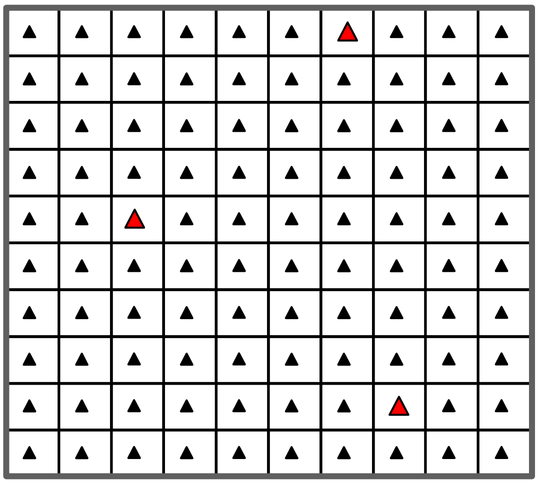
This section describes how to undertake the common soil sampling methods outlined in Table A3.1. It will include how to set up the sampling regimes, correct sampling tools, mass and number of increments required, sample replicates and sample handling. In addition, splitting and subsampling of samples in the field are described in Section A.5 as both processes adversely affect the quality of the field sample after collection.

A.4.1 Grab Sampling

Grab (or discrete) samples are taken to establish the presence of contamination at a physical point. Grab samples are best used for a spillage that has recently occurred. Grab samples should be taken within the known area of contamination as soon as possible after the spillage event. Each grab sample point needs to be recorded using a GPS. The most appropriate tools to use are a small spatula for VOCs or a coring tool for other contaminants, such as PAHs, POLs, TPHs or PCBs. For VOCs collect at least 5 g of soil. For other liquid based contaminants, the mass should be determined by the analysis requirements. Sample replicates may be taken, although their value will be extremely limited as each replicate represents only the soil taken from the point of excavation. However, if replicates are taken, then no less than five should be taken to enable statistics to be performed on the data. Samples containing liquid contaminants are usually collected, stored, and shipped in sealed glass jars, which are kept cool. Ensure samples are labelled correctly, as outlined in section 3.6.

Grab samples are of extremely limited value and can't be used to characterize a site or to derive a DU mass inference. Neither FSE nor GSE are controlled through the use of grab samples. Thus, grab samples should not be used for the determination of remediation decisions.

Figure A4.1 - Grab or Discrete Sampling within a Decision Unit (Sampling Area)



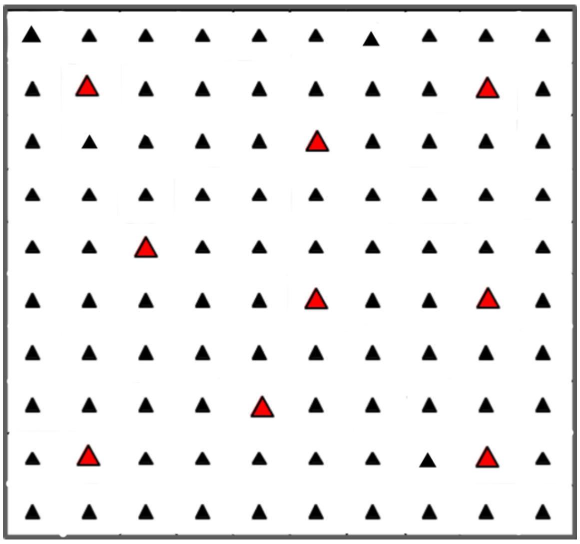
▲Possible collection points for Grab Sample (100 possible locations shown)

🔺Typically, only a 1 - 3 samples are collected for Grab Sampling

A.4.2 Composite Sampling

Composite samples are taken to establish the concentrations of contaminants within a defined area. Composite samples usually consist of five to nine increments but may consist of up to 25 or 30 increments (Figure A4.2). In either case, only a rough estimate of contamination levels can be determined in most cases. Each increment must represent an equal area of the decision unit. Increment locations should be decided prior to actual field work and a GPS used to locate where the increments are to be taken. Sample depth and cross-section should ensure that at least 500 g of material is collected, although collecting at least 1 kg is recommended. The most appropriate tools to use are a coring device, a post-hole digger, or a spade to ensure that the volume sampled is cylindrical in shape and consistent across increments. Sample replicates may be taken although the value is limited as there will be distributional error attributable to the small number of increments. Reproducibility of the samples is unlikely to satisfy data quality objectives if the contaminant is randomly distributed. Unless the contaminant is relatively uniformly distributed throughout the DSU, achieving reasonable relative standard deviations for the data, used in the determination of data robustness and reproducibility, is very difficult for most cases where composite sampling is used. For QA, at least three replicates should be taken from randomly-chosen DUs to allow statistical analyses. Samples are usually collected, stored, and shipped in sealed lab-grade clean plastic bags, which are kept cool. Ensure samples are labelled correctly, as outlined in Section 3.6.

Figure A4.2 - Composite Sampling within a Decision Unit (Sampling Area)



▲Possible collection points for 100 discrete locations

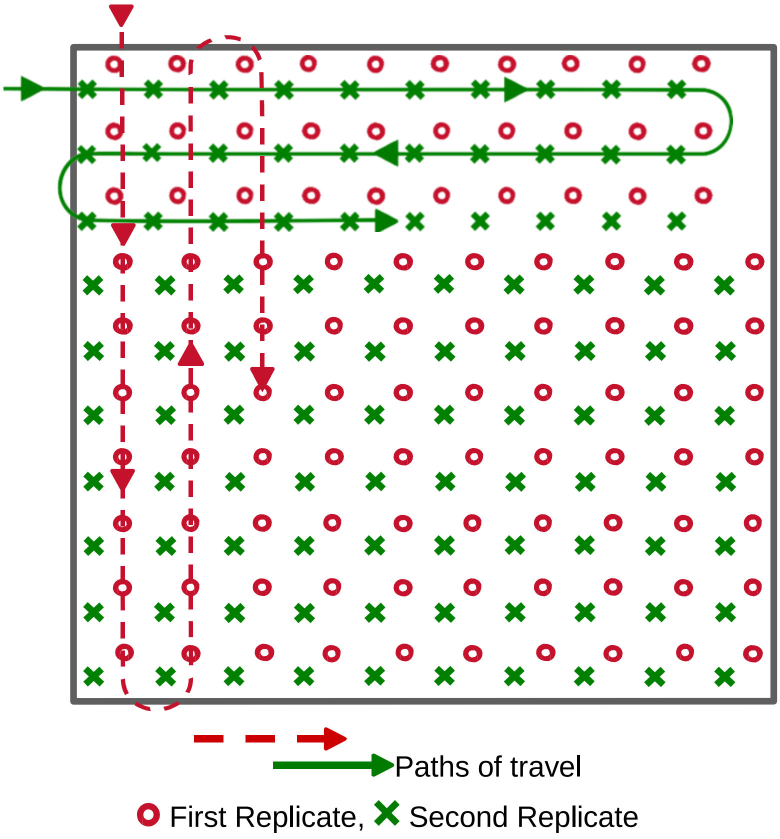
🔺Typically, only a 5 – 9 increments are collected for Composite Sampling

Composite samples are of limited value and should not be used to characterize a site (DU) or to derive an inference as to the mass of the contaminant in a DU. The limited number of increments and, typically, insufficient mass will lead to large FSE and GSE errors, diminishing both the reproducibility and robustness of the samples (RSD). Because of these errors, using composite samples for the determination of remediation decisions presents a high and often unacceptable risk.

A.4.3 Multi-increment Sampling

Multi-increment (MI) sampling is used to establish the concentrations of contaminants within a defined area or an excavated soil pile where there may be contamination. MI samples usually consist of 100 increments or more but may consist of less in a very small decision unit, e.g. 40 increments in a 4-m2 area (Figure A.4.3). Each increment must represent an equal portion of the decision unit. MI sampling employs a systematic-random approach. The decision unit is divided into a grid, the number of cells within the grid being equivalent to the number of increments required. The starting location in the first cell is randomly chosen and the increment locations in all the following cells are the same relative to the first cell. Sample depth and cross-section should ensure that at least 1 kg of material is collected. The most appropriate tool to use is a small coring device, which will enable the efficient collection of many consistently-sized cylindrical increments. The coring tool reduces materialization error by not biasing the material in the increment, collecting equal volumes of soil throughout the depth of the increment. Sample replicates may be taken by varying the randomly-assigned starting point in the first cell and resampling the area. Do not co-locate replicate sample increments with each other. FSE and GSE are addressed through the many consistently-sized increments that will add up to 1 kg or more in mass. Reproducibility of the samples is more likely to satisfy data quality objectives, even if the contaminant is randomly distributed. As above, at least three replicates should be taken from randomly-designated DUs to allow statistical analyses (e.g. RSDs) to be performed. Samples are usually collected, stored, and shipped in sealed lab-grade clean plastic bags, which are kept cool. Ensure samples are labelled correctly, as outlined in section 3.6.

Figure A4.3 - Multi-increment sampling (Replicate sampling)



Because MI sampling directly addresses both FSE and GSE, inferences can be made from the samples as to the mean concentration of the contaminant as well as deriving a reproducible estimate of the mass of the contaminant within the DU. RSD objectives are typically met by obtaining 100 increments per sample, although increasing the number of increments may be necessary for lower-concentration, more highly heterogeneously distributed particulate contaminants.

A.5 FIELD SPLITTING AND SUBSAMPLING

Field splitting and laboratory subsampling of a sample are two completely different actions and will provide very different information. Field samples will contain contaminants that are distributed within the sample matrix to varying degrees of heterogeneity, i.e. an MI sample will be no less heterogeneous than the area from which it was taken. Field splitting of a sample will not result in a reduced sample mass that represents the original unsplit sample and the DU from which the sample was collected. Many studies have proven this over the years. Subsampling done correctly occurs in the processing lab where the sample is properly ground or whole-sample extraction of the contaminant from the sample occurs. Studies have shown that subsampling a properly ground sample will result in consistent, normally distributed data. Comparisons between whole-sample extraction and subsampling of ground samples have shown very similar data for the same sample.

An important note: Unless specified, a processing or analytical lab will remove the required subsample from a sample received from the field from the top of the sample in only one location. At best, the sample may be dumped out and the subsample taken from one location. This is the equivalent of collecting the mass for the analytical subsample by going into the field and placing the required mass for analysis in a small jar from one location in the field. This will negate all the effort and care taken in the field to collect a representative sample. There will be no ability to conduct replicate subsamples when the sample is subsampled in this manner, and the analytical data will not represent what is present in the field.

Field splitting, whole-sample extraction, or subsampling will not result in field replicates of the samples. Subsampling and replicate extractions can be used to obtain replicate subsamples, but these are used for QA of the original samples and of the preparation and analysis portions of a characterization process. They are no substitute for the collection of replicate samples. Sample replicates are separate samples collected in the field in the same manner from the same area as the original sample.

A.6 SAMPLE PROCESSING

Although sample processing is outside the scope of this document, it is an important part of the characterization process directly related to the samples collected in the field. Incorrect sample processing will result in severely degraded data quality, including the inability to achieve representative subsamples of the original samples, and greatly reduce the value of the field samples. As is indicated in Table A3.1, errors of 300% of true values will result from processing errors. Soil grinding or whole-sample extraction can be used to process samples in the lab. To obtain a representative soil subsample, a puck mill that will grind the sample to a near-uniform consistency is required. The process is called comminution and distributes the contaminant more uniformly throughout the ground matrix than occurred in the original sample. Subsampling of the ground sample is performed in a similar manner as taking the original sample: Up to 30 increments are taken from the ground sample that has been spread out in a thin layer on a clean metal sheet plan. Replicate subsamples are obtained by offsetting the first increment and carrying the offset throughout the sample. Studies have indicated a high degree of reproducibility between subsamples of a properly ground sample, even at very low concentrations of contamination. For this reason, a processing request should accompany samples sent to the analytical lab.

A.7 SUMMARY

The process of obtaining representative and reproducible data for the characterization of contaminants on a site requires adherence to all procedures in that process. Fundamental, grouping and segregation, and materialization errors must all be controlled in order to reduce the global error estimate and obtain reproducible data. However, collecting the best possible samples will mean nothing if the samples are not properly processed and analyzed. For example, L. Penfold at TestAmerica Laboratories, Inc., in the US, conducted a metastudy on sample grinding to determine the relative standard deviation for different soil grinding methods (Table A.7.1). The data are quite clear: Using the wrong type of grinding equipment will greatly compromise the quality of the data. The sample holds true for other sample processes such as subsampling or extraction.

Table A.7.1. - Relative standard deviations for data from five soil grinding methods

Method	RSD
Automated Mortar	142 - 217%
Shaker Ball Mill	20 - 114%
Coffee Grinder	49 – 64%
Roller Ball Mill	27 – 30%
Puck Mill	<1 – 3%

This Annex should provide the reader with a basic understanding of the protocols used for the environmental sampling of water and soils. Carrying out these protocols will provide the best field samples for the next steps in the process of obtaining robust, reproducible, defensible, and cost-effective data.

A.8 REFERENCES

The following references were used in developing this Annex:

a. Rasemann, W. (2000) Industrial waste dumps, sampling and analysis. In: Encyclopedia of Analytical Chemistry (Meyers, R.A., Ed.) John Wiley and Sons, Ltd, Hoboken, NJ USA.
b. Voie, ØA, and MR Walsh, eds (2016) Munitions-Related Contamination – Source Characterization, Fate, and Transport. NATO STO Technical Report TR-AVT-197 [AC/323(AVT-197) TP/668]. NATO Science and Technology Office, Neuilly sur Seine, France.

c. Walsh, MR, and ØA Voie, eds (2015) Munitions-Related Contamination. NATO STO Meeting Proceedings MP-AVT-197 [AC/323(AVT-244)TP/659]. NATO Science and Technology Office, Neuilly sur Seine, France.
d. Walsh, MR (2009) User’s Manual for the CRREL Multi-increment Sampling Tool. ERDC/CRREL Special Report SR-01-1. USA CRREL, Hanover, NH USA.

ANNEX B LIST OF ACRONYMS

COC	Chain of Custody
CRREL	Cold Regions Research and Engineering Laboratory
DU	Decision Unit
DQO	Data Quality Objective
EBS	Environmental Baseline Survey
ECR	Environmental Condition Report
ECS	Environmental Closeout Study
EHSA	Environmental Health Site Assessment
EPA	Environmental Protection Agency
EU	Exposure Unit
GPS	Global Positioning System
ISO	International Standards Organization
MGRS	Military Grid Reference System
MI(S)	Multi-Increment (Sampling)
NATO	North Atlantic Treaty Organization
PAH	Poly-Aromatic Hydrocarbon
PfP	Partnership for Peace
POL	Petroleum, Oil, Lubricants
QA	Quality Assurance
RSD	Relative Standard Deviation
TPH	Total Petroleum Hydrocarbon
VOC	Volatile Organic Compound

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