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State of Alaska

DEPARTMENT OF ENVIRONMENTAL CONSERVATION

DIVISION OF SPILL PREVENTION AND RESPONSE

CONTAMINATED SITES PROGRAM

Draft Guidance on *MULTI INCREMENT* Soil Sampling

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I. Purpose and Applicability

Introduction

The purpose of this guidance is to summarize the requirements for effective design and implementation of *MULTI INCREMENT*[1] soil sampling undertaken as part of the remediation of contaminated sites in Alaska. The *MULTI INCREMENT* sampling (*MI*) process, as described in this guidance, may provide a more representative view of mean contaminant concentrations than traditional sampling approaches if applied correctly.

By regulation, the Alaska Department of Environmental Conservation (DEC) Contaminated Sites Program relies upon either of two methods to guide its decisions on the completion of remedial activities at sites contaminated with oil and hazardous substances (18 AAC 75.380(c)(1)) and (18 AAC 78.276(e)(1)). These methods are the maximum contaminant concentration detected in soil, or a statistically valid 95% Upper Confidence Limit (UCL) of the mean. An *MI* approach, if systematically planned and implemented, can accurately determine an average concentration representative of the soil contained within a defined area, i.e. the "decision unit." DEC will evaluate the *MI* sampling results, including the 95% UCL and calculated Relative Standard Deviation (RSD) of triplicate samples, for contaminated site status determinations. DEC has determined that an *MI* approach is acceptable when supported by the project-specific data quality objectives and if applied according to this guidance and an approved work plan. DEC has further determined that an *MI* approach, if applied according to this guidance, fulfills the intent of the regulations to protect human health and the environment.

Applicability

DEC will consider the use of *MI* for characterization or confirmation sampling purposes in order to meet data quality objectives that rely upon the mean soil concentration of an approved decision unit. Some examples of circumstances where *MI* may be appropriate include characterization from a surface release (i.e., aboveground storage tank), characterization or confirmation sampling of a stockpile or biocell, and excavation pit confirmation sampling.

DEC initially encouraged the use of *MI* at sites where soil is contaminated with petroleum hydrocarbons only. However, *MI* sampling may be applicable to contaminated sites with non-petroleum related contaminants. These may include PCBs, SVOCs, munitions' components, etc. DEC should be notified prior to initiating the systematic planning process if it appears there is an appropriate use of *MI* for non-petroleum contaminants. This guidance will be updated periodically to incorporate sampling for additional contaminants of concern and to address the possible use of *MI* in conducting risk assessments. *MI* is meant to supplement, not replace, existing department approved approaches or statistical approaches. This guidance is not a comprehensive procedures manual, nor does it substitute for multi-day *MI* training courses offered by private vendors.

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[1] *MULTI INCREMENT*® is a registered trademark of EnviroStat, Inc.

II. *MULTI INCREMENT* Sampling Theory

The objective of environmental sampling is to quantify some property of the media sampled, such as the amount of a contaminant present in soil at a given site. Traditionally, environmental cleanup programs across the nation have relied on discrete sampling to characterize environmental media. However, the number of discrete samples often collected at a contaminated site does not lend itself to statistically valid interpretation and cannot accurately quantify contaminant concentrations due to the heterogeneity of environmental media. In other words, it is impossible to identify the true mean of a population without the census of every data point. In the case of a 3,000 cubic yard soil stockpile, for example, the entire mass would have to be analyzed to determine the true mean concentration. Since it is impossible to sample and analyze the entire population due to practical considerations and cost limitations, statistical methods are used to determine a representative concentration.

A theory of particulate sampling was developed by geologist Pierre Gy to improve the quality of data gathered in support of mineral exploration and mining (Pitard, 1993). The *MI* approach described herein is based upon Gy's theories and is applicable to environmental sampling at contaminated sites.

Sources of Error

Heterogeneity is the norm when dealing with contaminated environmental media. A large portion of sampling error is a result of compositional and distributional heterogeneity. **Compositional heterogeneity** describes the variability of contaminant concentrations between the particles that make up the population. This type of heterogeneity results in fundamental error (FE). FE is a result of not representing proportional concentrations of all of the particles in the population. To minimize FE, it is imperative that enough mass be collected and analyzed to represent all particles in the exact proportion found in the population.

Distributional heterogeneity occurs when particles are not randomly distributed across the population due to slight spatial variations. Spatial variability will be missed if all samples are collected from one place. This type of heterogeneity results in grouping and segregation error (GSE). To minimize GSE, it is imperative to collect sample increments randomly and in enough locations to capture the spatial variability.

MI controls these two major types of sampling error in most situations. GSE is controlled by collecting multiple randomly located sample increments to address distributional heterogeneity. In general, a minimum of 30-50 random increments are required to address GSE; however, if greater distributional heterogeneity is expected more increments would be required.

FE is managed by collecting and analyzing sufficient sample mass to adequately address compositional heterogeneity. FE is directly related to the particle size of the population and the sample mass analyzed as illustrated by the following equation (Pitard, 1993).

$$FE = \sqrt{\frac{20(d^3)}{m}}$$

Where:
FE = Sampling fundamental error
20 = Sampling constant
d = maximum particle size (centimeters)
m = sample mass analyzed (grams)

Fundamental Error
The maximum fundamental error recommended by DEC for the purposes of this guidance is 15%.

The majority of organic contaminant mass in most situations is present in the 2mm fraction or less (medium sand to clay). Assuming a soil sample sieved to 2 millimeters (mm) and a minimum sample mass of 30 grams (g) is analyzed; the calculated FE will be under 15%. For atypical situations where the particle size is greater than 2 mm or the sample mass is less than 30 g, FE must be calculated using the above equation and reported to DEC. If FE exceeds 15% the data may be rejected.

Contaminant and Matrix Considerations with MI Sampling

Volatile samples ***must not*** be sieved (as discussed in the sampling procedures section).

Additionally, standard *MI* sampling procedures, as described in this guidance, may not be applicable to peat, tundra and other matrices not amenable to sieving. Alternate sample collection, processing, and sub-sampling methods would be required for such matrices. If *MI* is proposed at a contaminated site with these types of media, alternate *MI* techniques must be thoroughly detailed in a proposed plan submitted to the department for approval.

The default assumptions described above to address fundamental error (2 mm and 30 g) do not offer the same benefit for metals analyses for several reasons. 1) The physical sieving of the soil to a < 2 mm fraction may remove the contaminant of concern thus biasing the results. This would occur for example at landfill (dump) sites and firing ranges where some or all the metal(s) of interest are expected to be in a form larger than 2 mm particles (nuggets). 2) The sample mass normally digested and analyzed in the laboratory is relatively small (1 g). Control or reduction of fundamental error with this smaller sample mass is not feasible with sieving alone. For metals analyses, sample grinding (to decrease particle size) and/or increased digestion mass would be required. If grinding is proposed for metal *MI* samples, the sample preparation must be performed using a puck mill grinder. Considerations should also include possible metals being introduced into the sample from the grinder (e.g. chromium) and arsenic being released from the soil matrix via the grinding process. The alternate sample preparation, analysis, possible interferences, etc., must be detailed in a work plan submitted to the department for approval.

MI Contrasted to Composite Sampling

MULTI INCREMENT sampling ***is not*** the same as simple composite sampling. A *MULTI INCREMENT* sample is collected within a decision unit, whereas a composite may be collected without regard to a specific decision unit. Unlike *MI*, composite sampling does not adequately address sampling FE or GSE. A composite sample is a simple combination of discrete samples. A *MULTI INCREMENT* sample is a representative sample for a given decision unit. Although the physical process of collection is similar, the information derived from each process is different. As such, composite sampling cannot provide representative decision unit population data.

III. Decision Unit Identification

A decision unit is the defined area or volume in question, that is, the area or volume about which we need to make a decision. To be valid, *MI* sampling must be used in conjunction with an appropriate decision unit. Therefore, the identification and delineation of the decision unit is one of the most important factors when using *MI*.

The Dilution Effect

There is a critical item to keep in mind when identifying decision units and developing the *MI* work plan: *MI* may not be used to "dilute" contamination and therefore underestimate the need for cleanup. This may occur if the decision unit inappropriately incorporates large, uncontaminated areas in addition to real source areas.

Decision units will rarely be neat, geometric shapes, except perhaps in the case of a stockpile or treatment cell. It is unacceptable to simply draw a circle or a box around a source area and call it the decision unit for the purposes of site characterization without providing thorough documentation for the boundaries. If a source area is unknown or has been removed, the environmental professional must use all available means to delineate the decision unit, including historic photos, site information, interviews with knowledgeable parties, and field screening techniques. Three-dimensional decision units may be necessary when conducting a sub-surface site characterization because contaminants are not expected to be distributed evenly throughout the soil horizons.

Decision units are restricted to actual source zones and must not incorporate large, uncontaminated areas. Arbitrarily defined, large scale decision units are not allowed. Decision units may also be too small. For example, areas of high contaminant concentrations, or "hot spots," are essentially independent decision units, but knowing the mean concentration of a 5' x 5' petroleum surface stain is probably unnecessary when the hot spot can simply be excavated.

Two applications where *MI* can be applied in a relatively straightforward manner are treatment stockpiles or open excavations where contaminated soil or an underground storage tank has been removed. Stockpiles should be evaluated in terms of age and whether they have been actively mixed. For example, contaminant concentrations at the bottom of a static stockpile that has been in place for several years may be higher than near the surface. Decision units may need to be horizontal layers in this case.

Decision Unit Approval

Because of the importance of decision unit delineation, the decision unit must be approved by DEC prior to the sampling event in order to ensure DEC's evaluation of the results is not jeopardized. Decision units may not be changed without prior approval by the DEC project manager.

For an excavation, an *MI* sample would be collected for confirmation once field screening indicates all of the contaminated material has been removed. Sample

increments may be collected from the bottom and side walls of the excavation where contaminated soil has been removed. While circumstances will vary on a site-specific basis, typically the bottom of the excavation will be a distinct decision unit. Sidewalls may be combined into a single decision unit or treated independently.

For a source where the final excavation is significantly larger than the original footprint of an above ground or underground storage tank, it may be best to collect increments from beneath the original footprint rather than from over-excavated areas that are less likely to be impacted by potential spills or leaks from the former tank. If the excavation was hindered by the presence of buried utilities, buildings, or bodies of water, and contaminated soil is knowingly left in place, then the area that was left in place may become a new decision unit with the objective of characterizing the remaining contamination.

Many tank excavations also require evaluation of piping and dispensers. These areas should be considered as potential separate decision units during the planning process.

IV. Sampling Locations

One of the basic tenets of *MI* is to collect increments from multiple random locations. Random sampling works to eliminate error and addresses distributional heterogeneity by collecting samples from multiple, randomly selected locations (recall that mass is used to eliminate compositional heterogeneity). For additional information on sampling design, refer to *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan, QA/G-5S* (U.S. EPA 2002).

The random sampling approach must be proposed in the work plan, and the work plan must be submitted to DEC for approval prior to mobilizing to the field, as required under 18 AAC 75.335 (b). There are several types of random sampling techniques including simple random, stratified random, and systematic random. For the purposes of this guidance, a systematic random approach is recommended in order to establish a consistent protocol. As long as the sampler is not introducing bias into the sampling scheme, however, a different method may be proposed in the work plan if it appears more suitable to the site-specific situation.

In addition to surface sample increment locations, sample depth must also be taken into consideration. For instance, sample increments from a 24-inch deep stockpile should be taken at random depths throughout the stockpile so that samples are not collected directly from the surface. As stated earlier, for deeper or older stockpiles more than one decision unit may be required. For example, one decision unit might be the top two feet of a four-foot deep stockpile, and another decision unit might be two to four feet deep. The objective of dividing the stockpile into more than one decision unit is to characterize deeper soils separately because these soils may not experience the same level of volatilization and contaminant reduction as surface soils unless frequent tilling has occurred. For layered sampling, each increment location within the layer will need randomly generated, three-dimensional sampling coordinates.

Sometimes it may be more efficient to sample directly from the excavator bucket rather than wait for stockpile construction. Because increments need to be spaced equally across the entire decision unit (i.e., every 5th bucket), this works particularly well if the estimated volume determined through site characterization is expected to be relatively accurate. This becomes more difficult when soil is separated into several different stockpiles based on field screening results, or when the estimated volume is not well delineated. In these cases it may be difficult to determine the proper sampling frequency to ensure the entire decision unit is adequately represented.

V. Sampling Procedures

The primary objective of *MI* is to control the fundamental error (FE) and grouping and segregation error (GSE) associated with discrete sampling. Therefore, **strict adherence to correct field sampling procedures is essential**. The analyses that are applicable to the sampling procedures detailed herein include gasoline range organics (GRO); diesel range organics (DRO); residual range organics (RRO); benzene, toluene, ethylbenzene, and xylenes (BTEX); and polycyclic aromatic hydrocarbons (PAHs). Other volatile- and semi-volatile analyses may be required on a site-specific basis depending on the source of contamination.

MI sample collection, sieving, sample preparation, sub-sampling, etc., should be documented, where applicable, both photographically and in the text of the report submitted to the department for approval.

Volatile Analyses – GRO, BTEX, Volatile Organic Contaminants

Samples for volatile analyses must be collected before non-volatiles to reduce contaminant losses due to volatilization. To do this, the sampler should go to each of the sample increment locations and collect the much smaller increment for volatile analyses directly into the sample jar that contains the methanol. A second, unpreserved portion should be collected in the same manner for percent moisture (%moisture) determination for the volatile analysis. This would then be followed by the collection of the larger soil aliquot to be sieved for non-volatile analyses, if applicable.

The concern with *MI* is that the collection and sieving of the sample material will lead to volatilization of the contaminants, so **sieving *must not* be performed for any volatile analyses** (GRO, BTEX, or VOCs). To minimize volatilization, each sample increment must be deposited directly into a methanol-preserved sample container.

Due to the potential loss of volatiles during the *MI* sampling procedure, the department recommends that volatile samples be collected utilizing a coring type soil sampling device and extruded directly into a narrow mouth amber jar containing the appropriate volume of methanol preservative. Soil matrices not amenable to this type of sampling, e.g. compacted gravels, may be approved on a site specific basis to use an alternate volatile sampling technique utilizing “spoon” type sampling into wide mouth amber jars.

<u>Recommended Volatile Sampling Equipment</u>

- Disposable plastic syringe or similar “coring” type soil sampling device
- Volatile sample container
    - Pre-tared, narrow mouth, amber bottles with Teflon lined lids to prevent leakage. Bottle volume as appropriate, 250-500 milliliters recommended

<u>Alternate Volatile Sampling Equipment approved on a site specific basis</u>

- Small spoon, spatula, etc.

- Pre-tared 4-8 ounce (oz) amber jars with Teflon lined septum lids to prevent leakage

Volatile organics require that samples be field preserved with a minimum1:1 ratio of sample preservative to sample material (1 gram (g) soil to 1 ml methanol). This is a minimum required ratio, and additional soil mass is preferred as long as it is completely submerged by the methanol

The proper pre-tared containers and methanol volume must be provided by a CS approved laboratory. It is recommended that the laboratory provide the correct pre-tared bottle already containing methanol preservative to facilitate *MI* field sampling. The amount of sample to be collected, as well as the necessary volume of methanol, must be taken into account when choosing the container. Additionally, the container should be large enough to prevent methanol loss due to splashing, leaking, etc, during the sampling event.

In order to minimize the potential loss of volatiles, sample increments must be collected with minimal disruption and as quickly as possible to minimize exposure to ambient air. Begin by placing the appropriate amount of methanol into the sample container, if not pre-preserved by the laboratory (recommended). Next, go to each of the pre-determined, randomly selected sample increment locations and remove the soil to a depth of six inches or deeper by hand or using a coring device. If using narrow mouth amber bottles, a small, calibrated syringe or coring device is used to "plug" the soil. Depending on site-specific soil types, sampling into alternate, approved volatile containers may require the use of a small spoon or spatula. Collect approximately 2 -5 g and <u>immediately</u> place the soil sample directly into the methanol. Replace the lid onto the container. Collect a second 2-5 g portion into an unpreserved 4 oz sample jar. This unpreserved sample must be submitted to the laboratory for percent moisture determination for the volatile analysis. Proceed to the next increment location and repeat the collection process, extruding the soil increments into the same (1) methanol preserved bottle and (2) unpreserved jar.

When sampling from an excavator bucket, be sure to sample from the center and remove at least six inches of soil. For subsurface sampling, collect the soil directly from the hand auger or split spoon into the methanol. Use caution to ensure that the sample increment selected represents soil from the desired depth and not "sluff" material from an upper level.

Because samples for analyses of volatiles cannot be sieved, DEC recommends that total sampling error be minimized by submitting additional mass to the lab for analyses, such as 60 -150 g of soil. Additionally, to the extent possible, the individual increments should consist of the smaller particles (< 2 millimeter (mm)) to be similar to the non-volatile sieved sample matrix and to minimize FE. Large rocks or clumps of soil must not be collected as part of the sampling of volatiles, as this will increase the sampling error.

The volatile sampling procedure should be accomplished as quickly as possible to reduce the loss of soil contaminants and methanol due to volatilization. Care must also be taken to prevent the loss of methanol due to splashing during the addition of soil increments and/or spillage during the entire sampling procedure.

Ideally, samples for volatile analyses will be collected after the sampling tools have been field "calibrated" so that the sampler does not end up with fewer increments or soil mass than required. This can be done by weighing the soil to be sampled on a small balance to determine the approximate mass required from each random increment location. If the final sample mass does not meet minimum requirements, additional soil increments from randomly selected locations may be added, remembering to keep a minimum 1:1 methanol to soil ratio and that the soil must be completely submerged in the methanol. Additional methanol may be necessary and must be documented on the chain of custody appropriately.

Non-Volatile Analyses – DRO, RRO, SVOCs, PAHs, PCBs, etc.

The project laboratory must be contacted prior to mobilizing to the field to determine the sample mass normally extracted for the required non-volatile analyses. Alaska Methods AK102 and AK103 call for the extraction of from 10-30 g of sample material (soil). For *MI* purposes, the minimum required amount of material per analysis is 30 g. The DEC project manager must be assured that the laboratory is willing to meet *MI*-specific requirements prior to approving the work plan. Clear communication between the environmental professional, the lab and DEC prior to field mobilization is essential. A note in the comments section of the Chain of Custody form is also recommended. Remember, the more material that can be analyzed, the lower the fundamental error. As long as the lab is capable of handling samples of this size, a sample mass larger than 30 g is always preferred. The analyzed mass should be stated in the lab data report for verification.

> **Sample Mass**
> A sample mass larger than 30 grams is always preferred as long as the lab is capable of handling these samples. Clear communication between the environmental professional, the lab and DEC prior to field mobilization is essential

Equipment

- Large stainless steel spoon or scoop
- Large clean container (a large stainless steel bowl, Ziploc bags, or 5-gallon bucket)
- #10 (2mm) sieve
- Steel cookie sheet or other tray
- Small spatula or spoon
- Sample containers

For surface sampling, remove the soil to a depth of at least six inches prior to collecting the sample. When sampling from an excavator bucket, be sure to sample from the center

and remove at least six inches of soil. For subsurface sampling, collect the soil directly from the hand auger or split spoon. Use caution to ensure that the sample increment selected represents soil from the desired depth and not "sluff" material from an upper level.

Using the large spoon or scoop, collect the sample increment from the appropriate sample location and depth according to the pre-approved work plan. Scoop approximately 30-60 g (1-2 ounces) into the large, clean container and move on to the next sample increment location. Be cautious of oversize material, which means more mass may need to be taken from each increment to end with the 30 – 50 g sub-sample after sieving (a 5 kg field sample is not uncommon). Increments can be sieved directly into the bucket, or they can be bagged and sieved later.

Sub-Sampling for Non-Volatiles

Sub-sampling can be accomplished either in the field or in a laboratory set up to conduct sub-sampling according to the following procedure.

Laboratory Analysis
The laboratory must extract and analyze the entire contents of the submitted jar, minus the portion for the percent solids determination. The results may be less defensible if only a sub-sample or fraction of the jar contents is analyzed.

After the 30-50 sample increments have been collected into the bucket, use the #10 sieve (2mm) to sieve the soil into another clean container (another option is to sieve directly into the bucket at the time of collection). It is assumed that for organic contaminants the < 2mm fraction contains equal to or greater concentrations of the constituent of concern than the > 2mm fraction. If the >2mm fraction has or potentially has higher concentrations than the < 2mm fraction, sieving is not appropriate and alternate sample collection or preparation is required.[2]

> Note: The <u>entire</u> "bulk" *MI* sample must be sieved. Sieving only enough bulk sample to collect sufficient analytical amounts invalidates the *MI* process and, therefore, is not allowed.

Approximately 500 – 1000 g of material following sieving should be available. Once the > 2mm fraction has been removed, spread the remaining soil evenly on the steel tray approximately ½ inch in depth. Roughly divide the tray into 30-50 sections and using the small spatula, collect approximately 1 g (approx. ½ tablespoon) from each of the sections. Because fines tend to settle, scrape the spatula along the bottom of the tray to

[2] 18 AAC 75.990(117) identifies soil as "an unconsolidated geologic material, including clay, loam, loess, silt and gravel, tills, or a combination of these materials." The Petroleum Guidance on oversized material: page 41, states that for sites contaminated with gasoline or diesel type products, oversized material (greater than 2 inches in diameter) does not need to be treated or tested unless it has a potential to hold excessive amounts of contamination or contain visible petroleum product on the surface. Shale, schist, limestone, pumice or other porous types of rocks are examples of material that may hold excessive amounts of contamination. These factors should be discussed with the DEC project manager early in the planning process. Such material may need to be addressed through another sampling methodology.

make sure that every particle size is equally represented in the sample. Place all scoops into a single sample jar (2 or 4 oz as appropriate) to be submitted to the lab. As stated earlier, it will be beneficial in the beginning to use a balance to ensure the proper sample mass is submitted to the lab. The final sample mass per jar submitted to the laboratory, 30-50 g (30 g = approx.1 ounce), must meet the minimum amount of material to be analyzed by the lab. Repeat the process to collect a second sample into a separate jar and submit to the lab for percent moisture or as backup if re-analysis is required. A minimum 30 gram sample size is required for extraction and analysis, if additional material is available in the primary sample, then this material may be used for the percent moisture analysis.

Soil drying may be necessary to facilitate sieving of the <2mm fraction. Drying should only be performed if necessary. If drying is required, the entire bulk sample should be evenly spread on a tray approximately ½ to 1 inch in thickness. Dry at ambient room temperature only until the soil matrix is amenable to sieving. Drying at elevated temperature, i.e. "baking," is not allowed. Turning the soil on a daily basis may be necessary to facilitate drying. Sieve the entire dried sample to the <2mm fraction and sub-sample to collect analytical and percent moisture aliquots as described above. Drying may not be appropriate for some contaminants, e.g. pesticides or PAHs, as there is currently insufficient data to document whether or not the drying process results in the loss of analytes. Drying, if necessary, is acceptable for less temperature or photo-sensitive contaminants such as DRO, RRO, PCBs, etc. Loss of these types of contaminants due to temperature, light, biodegradation, etc. for normal drying times (1-3 days) is assumed to be minimal. Excessive drying times, e.g. 3-7 days, are not recommended and may impact analytical holding times and data quality. If this occurs, the data may be considered estimated and flagged appropriately.

VI. Quality Assurance and Control

Triplicate Sampling

Triplicate samples must be collected in order to verify that an *MI* sample truly represents the decision unit. The collection of triplicate samples allows for the calculation of relative standard deviation (RSD). This is markedly different from the typical duplicate sample that is collected from the same material as the primary sample. Results of all three samples must be included as part of the report submitted to the DEC. A minimum of one triplicate set is required for all *MI* sampling projects.

Triplicate samples must be collected from decision units with known or suspected reportable levels of contamination. Non-detect (ND) results may prohibit the RSD and 95% UCL calculations and the evaluation of the *MI* sampling representativeness. This may not always be practical for confirmation sampling or if source information is not available, however, should still be considered when selecting the triplicate decision unit. For example, for excavation confirmation sampling it may be more appropriate for the triplicate *MI* samples to be collected from the bottom of the excavation rather than a sidewall.

For sites with only one decision unit, triplicate sampling and analysis is required. For sites with multiple, similar, decision units, a minimum of one triplicate sample set must be collected for every 10 decision units or at a rate of 10%. Additional triplicate samples may be required based on site conditions and/or non-similarity of the decision unit(s). The final number of triplicate samples required will be determined by DEC during work plan development. The appropriate triplicate frequency must be documented and pre-approved in the *MI* work plan.

To collect samples in triplicate, the sampler may find it useful to mark the initial sample increment locations with flags or stakes. Triplicate samples should never be taken from co-located or adjacent locations. A practical way to achieve this is to move to the right (or left, forward, backward) a pre-determined distance and collect another sample increment for the second sample. Return to the initial sample increment location and move in a different direction and repeat the procedure. The distance between the original and triplicate samples must be adequate enough to evaluate variability. A minimum distance of one-half the *MI* quadrant size is recommended between primary, duplicate, and triplicate increment locations. Triplicate sampling locations that are co-located with or closely adjacent to the original *MI* sampling point are not acceptable. The exact method the sampler employs to collect the triplicate samples, the approximate locations and how these locations will be determined must all be specified in the work plan submitted for approval. The resulting sampling pattern essentially becomes systematic random so long as the sampler does not introduce any bias to any of the sample increment locations.

Triplicate sampling for excavator buckets will depend on the estimated number of buckets. For 30-50 buckets, three increments would be collected from each bucket; left

edge, center (original), and right edge. For excavations estimated to be greater than 50 buckets, triplicate samples must not be collected from the same bucket as the original increment. Rather, the two additional increments should be collected from unique buckets, again to assess variability. For example, if 90-100 buckets were estimated, the original increment would be collected from buckets 1, 4, 7, etc., the duplicate from buckets 2, 5, 8, etc., and the triplicate from buckets 3, 6, 9, etc. Again, triplicate collection must be documented in the work plan submitted for approval.

All *MULTI INCREMENT* sampling data must be reported and reviewed in accordance with Technical Memorandum 06-002, *Environmental Laboratory Data and Quality Assurance Requirements*, and the associated Laboratory Data Review Checklist.

Relative Standard Deviation and 95% UCL Calculations

Field triplicates are used to calculate the Relative Standard Deviation (RSD), a measure of data precision. The RSD is calculated as presented below:

$$\text{RSD (\%)} = \frac{100s}{\bar{\chi}}$$

where:
s = standard deviation
$\bar{\chi}$ = mean

Relative Standard Deviation
DEC requires a RSD of 30% or less. At RSDs greater than 35%, the data distribution starts to become non-normal and confidence in the representativeness of the *MI* sample results diminishes. To ensure an RSD of 30% or less it is imperative to control sampling error as described in this guidance.

The RSD is used as a quality control measure to assess the *MI* sampling procedure and the mean concentration of the decision unit. The RSD is an indicator of the data distribution. It is assumed that the data has a normal distribution with a RSD of 30% or less. Analytical results at or near the method reporting or detection limits may exhibit a greater variability and, therefore, an elevated RSD. These situations are evaluated on a site specific basis. Re-sampling may or may not be required. Contact the CS project manager for final evaluation and determination of any required actions.
Additionally, the standard deviation and the mean are used to calculate the 95% Upper Confidence Limit (UCL) of the contaminant. This is especially relevant for concentrations at or near the action or cleanup level. Site decisions will only be determined utilizing the 95% UCL as determined by the following equation:

$$\text{95\% UCL} = \bar{\chi} + \frac{ts}{\sqrt{n}}$$

where:
$\bar{\chi}$ = mean
t = 95% one-sided student t factor (e.g., for n = 3, t = 2.92)

s = standard deviation
n = number of samples

For *MI* triplicate data sets that include one or two non-detect (ND) results, the lowest value reported by the laboratory, either the MDL or PQL, should be substituted for the sample result to perform the RSD and 95% UCL calculations. One-half (1/2) the MDL substitution should not be performed. If all three *MI* results are ND, RSD and 95% UCL calculations are not required.

For example, the DRO Method 2 cleanup level is 250 mg/kg to achieve final site closure. Triplicate sample results are 227, 240, and 281 mg/kg respectively. The mean of this data set is 249 mg/kg, the standard deviation is 28, and the $ts/\sqrt{n}$ factor is 47. The resulting 95% UCL is 296 mg/kg. The cleanup level to achieve final site closure has not been met based on the 95% UCL.

For sites with multiple decision units, the 95% UCL must be calculated for each decision unit utilizing the above equation. In this situation, the $ts/\sqrt{n}$ factor calculated from the triplicate *MI* results shall be added to the *MI* result(s) for the remaining decision units. In the above example, if the *MI* result for a second decision unit at the site was 232 mg/kg, the 95% UCL for this decision unit would be 279 mg/kg (232 mg/kg + 47).

For sites where multiple triplicate *MI* samples are collected, the 95% UCL calculation for individual decision units must be discussed in the submitted work plan and approved by the department.

The standard deviation, mean, RSD, and 95% UCL for all decision unit(s) must be calculated by the environmental professional and submitted to DEC as part of the site characterization or cleanup report. For sites with multiple decisions units, the 95% UCL must be calculated and reported per decision unit, utilizing the approved work plan approach.

Only the 95% UCL will be utilized by the department for site management decisions. In cases where the 95% UCL for a given decision unit is above the applicable cleanup level, the entire decision unit is deemed contaminated. Options would include remediation of the entire decision unit or further characterization to delineate the contaminated zone. Additional characterization may be accomplished in one of two ways, through division into smaller decision units and re-*MI* sampling or through discrete sampling to <u>locate</u> and <u>delineate</u> the contaminated zone within the decision unit. Re-sampling using a few randomly selected discrete samples to possibly obtain an alternate result for the decision unit is not allowed.

VII. Summary

MI is a valid alternative to traditional discrete sampling for both characterization and site closure when conducted appropriately and supported by the data quality objectives for the project.

The following steps summarize a valid *MI* sampling approach:

1. Define the decision unit(s) with DEC input and approval.
2. Identify the random sample locations and depths within each decision unit.
3. Submit the work plan for DEC approval.
4. Collect 30-50 increments per decision unit.
5. Collect triplicate samples at independent locations.
6. For volatiles, field preserve sample directly in methanol; do not sieve.
7. For non-volatiles, sieve to 2 mm, sub-sample appropriately, and submit 30-50 g to the laboratory.
8. Conduct data package Quality Assurance review when laboratory results are received.
9. Calculate and report all relevant quality control parameters.
10. Submit report for DEC review.

References

Pitard, Francis F. *Pierre Gy's Sampling Theory and Sampling Practice*. 2nd edition, CRC Press. 1993.

Ramsey, Charles. EnviroStat: *Sampling for Defensible Environmental Decisions*. April 25-28, 2006.