CLU-IN Contaminated Site Clean-Up Information

Welcome to the CLU-IN Internet Seminar

Incremental-Composite Sampling Designs for Surface Soil Analyses, Module 1 of 4

Delivered: February 16, 2012, 2:00 PM - 4:00 PM, EST (19:00-21:00 GMT)

Presenters:

Deana Crumbling, EPA Superfund (crumbling.deana@epa.gov)

Robert Johnson, Argonne National Laboratory (rljohnson@anl.gov)

Moderator:

Jean Balent, U.S. EPA, Technology Innovation and Field Services Division (balent.jean@epa.gov)

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Although I'm sure that some of you have these rules memorized from previous CLU-IN events, let's run through them quickly for our new participants.

Please mute your phone lines during the seminar to minimize disruption and background noise. If you do not have a mute button, press *6 to mute #6 to unmute your lines at anytime. Also, please do NOT put this call on hold as this may bring delightful, but unwanted background music over the lines and interupt the seminar.

You should note that throughout the seminar, we will ask for your feedback. You do not need to wait for Q&A breaks to ask questions or provide comments. To submit comments/questions and report technical problems, please use the ? Icon at the top of your screen. You can move forward/backward in the slides by using the single arrow buttons (left moves back 1 slide, right moves advances 1 slide). The double arrowed buttons will take you to 1st and last slides respectively. You may also advance to any slide using the numbered links that appear on the left side of your screen. The button with a house icon will take you back to main seminar page which displays our agenda, speaker information, links to the slides and additional resources. Lastly, the button with a computer disc can be used to download and save today's presentation materials.

With that, please move to slide 3.

Module 1.1

Incremental-Composite Sampling Designs for Surface Soil Analyses

Introduction to 1st Day

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Overall Webinar Agenda

- 4-part series of 2-hr Clu-In webinars (Feb 20, 21, 24 & 27)
- Soil sampling fundamentals (Day 1)
- Terminology and existing sources of information (Day 1)
- Incremental and composite sampling designs (Day 2)
  - Incremental: Estimating average concentrations for a decision unit
  - Composites: Looking for contamination "hot spots"
  - More representative discrete samples
- Calculations and using Visual Sample Plan (VSP) (Day 3)
- Several case studies (Days 3 & 4)
- Interspersed periods for Q & A

Today's Agenda

- Webinar purpose and goals
- Instructor introduction
- Soil sampling fundamentals
  – 10-min Q & A
- Terminology and existing guidance/information
  – 10-min Q & A

Webinar Purpose and Goal

- Multi-increment / Incremental / Composite soil sampling designs are generating interest
- EPA guidance clear that such designs are useful; but more implementation detail needed
- Other information sources are now filling that gap
- When done correctly these soil sampling strategies provide significant benefits to soil characterization programs
- Goal is foster understanding incremental and composite sampling strategies, and how to apply them

Instructors for the Webinar Series

- **Deana Crumbling,** crumbling.deana@epa.gov
  Office of Superfund Remediation &Technology Innovation
  U.S. Environmental Protection Agency
  Washington, D.C.
  (703) 603-0643

- **Robert Johnson,** rlj@anl.gov
  Environmental Science Division
  Argonne National Laboratory
  Argonne, Illinois
  (630) 252-7004

Does anyone feel that having a little fun during the day is too juvenile, like working for reward by answering questions in quizzes?

We try to liven up a dry, dry…dry, dry topic and a long course to keep you interested and awake.

Speak now or forever hold your peace.

Instructors (cont'd)

- **Michael Stroh,** Micheal.Stroh@dnr.mo.gov
  Hazardous Waste Program
  Missouri Department of Natural Resources
  (573) 522-9902

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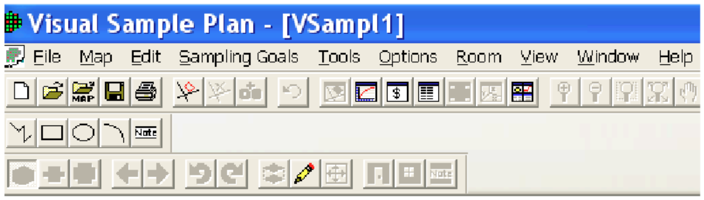
Software Resources and Disclaimer

- Disclaimer: “References to product or service providers are for information purposes only and do not constitute endorsement”
- Several documents and 2 software programs referenced
- For more information on the software programs:
  - Visual Sample Plan (VSP) (http://vsp.pnl.gov/)
  - ProUCL (http://www.epa.gov/esd/tsc/software.htm)

A Free (DOE & EPA Funded) Statistical Calculator & Visualization Tool to Aid Planning

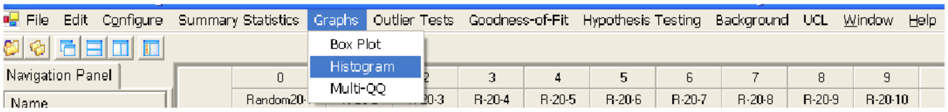


Welcome to Visual Sample Plan

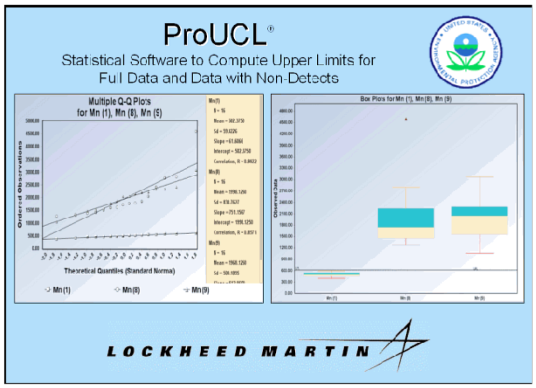
Visual Sample Plan

http://vsp.pnnl.gov/

A Free (EPA Funded) Data Exploration & Analysis Tool



http://www.epa.gov/esd/tsc/software.htm



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Incremental vs. Composite Sampling
Basic Differences

- Goal of incremental sampling is to find an average concentration over some defined area (a DU).
  - Uses 3 or more independent replicates to obtain a UCL
- Goal of composite sampling is to gain information about the spatial distribution of contaminants.
  - Accurate estimate of the concentration mean within the given area not required
  - Typically not use replicates over the composite area
  - Suppresses short-scale heterogeneity for a "single" sample & improves representativeness
- Composite uses fewer increments per sample than incremental sampling

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Key Take Away Points…

- Incremental and composite sampling designs provide significant advantages for soil characterization
- There are reasons for the different labels
- Strategies used need to match project goals (i.e., DQOs)
- Systematic planning and conceptual site models (CSMs) are critical to “getting it right”
- CAN find “hot spots” via an incremental-composite sampling strategy, BUT “hot spot” must be <u>defined</u>!
- Sample processing and correct subsampling prior to analysis is vital to data quality

Module 1.2

Incremental-Composite Sampling Designs for Surface Soil Analyses

Fundamental Concepts

Soil Sampling Fundamentals

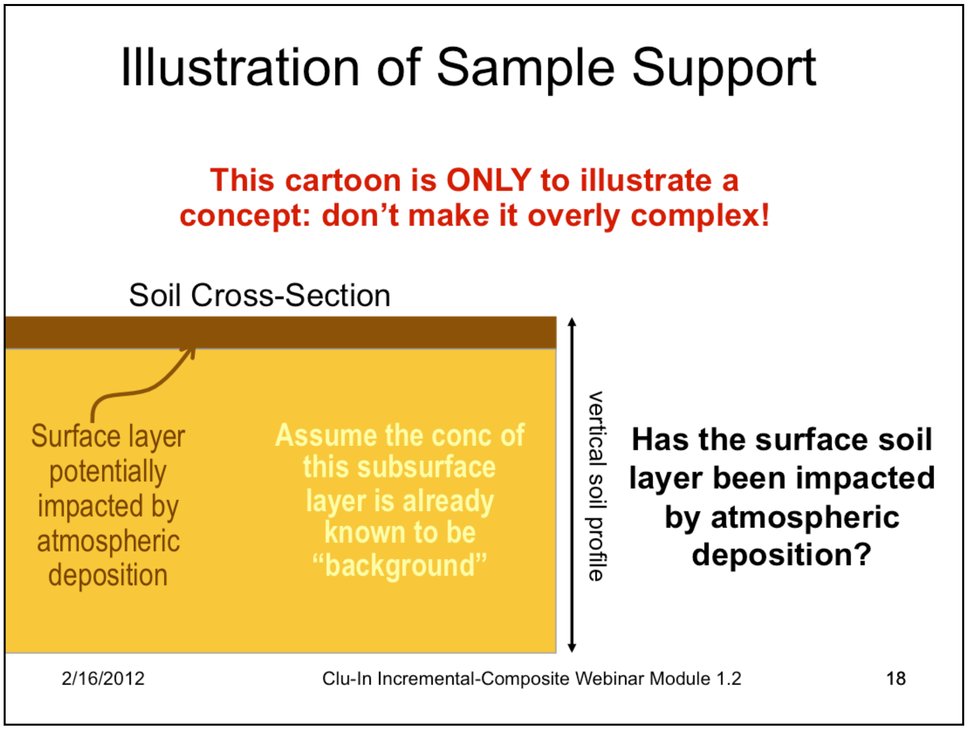
- Soil sampling goals
  - What's the question?
- The challenges of sampling soil
- Basic statistical concepts

Typical Soil Characterization Goals

- Looking for the presence of contamination above some threshold
  - Comparison to background
  - Comparison to not-to-exceed cleanup criteria
  - Looking for hot spots
- Calculating the average concentrations over an area of interest
  - Determining exposure point concentrations (EPC) for risk assessment (using UCL on the mean)
  - Comparison to area-averaged cleanup requirements

Basic Concepts

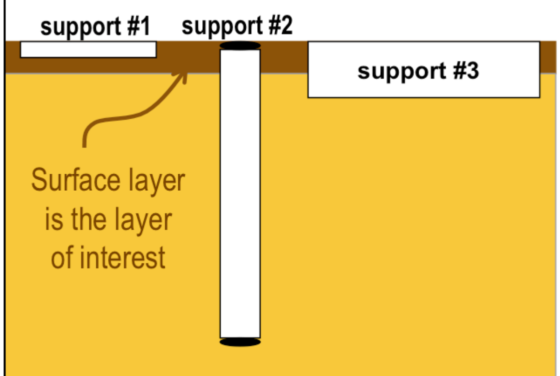
- **Sample:** A portion of a population collected to characterize a population parameter of interest
  - Beware: Physical "sample" (1 jar or bag) vs. a statistical "sample" (a set of physical samples)
- **Sample Support:** The physical dimensions and characteristics of a physical sample
  - Critical concept for dealing w/ soil contamination
  - Sample support determines concentration result!!



Emphasize that this is cartoon whose only purpose is to illustrate the sample support concept.

Which of these sample supports is <u>most</u> representative of the soils of interest?

All soil within the sample support outline (white area) is considered to be a single sample. The sample is *thoroughly* homogenized before analysis.



<u>Answer:</u> Support #1.

Why?: This sample contains only soil from the layer of interest.

BUT…

How could sample support #1 be modified to *better* represent the layer of interest?

Emphasize that this is cartoon whose only purpose is to illustrate the sample support concept.

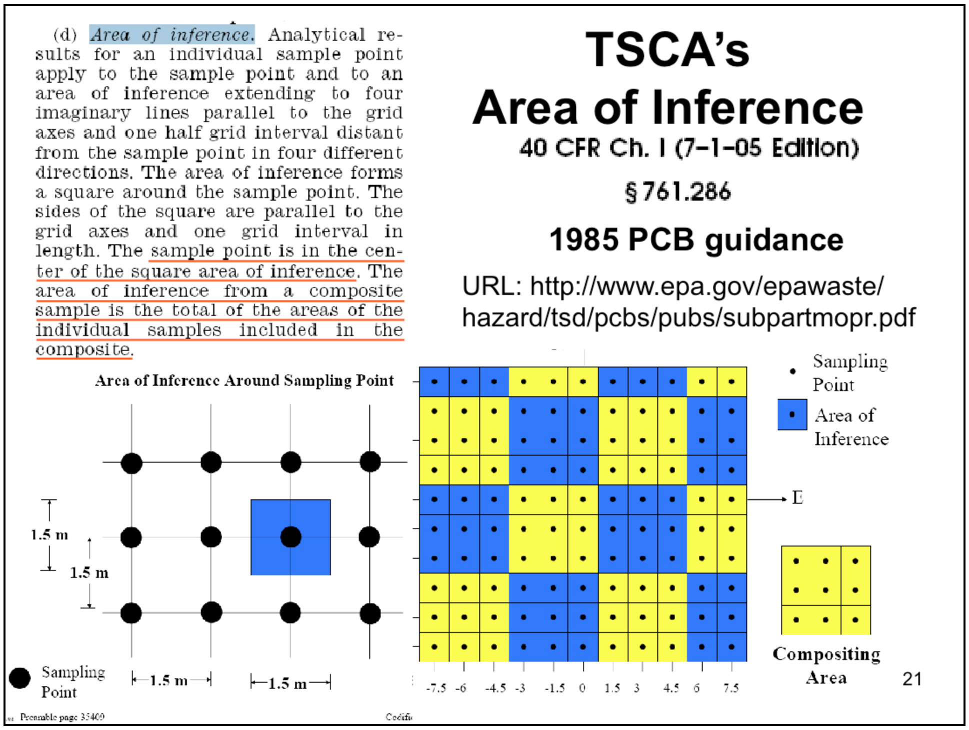
#1 would represent the layer of interest better if the depth of the sample was the same as the depth of the layer of interst.

Representativeness

The degree to which sample reflects the original population in one or more key characteristics of interest as defined by the context of the decision

RCRA (40 CFR 260.10): "Representative sample means a sample of a universe or whole (e.g., waste pile, lagoon, ground water) which can be expected to **exhibit the average properties of the universe or the whole**."

Having a "representative sample" implies the decision maker can confidently extrapolate concentration results from the tiny analytical subsample mass back to the much larger soil mass from whence it came

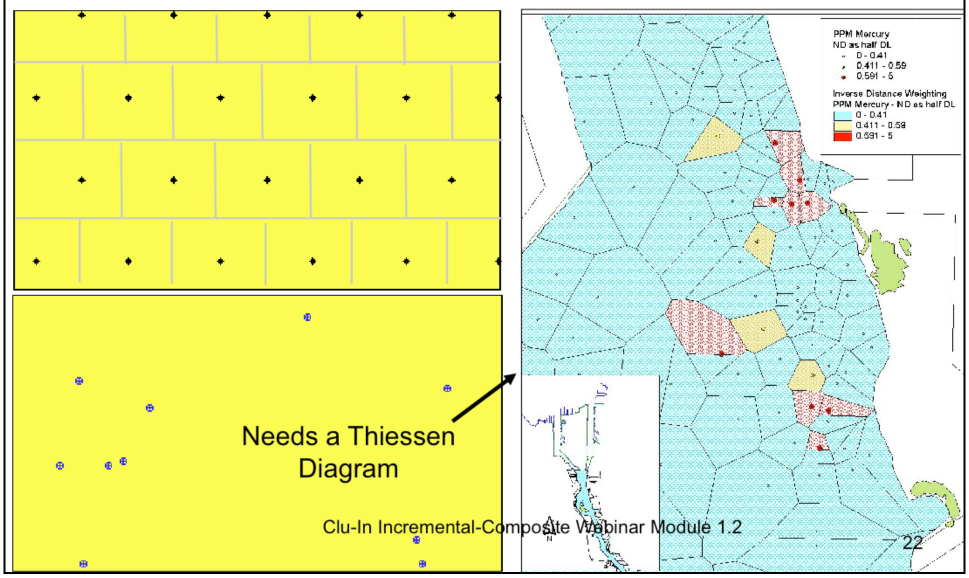


8/5/98 Preamble page 35409; Codified 63FR 35465 & 35466

1.5 m x 1.5 meters = 4.5 ft x 4.5 ft = 20.25 sq ft

1985 PCB guidance on EPA.gov http://www.epa.gov/epawaste/hazard/tsd/pcbs/pubs/subpartmopr.pdf

The “area of inference” is easy to determine for gridded designs; much harder for random designs



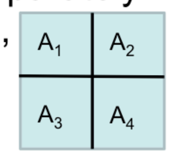
Animated slide for presentation

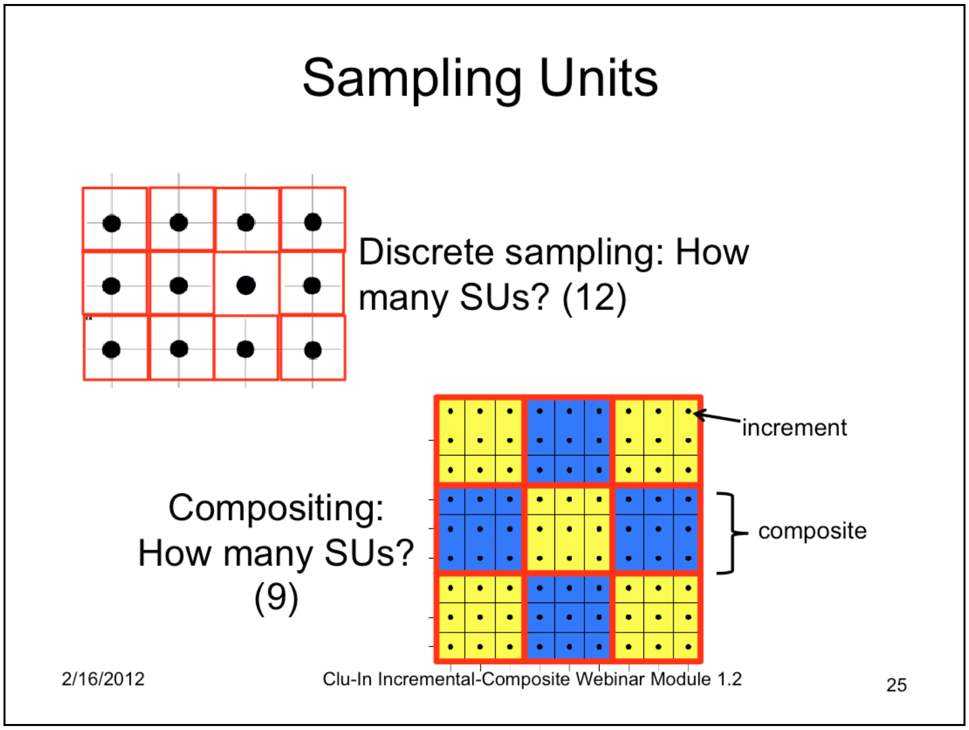
Sampling Unit (SU)

- Sampling Unit = the volume of soil represented by a single sample and data result; the "area of inference" for a sample
- If 10 individual samples for analysis are taken from Area A, then there are 10 SUs in Area A
  - If 1 of those 10 samples gets a collocated QC sample, and another gets a field duplicate QC sample, there are still 10 SUs
- If a single sample for analysis is taken for Area A, then there is 1 SU for Area A (Area A = the SU)

Sampling Unit (SU) (cont'd)

- If 30 increments from Area A are combined into a single composite sample for analysis, Area A has 1 SU (1 data result generated from entire Area A)
  - If 3 replicate 30 increment composites are taken from Area A, there is still only 1 SU
- If Area A is divided into 4 adjacent, non-overlapping areas which are sampled separately (by a discrete sample or by a composite), there are 4 SUs in Area A





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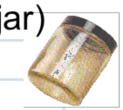
1985 PCB guidance on EPA.gov http://www.epa.gov/epawaste/hazard/tsd/pcbs/pubs/subpartmopr.pdf

Spatial Scales of Data Variability

Data Variability: variations in the measured magnitude of a population characteristic

Micro-scale, within sample jar: measured by lab duplicates (2 analytical subsamples taken from same sample jar)



Lab dup examples from actual site data. Population characteristic being measured = Pb conc

Pb: ICP lab duplicates		
	original	dup
	1038	688
	332	412
	874	2187
	34	49
	248	223
	69	45

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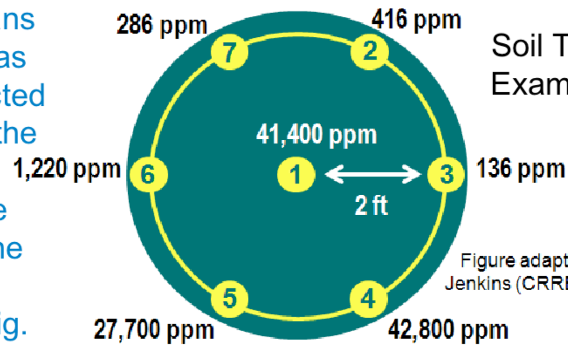
Next Higher Spatial Scale

"Co-located" samples

Collocated samples are located close to each other (inches to a few feet) and considered <u>equivalent</u>

"Equivalent" means that 1 sample is as likely to be collected as another from the "same" sampling location. Chance governs where the field technician bends down to dig.



Soil TNT Example

Figure adapted from Jenkins (CRREL), 1996

Short-Scale Heterogeneity

Arsenic in Samples from 3 Residential Yards
(Note: micro-scale effects were controlled)

From actual site

Yard #1

1 ft apart over 4 ft

As 129 221 61 39 14

Yard #2

Transect points 2 ft apart

As 37 290 625 94

Same yard, 8 ft away from group to left; 2 ft apart

As 27 29 45 34

Yard #3

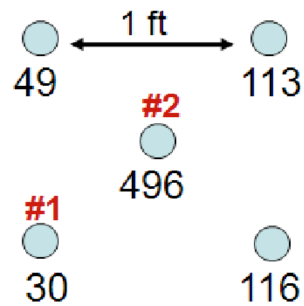
Evenly spread over 7 ft

As 17 41 367 351 268

Same yard, 15 ft away from group to left & spread over 4 ft

As 29 24 79 120

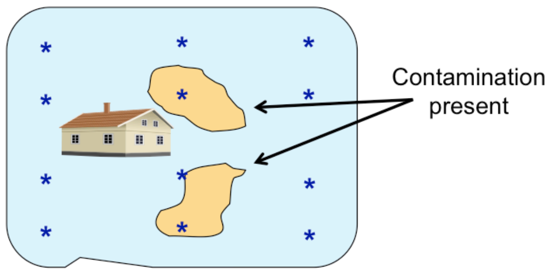
Short-scale Data Variability for U



Uranium investigation collocated sample group.
"Collocated" means that any one of these sampling locations could be selected as a sampling point for the "same" discrete sample.
Simple chance can lead to very different conclusions!

Variability at Highest Spatial Scale

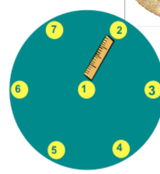
Large-scale variability: differences in concentration at the scale of typical sampling design spacing; the kind of conc. variability traditional sampling designs are trying to find



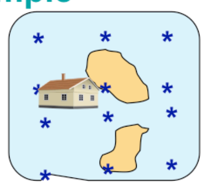
Heterogeneity Causes Data Variability

- **Heterogeneity**: Variations in matrix properties
  - **Within-sample** micro-scale heterogeneity

  - **Short-scale, between-sample** heterogeneity (affects agreement between collocated samples)

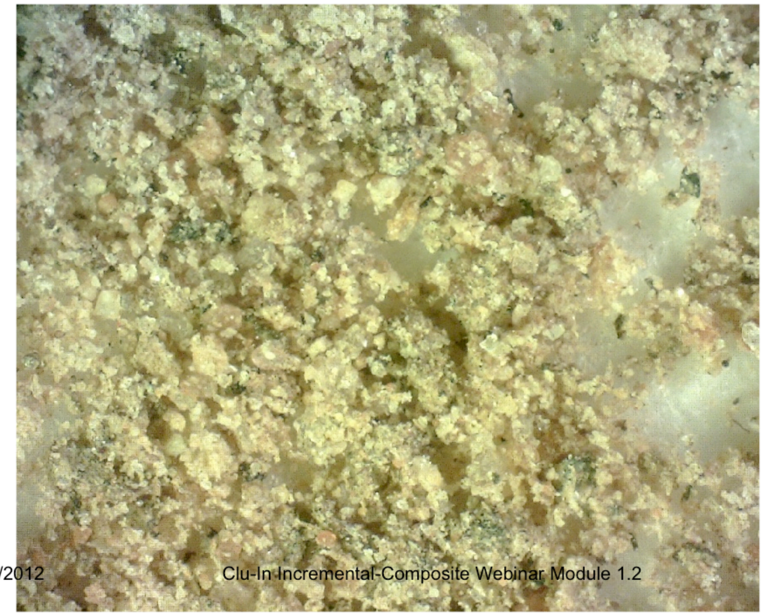


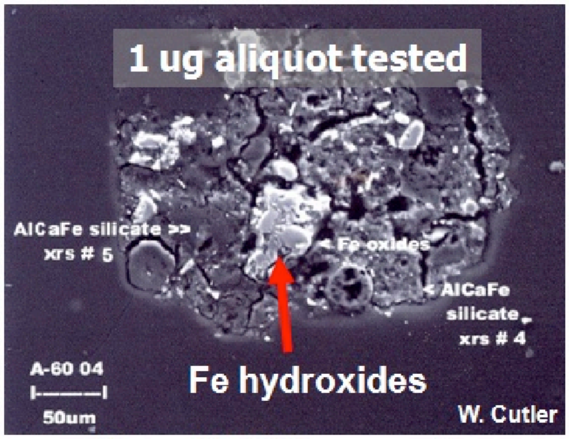
  - **Large-scale, between-sample** heterogeneity (on scale of conventional distances between samples)



The Properties of Soil Matrices Create Heterogeneity

Soil with low organic content, showing mineral grains with sizes spanning orders of magnitude (10X mag.)





The Nugget Affect

The iron in a cubic yard of soil can adsorb 0.5 to 5 pounds of metals & organics (Vance, 1994)

As = 5,000++ mg/kg but arsenic <u>mass</u> is only 5 ng

(SEM analysis of Fe hydroxides)

(SEM = scanning electron microscopy)

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“Given the average concentration in soil, the iron in a cubic yard of soil is capable of adsorbing from 0.5 to 5 pounds of soluble metals as cations, anionic complexes, or a similar amount of organic[s].” (Vance, 1994). [Reference = David B. Vance. National Environmental Journal. May/June. 1994 Vol.4 No. 3 page 24-25.

Mass of As in 1 ug soil with 5000 ppm conc (0.5%)= 5 ng

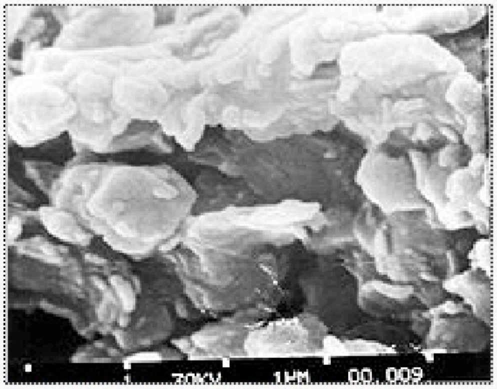
If that 5 ng As dropped into 1 g soil = 5 ppb

If that 5 ng As dropped into 1 kg soil = 5 ppt

5 ng dropped into 0.5g soil (ICP) = 10 ppb

5 ng dropped into 2 g soil (ICP) = 2.5 ppb

Clays Have a Layered Structure and a Very Small Particle Size

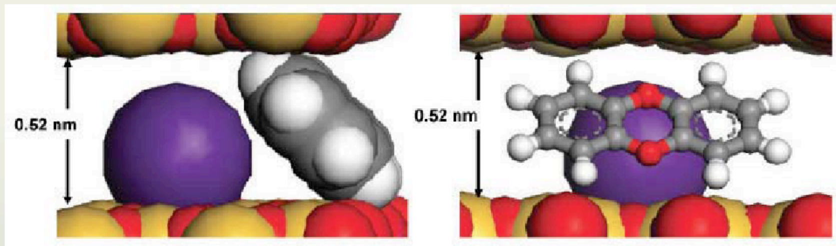


electron microscope photograph of smectite clay - magnification 23,500

Small size & layers make for very large surface area to sorb contaminants. Also, layers are lined with negative charges, so are especially attractive to positively charged metals. Creates nugget effect.

Clay particles showing plates. Uncopywrited photograph from http://en.wikipedia.org/wiki/Clay

Organic Contaminants Can Get Stuck between Clay Plates



Proposed molecular model of DD-smectite complex showing the arrangement of DD in the interlayer space of the smectite clay.

Clay plates with embedded positively charged metallic ion (e.g., Ca++). Plates increase surface area and attractive forces available to bind contaminant molecules (DD = dibenzo-p-dioxin) Source: SRP Research Brief 183, 3/3/10, page 2

Cartoon of smectite plates with an embedded positively charged metallic ion (such as calcium, purple ball). The plate structure increases the surface area and attractive forces available to bind a contaminant molecule (DD = dibenzo-p-dioxin; red balls represent oxygen atoms) (Source: SRP Research Brief 183, 3/3/10, page 2).

Organic Matter Also Binds Contaminants

Soil with high organic content. Mineral particles are embedded in the organic soil structure created by microbial activity breaking down vegetable matter. (40X magnification)

Composition & Particle Size Create Within-Sample (Micro-Scale) Heterogeneity

Firing Range Soil Grain Size (Std Sieve Mesh Size)	Pb Concentration in fraction by AA (mg/kg)
Greater than 3/8" (0.375")	**10**
Between 3/8" and 4-mesh	**50**
Between 4- and 10-mesh	**108**
Between 10- and 50-mesh	**165**
Between 50- and 200-mesh	**836**
Less than 200-mesh	**1,970**
Bulk Total	**927** (wt-averaged)

Adapted from ITRC (2003)

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Data adapted from the Interstate Technology and Regulatory Counsel (ITRC). 2003. *Characterization and Remediation of Soils at Closed Small Arms Firing Ranges.* January. Available on-line at http://www.itrcweb.org/SMART-1.pdf

The results of this study show how different particle sizes within the same jar of soil have different lead (Pb) concentrations. We call this "within-sample" or "micro-scale" soil heterogeneity because different concentrations of analyte occur on very small spatial levels within in a single jar of soil. Although the soil may look "homogenized," it really isn't as long as different particles sizes exist in the sample jar. This would not matter IF the entire volume of soil in the jar was analyzed all at once. Analyzing the whole sample gives you the true concentration of the jar contents. However, jars usually contain 100 grams or more of soil. Common analytical methods for Pb (and other metals) use between 0.5 and 2 grams of soil for the analysis, depending on the lab's SOP. So the analytical sample is much, much smaller than the mass of soil in the jar.

For this study, a large soil sample was taken from a firing range with Pb contamination. The soil sample was dried and clods were broken apart, but no grinding was performed. Visible fragments of Pb bullets were removed. The soil was then sieved into different-size fractions. The 6 particle size fractions that resulted are provided above. Particle size gets smaller as the mesh size increases. Each particle-size fraction was analyzed for Pb separately by atomic absorption (AA), a routine laboratory method for analyzing metals.

An obvious trend exists for this site's soil: the Pb concentration in a particle size fraction increases as the particle size decreases. Why should this be? There are a few reasons. The smaller the particle size, the more surface area is available to adsorb contaminants like Pb. And the smallest fraction is more likely to have particles made of clay minerals. Clay minerals carry a negative charge that attracts and holds on to positively-charged metal ions. Over time, contaminants "partition" into the soil constituents that have properties that attract them. There may also be very, very tiny particles of Pb released by the gun's firing mechanism, from impacts of bullets into hard surfaces (like rocks), and by slow decay of bullet fragments.

Particle size effects on analytical results have ramifications for the sampling & analysis of soil. When soil is shipped to a lab, motions in transit cause a segregation of particle sizes within jars. When a sample jar arrives at the lab, larger particles are typically sitting on top, and smaller particles have moved toward the bottom. If a technician were to sample a jar by unscrewing the cap and simply scooping a subsample off the top, the Pb result would likely be a lot lower than the true Pb concentration for the whole jar of soil.

As mentioned above, metals analysis for soil typically involves digesting a very small mass, around 1 gram. So another variable that can affect the concentration of the analytical sample (and thus the reported result) is the size and shape of the utensil used to weigh out the nominal 1-gram. A variety of utensils of varying sizes and configurations can be used to scoop up small amounts of soil and ferry it from the jar to the weigh boat that sits on the balance. There is no standardization of what utensil should be used. Even within the same lab, different technicians may use different scoops. A larger, spoon-shaped utensil will retain the larger particles (which provide mass, but little Pb), but those particles could easily roll off a flat spatula or a much smaller scoop. Thus a larger bowl-shaped utensil will select FOR larger particles, whereas a flat or very small scoop surface will select AGAINST larger particle sizes.

Another variable is related to the motions the technician makes while weighing out the analytical sample. Say the target mass for an analytical sample was 1 gram. Weighing out samples takes time, and technicians are always under pressure to maintain high sample throughput. So the fewer scoops into the weigh boat needed to get close to 1 gram, the more samples a technician can process. So naturally, the technician will make the 1st scoop out of the jar larger to try to get close to 1 gram without going significantly over. If it does go overweight, the soil must be dumped and weighing started over. Although the analytical sample doesn't need to be exactly 1 gram, it should be close. If a larger sized scoop was used and the amount of sample in the scoop looks larger than 1 gram. the technician may give the scoop a little shake to dump some of the larger particles back into the jar. This action selects AGAINST larger particles.

Now, say the 1st scoop of soil brought the balance to 0.7 g. Then a smaller volume (with even fewer large particles that might "tip the weight over") may be scooped into the weigh boat. Say that now the balance says 0.9 g. To get the mass closer to 1.0, the technician will likely gently tap the side of the scoop while holding it over the weight boat in order to knock smaller particles in a little at a time. This action selects AGAINST larger, low-Pb content particles and preferentially adds smaller, high Pb-content particles.

These very common techniques are fine when weighing out materials that are truly homogenous and have a uniform particle size. But for soils, variable selection for and against various particle sizes in the analyzed subsample changes the result. These various weighing techniques may all occur in the same sample weighing, or only one or none may occur. The fact that these variables are not controlled in routine laboratory practice is part of the reason why split sample results can be very different, and explains why lab duplicates from the same jar often have poor precision.

Size conversions:

3/8" = 0.375 in. = 9.525 mm

ASTM (US std) nominal aperture mesh size (mm):

4-mesh = 4.76 mm

10-mesh = 2 mm

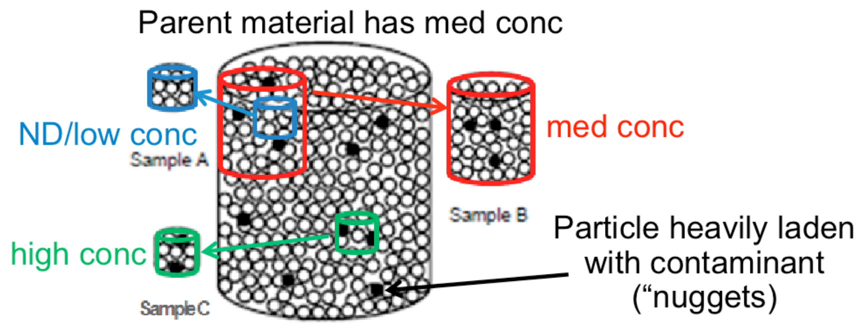
50-mesh = 0.297 = 0.3 mm

200-mesh = 0.074 mm

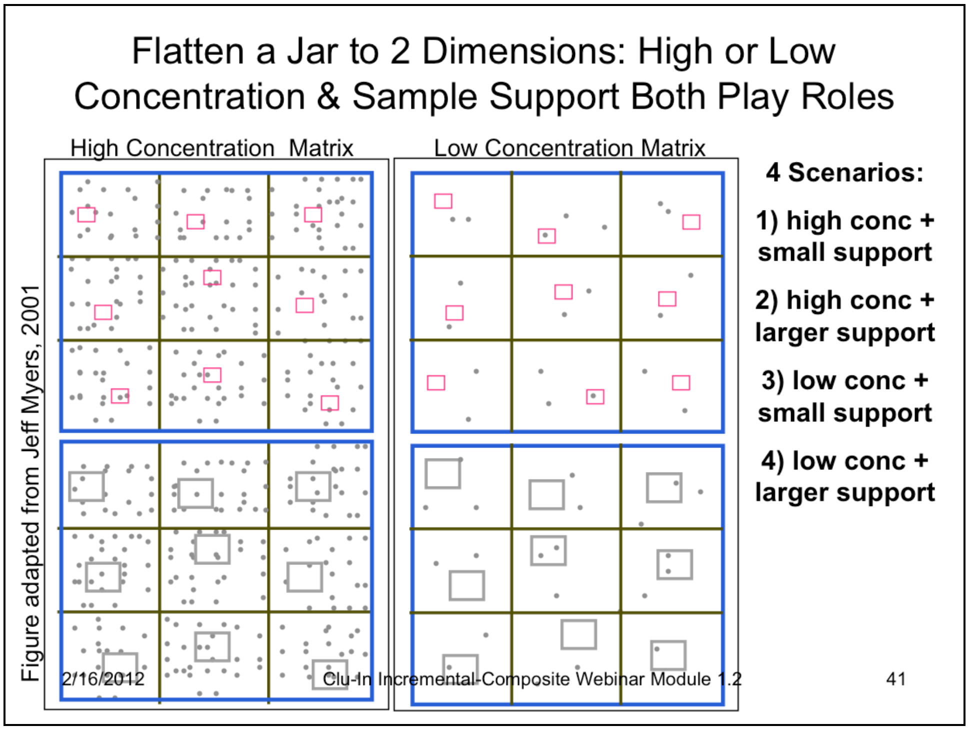
Representativeness & Particles

- A representative subsample of a particulate solid is one that has the same ratio of particle sizes as the targeted parent material
- "Targeted" means that the particle size(s) of interest to the decision are defined during up-front planning and are isolated before analytical subsampling
  - E.g., risk assessment could be interested in dust sizes
  - Disposal might be interested in the bulk material

Representative Sampling for Particulates



The difficulties of taking representative samples from particulate parent material (the large compartment in the center). Smaller samples are less likely to accurately represent the parent material than are larger samples.



The nugget effect can occur when contamination occurs in particulate form (such as explosives residues deposited as a powder or lead fragments in a firing range), or when contaminants partition onto mineral surfaces or organic carbon which are themselves heterogeneously distributed. Gy theory relates the size of the matrix particles to the mass of a sample support that can be representative of the true mean for the larger matrix volume.

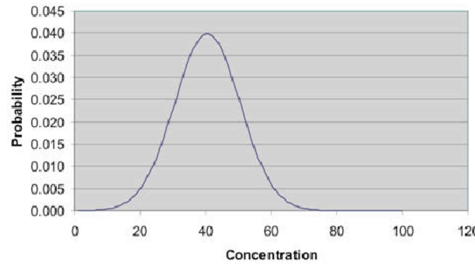
Specifying a regulatory threshold without specifying the sample support over which it applies (or at least recognizing that differences in sample support introduce variability into analytical data results) easily leads to widely different analytical results. Since the sample support is generally ignored in regulation, it is ignored in practice and the sample support is left to chance. This leads to uncontrolled (and usually undocumented) variations in sampling conditions and often widely varying results that are difficult to interpret. Unless the lab was in charge of field sampling and was involved in project planning and SAP/QAPP preparation, the lab cannot be held accountable for such variable results. The analytical result is probably correct; project planning was faulty for not ensuring that sample collection procedures would produce samples representative of the decision.

The volume of the sample is an important factor that influences the reported concentration for the sample, especially when contaminants are heterogeneously distributed throughout the parent matrix. The nature of the release, such as contaminant release in the form of a powder, increases the heterogeneity. Alternatively, contaminants will be heterogeneously distributed if they preferentially partition onto mineral surfaces or into organic carbon that are themselves typically heterogeneously distributed.

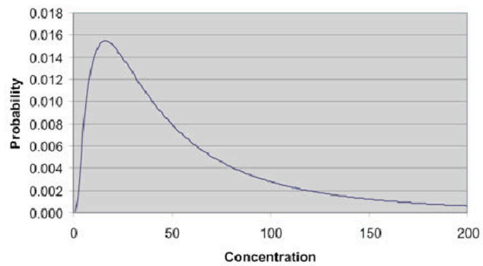
3 different color-coded sample volumes are illustrated here. From largest to smallest: green/brown, light blue, and red. The dark particles are “contaminated” particles in a matrix of “cleaner” particles. Variable capture of the “dirty” particles is illustrated for higher and lower contaminant concentrations and different sample volumes.

A Good Time to Introduce Common Statistical Data Distributions

- Normal (Gaussian) distribution:
  - Can take on any (+ or -) value
  - Symmetric

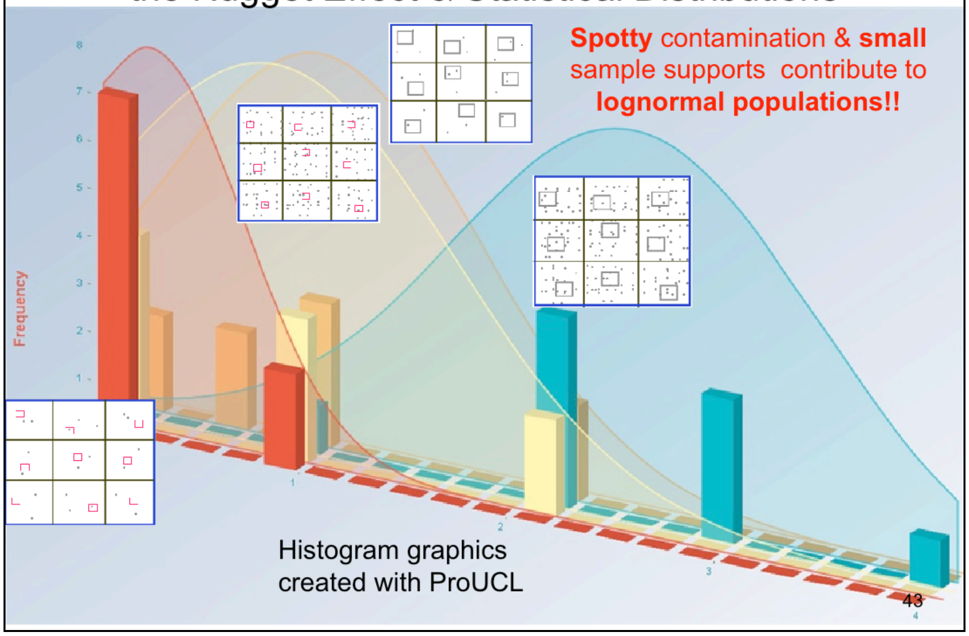


- Lognormal distribution:
  - No negative values
  - Skewed to right

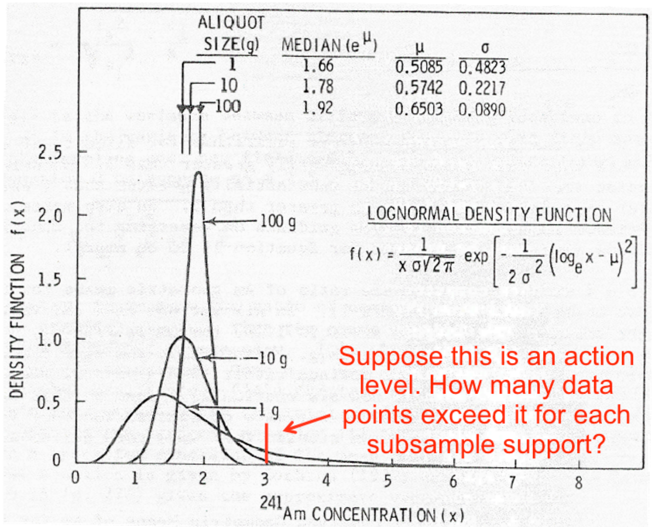


42

Relationship Among Sample Support, Concentration, the Nugget Effect & Statistical Distributions



Maximum Result Depends on Sample Support



Each analytical subsample support (1, 10, or 100 g) has its own distribution of data results.

The larger the subsample support, the closer to normal the distribution of data results becomes.

The more likely it is that any single result will be close to the true mean.

Graphic of actual data from a DOE americium-241 study (Gilbert & Doctor, 1985)

Gilbert, Richard O. and Pamela G. Doctor. 1985. Determining the Number and Size of Soil Aliquots for Assessing Particulate Contaminant Concentrations. Journal of Environmental Quality Vol 14, No 2, pp. 286-292.

How is This Relevant for Compositing?

Composites/incremental samples produce better estimates of the mean because the large sample support normalizes statistical data distributions.

Comparison: Discrete samples collected in 1-m x 1-m cells within a 10-m X 10-m grid. 30-increment samples collected randomly throughout entire 10-m X 10-m grid.

2,4-DNT Results:	Discrete	Multi-increment
Number of Samples:	100	10
Minimum:	0.0007 mg/kg	0.60 mg/kg
Maximum	6.4 mg/kg	1.35 mg/kg
Mean:	1.1 mg/kg	0.94 mg/kg
Standard Deviation:	1.2 mg/kg	0.24 mg/kg
Median:	0.65 mg/kg	0.92 mg/kg
Distribution:	Skewed	Gaussian

Adapted from Jenkins (USACE) presentation

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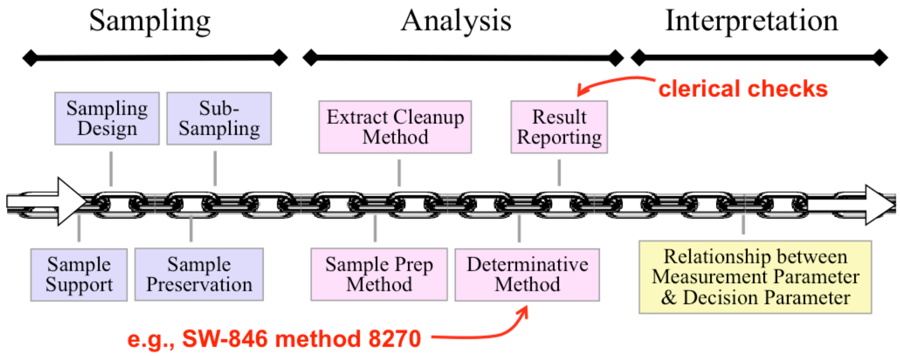
Subsampling Errors

- Opportunities to unintentionally discriminate against certain particle sizes
  - Scoop from top of sample jar
    - Particles segregated during transport or incorrect mixing (smallest particles fall to the bottom)
  - Shape of the scoop used to subsample the jar
  - Preferentially tapping in small particles to bring mass close to nominal when weighing out the analytical subsample

Historically, the DQO Focus Has Been on Analytical Quality

- Emphasis on fixed-base laboratory analyses following well-defined protocols
- Analytical costs driven to a large degree by QA/ QC requirements
- Result:
  - Analytical error typically small, on order of 30% or less for replicate analyses on an extract or control sample
  - Traditional laboratory data treated as "definitive"
    - But definitive (definite) about what?

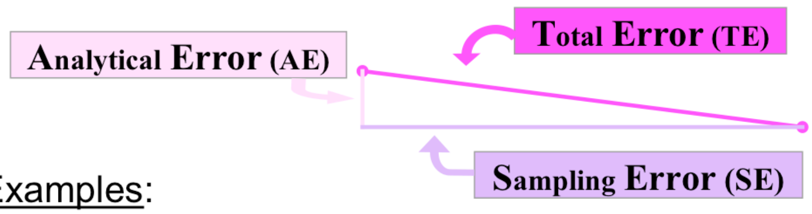
Decision Quality Only as Good as the Weakest Link in the Data Quality Chain



Each link represents a **variable** contributing toward the quality of the analytical result. All links in the **data quality chain** must be intact for data to be of decision-making quality!

Analytical vs. Sampling Error

Uncertainties (errors) add according to $(a^2 + b^2 = c^2)$



Examples:

- AE = 10 ppm, SE = 80 ppm: **TE = 81 ppm** (baseline)
- AE = 5 ppm, SE = 80 ppm: **TE = 80 ppm** (reduce analytical)
- AE = 10 ppm, SE = 40 ppm: **TE = 41 ppm** (reduce sampling)
- AE = 20 ppm, SE = 40 ppm: **TE = 45 ppm** (inc AE & dec SE)

How is Data Uncertainty Reduced?

- For big analytical errors:
  - Modify current analytical method or choose a different analytical technique
  - Improve QC on existing techniques
- For subsampling errors:
  - Improve sample preparation (better homogenization & larger subsamples)
- For errors due to low sampling density in the field: **Collect samples from more locations!**

More Fundamentals: Basic Statistical Terms

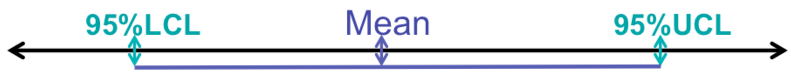
- **Mean:** population average over an area of interest
- **Median:** half of the population distribution is below the median, and half above; middle value or 50th percentile
- **Range:** interval defined by the minimum and maximum values
- **Variance:** a measure of the "spread" of values

More Basic Statistical Terms

- **Standard Deviation:** square root of the variance
- **Coefficient of Variation (CV):** ratio of standard deviation to mean, a measure of relative variability
  - also called relative standard deviation (RSD)
- **Skewness:** the degree to which one end of a statistical distribution is pulled out to one side
- **Confidence Interval:** range of values that estimates the uncertainty around a point estimate, such as a data result or a mean.
  - the true value is expected to be somewhere within that range some given amount (such as 95%) of the time

Using Confidence Intervals to Communicate Uncertainty

- **Confidence level** is usually set at **95%** by default, but others can be used
- The values at each end of the interval are called the **confidence limits**: lower (LCL) and upper (UCL)



- Values between the confidence limits make up the **confidence interval** around the mean
- Width of confidence interval driven by: the confidence level, variability present in the data, assumptions about underlying data distribution & number of data points (n).

The Mismatch Between Statistics and Environmental Sampling

- In traditional statistics, the population units being sampled are clearly defined:
  - How many people with blood type O? unit = 1 person
- Sampling of soil or water has no unique units
  - Any amount can be collected. What is correct volume? **Trouble is: volume determines the concentration!!**
- No spatial considerations in traditional statistics
  - **But, spatial relationships are paramount in environmental sampling**

More Terms

- **Decision Unit (DU):** the volume of soil or set of objects treated as **single unit** for decision-making
  - Such as ¼-acre area to 2-inch depth, a bin of soil, a set of drums
  - Examples: exposure units, survey units, remediation units…
- **Population:** Set of objects or material volumes sharing a common characteristic
  - Can be same object set as DU, but doesn't need to be

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Example of Setting Decision Units (DUs)

Future residential lots, DUs sized as exposure areas (EUs)

Pesticide mixing area, DUs sized to assist remediation

Smaller DUs in high-suspect areas to optimize potential remedial costs

We Can Pretend Heterogeneity Doesn't Exist, But Data Quality Suffers

Module 1.3

Incremental-Composite Sampling Designs for Surface Soil Analyses

Terminology and Existing Guidance

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What's in a Name?

MIS

Composite sampling

ISM

ICS

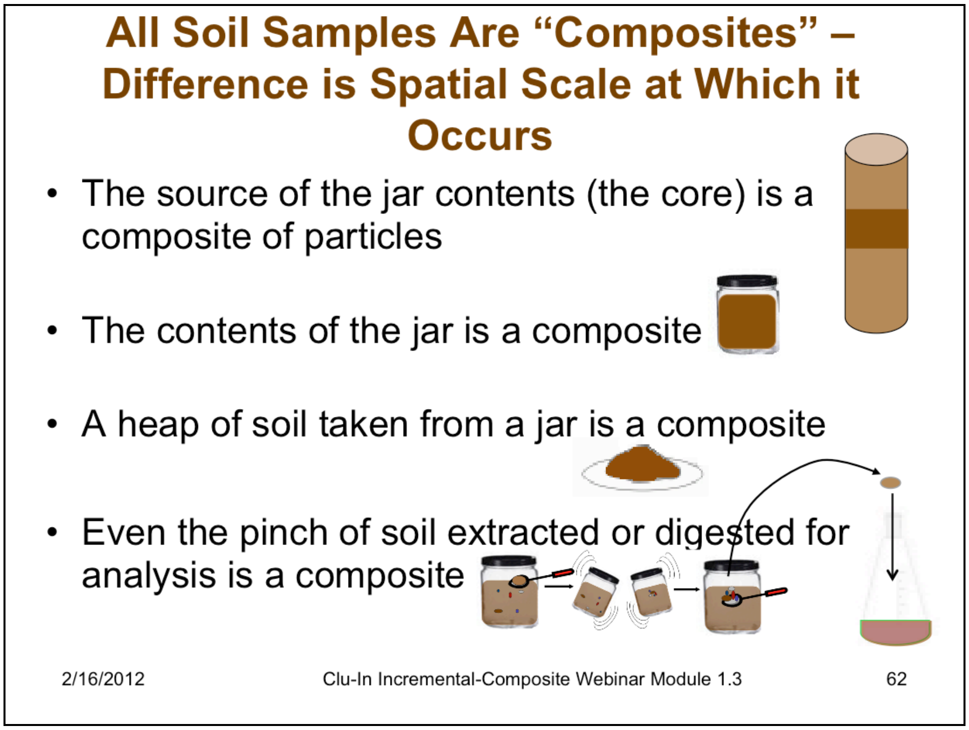
Incremental sampling

“Composite” or “Multi-Increment” or “Incremental” Sampling

- Composite sampling - term used since 1985 in USEPA guidance to convey idea of “pooling” for several purposes
- Multi-Increment Sampling ® – term coined by Chuck Ramsey which he has trademarked as “MIS”
- Incremental sampling methodology (ISM) – term used by ITRC due to copyright infringement concern over “MIS”; goal is to obtain average concentration over DU
- Incremental-composite sampling – USEPA term to combine ITRC’s ISM with features from USEPA’s existing compositing guidance; goals include more than the average

All Share Common Characteristics

- All refer to collecting soil samples:
  - From physically separate locations ("increments")
  - Then pooling to form one homogenized sample (termed a "composite sample" or "incremental sample")
- Primary difference lies in their purpose; e.g., finding an average (incremental) vs searching for hot spots (a compositing technique)
- MIS ® involves a specific incremental sampling protocol that has been optimized for explosives residues, but can be generalized to other analytes



When we think about measuring the characteristics of a human population (e.g., average height), the basic sampling unit is well-defined and obvious: a person.

Within the world of soil samples, however, there is no basic sampling unit that has been defined by guidance and is universally used. The sampling unit (or sampling support) varies from project to project; different sampling units are often used at different times for the same project.

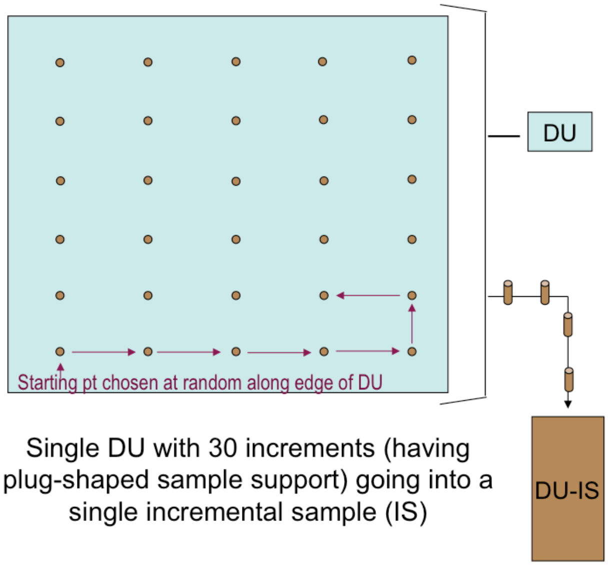
At the physical scale that most sampling work is done, the soils that one collects, prepares, and analyzes are aggregates or “composites” consisting of lots of tiny soil particles. This is true for the pinch of soil used for an extraction, the heap of soil that the original pinch was drawn from, the jar from which the heap of soil came, and the soil core that yielded jar of soil.

Incremental vs. Composite Sampling
Basic Differences (1)

- Goal of incremental sampling: estimate the average concentration over some defined area/volume of soil
- This defined soil area/volume is the subject of a decision of e.g., risk/no risk or exceedance/no exceedance
  - Therefore, the defined area/volume is called a decision unit (DU)
  - Having 3 or more independent replicates allows calculation of an upper confidence limit (UCL) on the mean (i.e., a conservative estimate of the mean concentration)

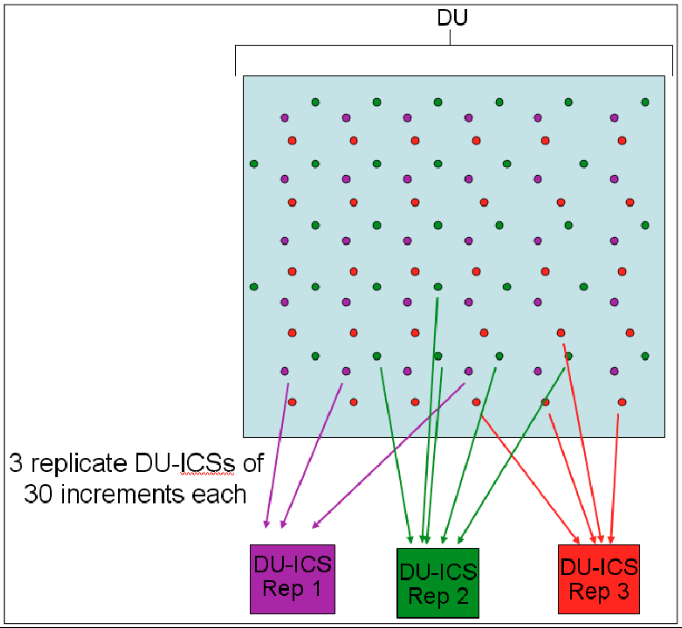
Incremental Sampling

Taking a single incremental sample over a decision unit (DU)



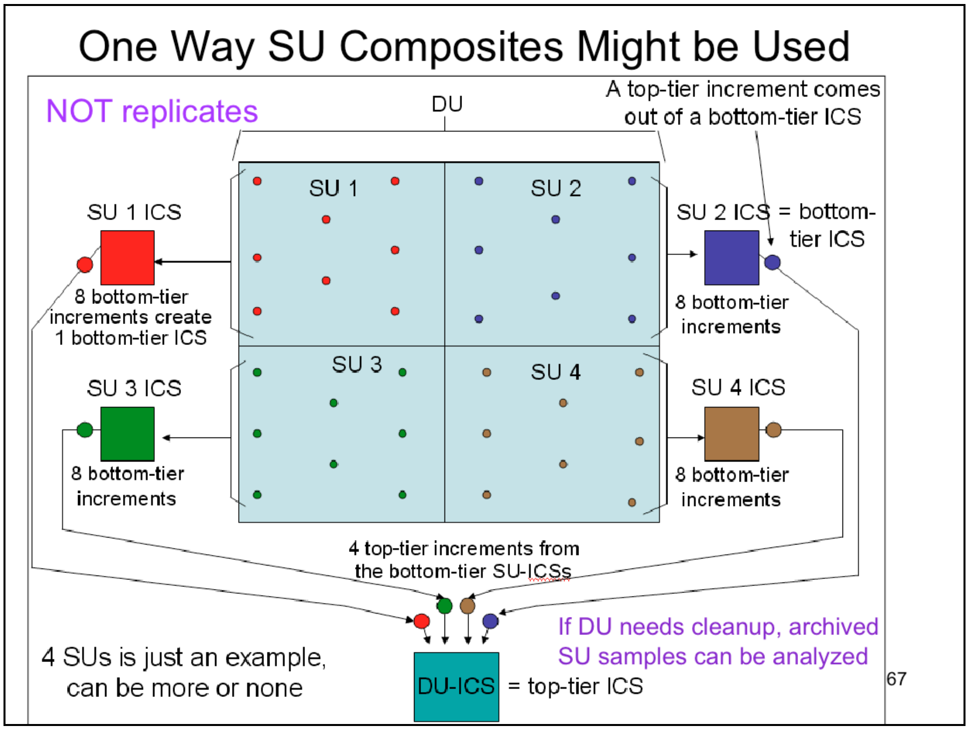
Single DU with 30 increments (having plug-shaped sample support) going into a single incremental sample (IS)

Replicate Incremental Samples



Incremental vs. Composite Sampling
Basic Differences (2)

- Common goal of composite sampling: gain information about contaminants' spatial distribution
  - Accurate or conservative estimate of the concentration mean within the given area not required
  - The "given area" is called a sampling unit (SU); typically smaller than the DU
  - Composite replicates not typically used
  - Improves representativeness of "single" sample representing a small area by suppressing short-scale heterogeneity
- Composite uses fewer increments per sample than incremental sampling


One Way SU Composites Might be Used
NOT replicates
DU
A top-tier increment comes out of a bottom-tier ICS
SU 1
SU 2
SU 3
SU 4
SU 1 ICS
8 bottom-tier increments create 1 bottom-tier ICS
SU 2 ICS = bottom-tier ICS
8 bottom-tier increments
SU 3 ICS
8 bottom-tier increments
SU 4 ICS
8 bottom-tier increments
4 top-tier increments from the bottom-tier SU-ICSs
If DU needs cleanup, archived SU samples can be analyzed
4 SUs is just an example, can be more or none
DU-ICS
= top-tier ICS
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MIS®, ISM and ICS Requirements

- Upfront **systematic planning**!!!
- QAPP must discuss
  - Rationale for **DU** & **increment number**
  - Detailed procedures for field **sample collection**
  - Detailed instructions to lab for **sample processing**
    - Sample mass reduction/spkitting? Sieving? Grinding? Target particle size? Incremental subsampling? Mass of analytical subsample?
  - Decision-making based on IS result (DU average) or UCL?
  - **QC** for the ISM/ICS process (Replicate ISs? Replicate subsamples?) Corrective actions?

A Long History of EPA Composite Sampling Guidance

- 6 documents since 1985 go into some depth
  - 1985 PCB Technical Guidance
  - 1995 EPA Observational Economy Series
  - 1996 Soil Screening Guidance, Part 4
  - 2002 EPA RCRA Sampling Technical Guidance
  - 2002 EPA QA/G-5S
  - 2006 EPA SW-846 Method 8330B (App. A)
- They do not cover all potential issues or details, but do provide a framework

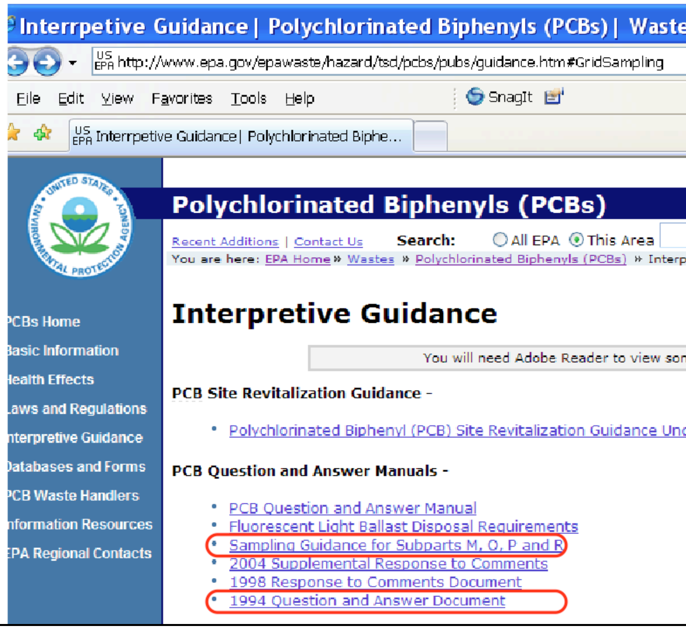
G-5S p 121

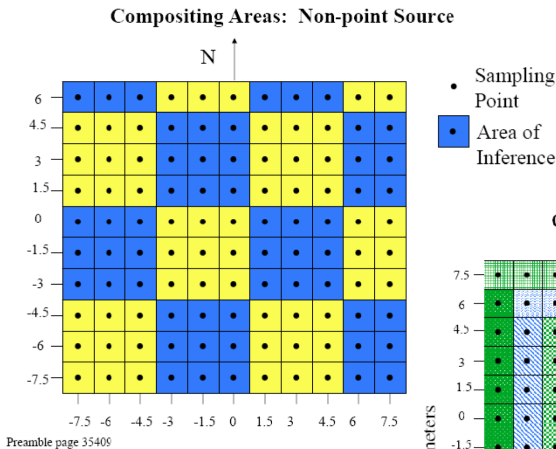
In the first two cases (Sections 10.2 and 10.3), there is interest in *making an estimate for a prescribed target population*—in the first case estimating the mean of a continuous measure (e.g, the mean concentration of contaminant) and in the second case estimating the proportion of the population with a characteristic. In these two cases, carrying out the Composite sampling means combining a sampling design with a compositing protocol. The sampling design describes the method for selecting units from the target population and indicates the number of units to be selected and which ones are to be selected. The compositing protocol describes the scheme for forming and processing (mixing and homogenizing) composites. It indicates whether entire samples or aliquots are to be combined, the number of groups of units to be formed (m), the number of units per group (k), which units form each group, and the amount of material from each unit to be used in forming the composite sample.

The last two cases (Sections 10.4 and 10.5) involve *decision making at the unit level* rather than at the target population level. As a consequence, these approaches involve composite sampling and retesting protocols that not only define how composites are to be formed but also define when and how subsequent testing is to be done to ultimately identify particular units. The retesting strategies for these cases are conditional on the results obtained for the composites. In order to retest individual samples, the identity and integrity of the individual samples must be maintained; this implies that aliquots from the individual samples, rather than the whole samples, must be combined in forming composites. Additional aliquots from the individual samples are then retested either singly or in other composites.

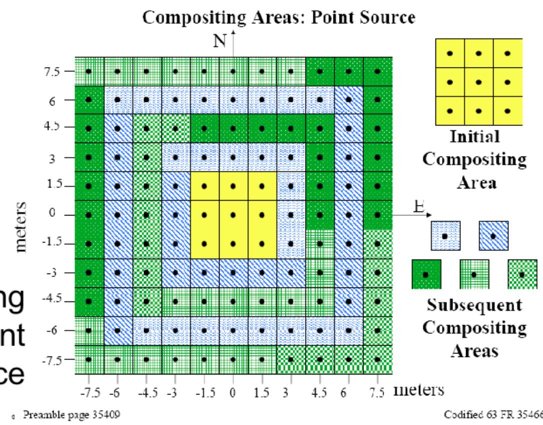
RCRA Waste Sampling Draft Technical Guidance: Planning, Implementation, and Assessment, EPA530-D-02-002, August 2002 (formerly SW-846 Chapter 9, until pulled out to expand sampling into its own document) This document and SW-846 Method 8330B contain the most implementation details.

1985 PCB Guidance on www.EPA.gov http://www.epa.gov/epawaste/hazard/tsd/pcbs/pubs/subpartmopr.pdf



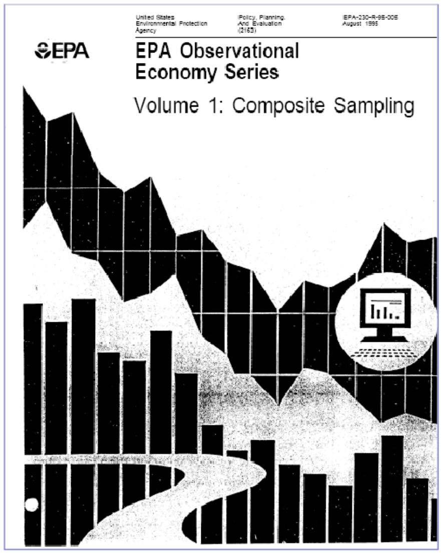


Characterizing an area while controlling short-scale heterogeneity



Determining contaminant extent around a point source

1995 Composite Sampling Guidance
www.triadcentral.org/ref/ref/documents/composite.pdf



August 1995

EPA-230-R-95-005

In 1995 EPA was Confident Compositing Would Catch On

In light of recent developments, composite sampling is increasingly becoming an acceptable practice for sampling soils, biota, and bulk materials when the goal is estimation of some population value under restrictions of a desired standard error and/or limits on the cost of sample measurement.

In response to an informal survey of various professionals, several favorable applications of composite sampling were received. They include:

- Establishing and verifying attainment of remedial cleanup standards in soils using sample compositing and bootstrapping techniques
- Use of compositing to obtain adequate support in geostatistical sampling
- Optimal compositing strategies for screening material for deleterious agents
- A soil sample design utilizing techniques of compositing, binary search, and confidence limits on proportions

pp 7 & 8

3.1. Soil Sampling

3.1.1. Characterization of Soil PCB Contamination at Gas Pipeline Compressor Stations

As part of a recent settlement between the Pennsylvania Department of Environmental Resources and the Texas Eastern Pipeline Company, PCB-contaminated soils had to be characterized and remediated at 19 sites. Because waste sources included indiscriminate dumping, disposal in trash pits, air emissions and even application as weed killer along fence lines, the resulting spatial distribution of contaminated soil was very heterogeneous, with hot spot locations unknown. Therefore, the only way to reliably characterize these sites required a very large number of soil samples, around 12,000 to be more precise. With each sample analyzed for total PCB,s, the cost for site characterization alone was around $33 million. Now to really appreciate the magnitude of the problem, one must realize this discussion only pertains to the Pennsylvania settlement. The problem extends along the whole pipeline from the Gulf Coast to New England.

Results of a retrospective study (Gore, Patil, and Taillie, 1992; Patil, Gore and Sinha, 1994), using the actual site characterization data, revealed that composite sampling methods potentially could have substantially reduced the analytical costs.

Three aspects of the data were evaluated: (i) estimation of the mean and variance of total PCB concentration as well as total PCB mass, (ii) classification of each individual (uncomposited) sample as above or below a specified critical level, and (iii) quantification of those individual samples with the highest PCB levels.

Results showed that unbiased estimates of the mean and variance could be obtained with one fourth the number of analyses (90 instead of 360). A small loss of precision resulting from compositing seemed quite acceptable in light of large analytical cost reduction.

Simulations run from actual site data show compositing produces accurate estimates of the mean but with much fewer analyses.

pp. 8 & 9

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EPA Observational Economy Series: Composite Sampling (1995) pages 8 & 9

1995 EPA Compositing Guidance, p. 9

A site was acceptably clean if 90% of the measured samples were below 10 parts per million (ppm) with no values exceeding 25 ppm. With characterization data from the worst of the nineteen sites, compositing could have reduced the analytical cost of classifying individual samples according to the 10 ppm criterion by 9%, relative to exhaustive testing. Starting from this nearly worst case scenario, the cost savings increase as we move to cleaner sites and should be dramatic when analyzing post-remediation verification data. For example, another site along the pipeline that is cleaner, although still contaminated, could have had all individual samples classified according to 10 ppm for 50% less of the analytical cost associated with exhaustive testing. (See Gore, Boswell, Patil, and Taillie, 1992).

Finally, if concerned with simply knowing which individual sample has the highest concentration, we could have discovered this by exhaustively retesting just two composite samples. In other words, with only eight measurements in addition to the 90 composite measurements, we could have identified the "hottest" spot. Furthermore, 12 additional measurements could have revealed the locations with the four highest concentrations (See Patil, Gore and Sinha, 1994).

Instead of 360 analyses, 98 provides an unbiased estimate of mean & variance AND finds **the** highest hotspot

A total of 110 analyses would find mean + the **4** highest hotspots

Put out by the SW-846 team

United States Environmental Protection Agency | Solid Waste and Emergency Response (5305W) | EPA530-D-02-002 August 2002 www.epa.gov/osw

Office of Solid Waste

EPA

RCRA Waste Sampling Draft Technical Guidance

Planning, Implementation, and Assessment

URL: www.epa.gov/epawaste/hazard/testmethods/sw846/samp_guid.htm

EPA530-D-02-002 **RCRA Waste Sampling Draft Technical Guidance** Planning, Implementation, and Assessment (Aug 2002)

http://www.epa.gov/epawaste/hazard/testmethods/sw846/samp_guid.htm

RCRA Waste Sampling Draft Technical Guidance
Planning, Implementation, and Assessment

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RCRA Waste Sampling Draft Technical Guidance
Planning, Implementation, and Assessment

Provides discussion of Gy theory and subsampling issues

United States Environmental Protection Agency | Office of Environmental Information Washington, DC 20460 | EPA/240/R-02/005 December 2002

EPA

Guidance on Choosing a Sampling Design for Environmental Data Collection

for Use in Developing a Quality Assurance Project Plan

EPA QA/G-5S

Quality

URL: www.epa.gov/quality/qs-docs/g5s-final.pdf

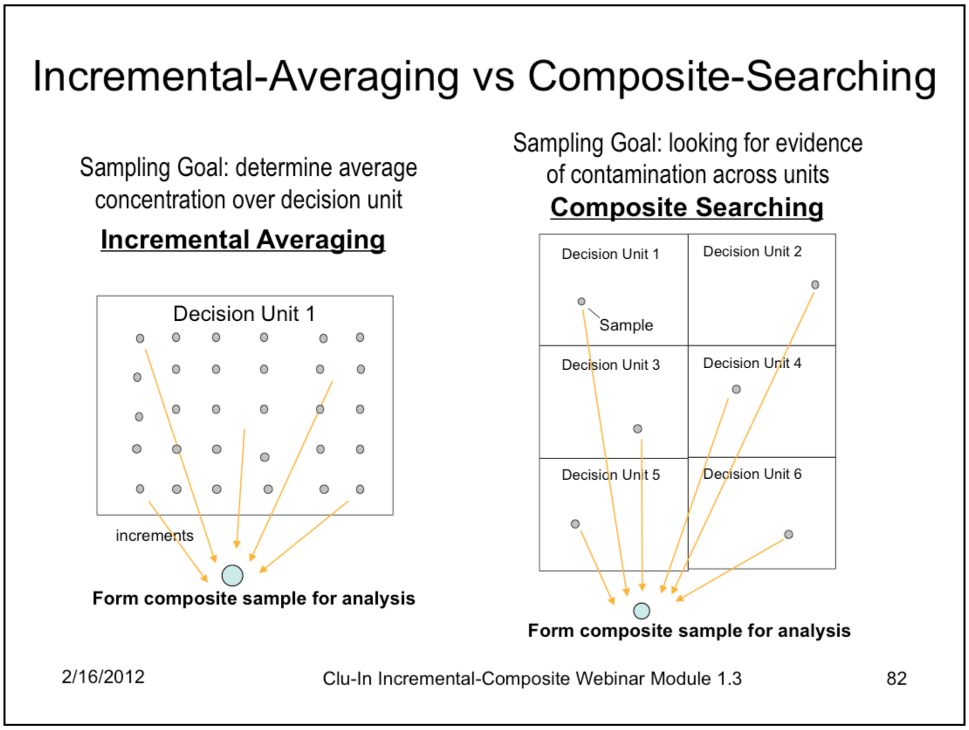
36 pages of text & appendices on composite sampling

79

http://www.epa.gov/quality/qs-docs/g5s-final.pdf

"INCREMENTAL-AVERAGE"

"COMPOSITE-SEARCHING"



Here's the distinction between composite averaging and composite searching for the purposes of this presentation.

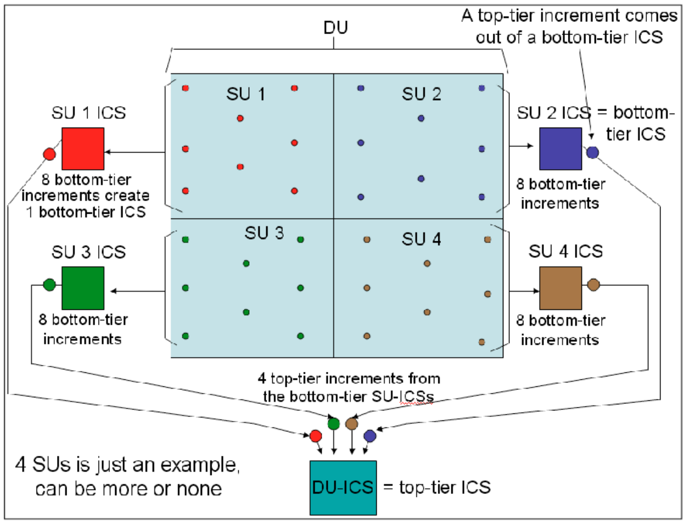
In the case of composite averaging (figure on the right), we collect multiple samples or soil increments from within a decision unit and then combine them into a composite sample for analysis. Our goal is to determine whether the average concentration within the decision unit is less than some cleanup criterion.

In the case of composite searching (figure on the left), we collect multiple samples or soil increments from either across decision units (as illustrated here) or within decision units and then combine them into a composite sample for analysis. Our goal is to determine whether any of those original increments might have had contamination levels above some specified threshold that would be indicative of the presence of contamination at levels of concern.

The balance of this discussion will focus on composite averaging. Composite searching will be discussed later (also referred to as adaptive compositing).

An important side point: "Dilution" is not a concern for composite averaging. "Dilution" is a concern for composite searching.

2 Tiers of Compositing: Composite-Searching Using 2 Tiers of Composite Sampling



Bottom-tier composites must WELL processed before splittig portion for archive & another portion to go into top-tier composite

SW-846

Incremental Sampling Recommended in Energetic Residues Analysis Method

http://www.epa.gov/epawaste/hazard/testmethods/pdfs/8330b.pdf

METHOD 8330B

NITROAROMATICS, NITRAMINES, AND NITRATE ESTERS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

APPENDIX A

COLLECTING AND PROCESSING OF REPRESENTATIVE SAMPLES FOR ENERGETIC RESIDUES IN SOLID MATRICES FROM MILITARY TRAINING RANGES

FORWARD

The information provided in this Appendix is based on EPA's evaluation of currently available data and technology as applied to the most appropriate sample collection, handling and processing procedures to determine representative concentrations of energetic material residues in solid matrices, such as soils, solid waste, or sediments. These procedures are designed to minimize the random error associated with heterogeneity of constituents that are distributed as particles into the environment. The intended users of this Appendix guidance are those individuals and organizations involved in the collection and preparation of samples for energetic material residue analysis during the characterization of solid materials under the Resource Conservation and Recovery Act (RCRA). The procedures and techniques described in this Appendix are not presented in any preferential order nor do they represent EPA

EPA/540/1-89/002
December 1989

**Risk Assessment
Guidance for Superfund
Volume I
Human Health Evaluation Manual
(Part A)**

Interim Final

RAGS Vol 1, Part A 1989

http://epa.gov/oswer/riskassessment/ragsa/index.htm

What Does RAGS 1989 Say?

Page 4-11

Heterogeneous nature of soils. One of the largest problems in sampling soil (or other solid materials) is that its generally heterogeneous nature makes collection of representative samples difficult (and compositing of samples virtually impossible -- see Section 4.6.3). Therefore, a large number of soil samples may be required to obtain sufficient data to calculate an exposure concentration. Composite samples sometimes are collected to obtain a more homogeneous sample of a particular area; however, as discussed in a later section, compositing samples also serves to mask contaminant hot spots (as well as areas of low contaminant concentration).

Designation of hot spots. Hot spots (i.e., areas of very high contaminant concentrations) may have a significant impact on direct contact exposures. The sampling plan should consider characterization of hot spots through extensive sampling, field screening, visual observations, or a combination of the above.

Page 4-19

Composite samples. Composite samples -- sometimes referred to as continuous samples for air -- combine subsamples from different locations and/or times. As such, composite samples may dilute or otherwise misrepresent concentrations at specific points and, therefore, should be avoided as the only inputs to a risk assessment. For media such as soil, sediment, and ground water, composite samples generally may be used to assess the presence or absence of contamination; however, they may be used in risk assessment only to represent average concentrations (and thus exposures) at a site. For example, "hot spots" cannot be determined using composite samples.

This statement was written before other EPA guidance was issued that explained how to use compositing to find hotspots.

United States Environmental Protection Agency | Office of Solid Waste and Emergency Response Washington, D.C. 20460 | Publication 9285.7-08I May 1992

Supplemental Guidance to RAGS: Calculating the Concentration Term

http://rais.ornl.gov/documents/UCLsEPASupGuidance.pdf

Calculating the Concentration Term – EPA 1992

Page 3

Why use the UCL as the average concentration?

Statistical confidence limits are the classical tool for addressing uncertainties of a distribution average. The 95 percent UCL of the arithmetic mean concentration is used as the average concentration, because it is not possible to know the true mean. The 95 percent UCL, therefore, accounts for uncertainties due to limited sampling data at Superfund sites. As sampling data become less limited at a site, uncertainties decrease, the UCL moves closer to the true mean, and exposure evaluations using either the mean or the UCL produce similar results. This concept is illustrated in **Highlight 2**.

Calculating the Concentration Term – EPA 1992

Page 3

Should a value other than the 95 percent UCL be used for the concentration?

A value other than the 95 percent UCL can be used, provided the risk assessor can document that high coverage of the true population mean occurs (i.e., the value equals or exceeds the true population mean with high probability). For exposure areas

Publication 9285.7-47
December 2001

Risk Assessment Guidance for Superfund: Volume I Human Health Evaluation Manual (Part D, Standardized Planning, Reporting, and Review of Superfund Risk Assessments)

Final

RAGS Vol 1, Part D 2001

http://epa.gov/oswer/riskassessment/ragsd/index.htm

RAGS 2001 guidance mentions compositing only once, but in a way that implies acceptance

Page 2-3

pathway (both current and future) and medium. The SAP should be accompanied by detailed sampling maps showing the location and type of samples (e.g., grab, composite, or duplicate). It is important to consider how sample results will be used to estimate exposure point concentrations. Background samples should be collected from appropriate areas (e.g., areas proximate to the site, free of potential contamination by site chemicals and similar to the site in topography, geology, meteorology, and other characteristics).

OSWER 9285.6-10

December 2002

CALCULATING UPPER CONFIDENCE LIMITS FOR EXPOSURE POINT CONCENTRATIONS AT HAZARDOUS WASTE SITES

http://www.epa.gov/oswer/riskassessment/pdf/ucl.pdf

Calculating UCLs…Dec 2002

page 1

of a chemical in the environment. This concentration, commonly termed the exposure point concentration (EPC), is a conservative estimate of the average chemical concentration in an environmental medium. The EPC is determined for each individual exposure unit within a site. An exposure unit is the area throughout which a receptor moves and encounters an environmental medium for the duration of the exposure. Unless there is site-specific evidence to the contrary, an individual receptor is assumed to be equally exposed to media within all portions of the exposure unit over the time frame of the risk assessment.

EPA recommends using the average concentration to represent "a reasonable estimate of the concentration likely to be contacted over time" (EPA 1989). The guidance previously issued by EPA in 1992, *Supplemental Guidance to RAGS: Calculating the Concentration Term* (EPA 1992), states that, "because of the uncertainty associated with estimating the true average concentration at a site, the 95 percent upper confidence limit (UCL) of the arithmetic mean should be used for this variable."

Incremental sampling reduces uncertainty in estimates of the true average

United States Environmental Protection Agency

Office of Solid Waste and Emergency Response Washington, DC 20460

EPA/540/R95/128 May 1996

Superfund

EPA

Soil Screening Guidance: Technical Background Document

URL = http://www.epa.gov/superfund/health/conmedia/soil/index.htm

Part 4: MEASURING CONTAMINANT CONCENTRATIONS IN SOIL

Compositing "how to" covered on pages 81-132

SSG-TBD page 5

1.3.2 Exposure Assumptions. SSLs are risk-based concentrations derived from equations combining exposure assumptions with EPA toxicity data. The models and assumptions used to calculate SSLs were developed to be consistent with Superfund's concept of "reasonable maximum exposure" (RME) in the residential setting. The Superfund program's method to estimate the RME for chronic exposures on a site-specific basis is to combine an average exposure point concentration with reasonably conservative values for intake and duration in the exposure calculations (U.S. EPA,

SSG-TBD page 89

Note that the size, shape, and orientation of sampling volume (i.e., "support") for heterogenous media have a significant effect on reported measurement values. For instance, particle size has a varying affect on the transport and fate of contaminants in the environment and on the potential receptors. Because comparison of data from methods that are based on different supports can be difficult, defining the sampling support is important in the early stages of site characterization. This may be accomplished through the DQO process with existing knowledge of the site, contamination, and identification of the exposure pathways that need to be characterized. Refer to *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (U.S. EPA, 1992f) for more information about soil sampling support.

SSG-TBD pages 89 & 90

Compositing. Because the objective of surface soil screening is to ensure that the mean contaminant concentration does not exceed the screening level, the physical "averaging" that occurs during compositing is consistent with the intended use of the data. Compositing allows a larger number of locations to be sampled while controlling analytical costs because several discrete samples are physically mixed (homogenized) and one or more subsamples are drawn from the mixture and submitted for analysis. If the individual samples in each composite are taken across the EA, each composite represents an estimate of the EA mean.

A practical constraint to compositing in some situations is the heterogeneity of the soil matrix. The efficiency and effectiveness of the mixing process may be hindered when soil particle sizes vary widely or when the soil matrix contains foreign objects, organic matter, viscous fluids, or sticky material. Soil samples should not be composited if matrix interference among contaminants is likely (e.g., when the presence of one contaminant biases analytical results for another).

Before individual specimens are composited for chemical analysis, the site manager should consider homogenizing and splitting each specimen. By compositing one portion of each specimen with the other specimens and storing one portion for potential future analysis, the spatial integrity of each specimen is maintained. If the concentration of a contaminant in a composite sample is high, the splits of the individual specimens from which it was composed can be analyzed discretely to determine which individual specimen(s) have high concentrations of the contaminant. This will permit the site manager to determine which portion within an EA is contaminated without making a repeat visit to the site.

Designation: D 6051 – 96 (Reapproved 2006)

INTERNATIONAL
Standards Worldwide - Home

Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities[1]

1. Scope

1.1 Compositing and subsampling are key links in the chain of sampling and analytical events that must be performed in compliance with project objectives and instructions to ensure that the resulting data are representative. This guide discusses the advantages and appropriate use of composite sampling, field procedures and techniques to mix the composite sample and procedures to collect an unbiased and precise subsample(s) from a larger sample. It discusses the advantages and limitations of using composite samples in designing sampling plans for characterization of wastes (mainly solid) and potentially contaminated media. This guide assumes that an appropriate sampling device is selected to collect an unbiased sample.

"Incremental Sampling" per ITRC

A structured sampling and subsampling protocol for representative & reproducible sampling of a well-defined soil area or volume (a decision unit) to obtain a concentration value representative of the average concentration over the entire decision unit

ITRC ISM-1 document at a URL to be announced; or do Internet search for "ITRC ISM"

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• ITRC = Interstate Technology and Regulatory Council (http:www.itrcweb.org)

• The ITRC ISM-1 document is a web-based document to accommodate a wealth of hyperlinks between sections and to additional information and pop-up definitions. The entire document will be able to be printed, but is over 600 pages long due to the number of appendices and case examples.

Other Recent Guidance Documents

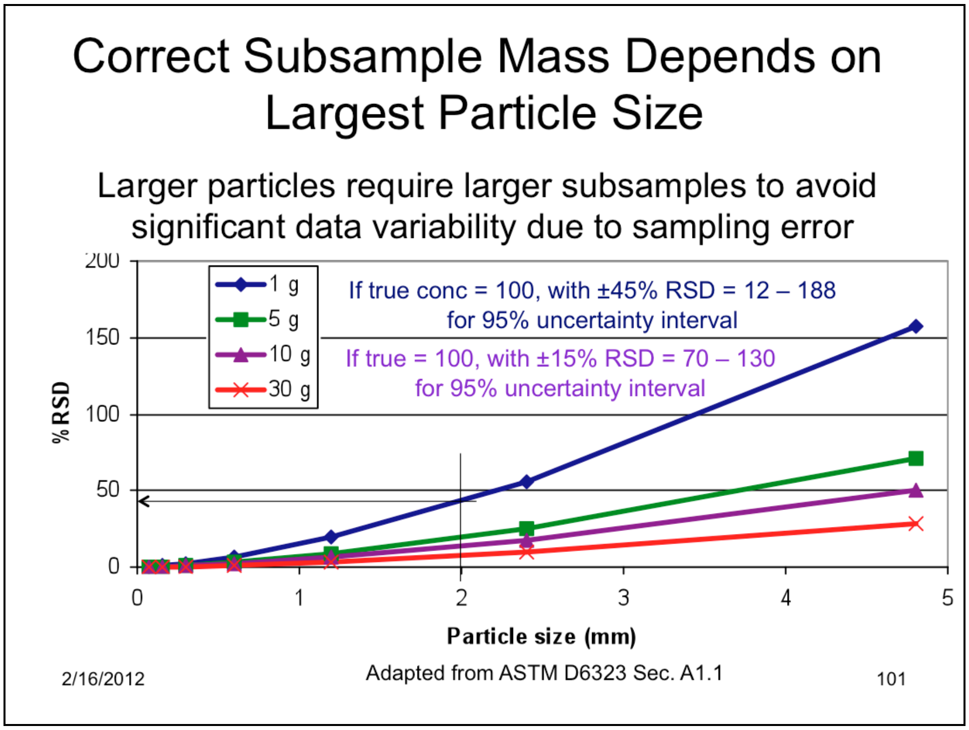
- Hawaii state guidance for MIS at http://www.hawaiidoh.org/tgm-pdfs/HTGM%20Section%2004-02.pdf
- Alaska state guidance for MIS at http://dec.alaska.gov/spar/csp/guidance/multi_increment.pdf
- Army Corps of Engineers http://www.hnd.usace.army.mil/oew/policy/IntGuidRegs/IGD%209-02v2.pdf

Key Components of Any Good Sampling Design

- **Project Planning & Field Sample Collection**
  - Well-formulated & explicitly stated sampling objectives to define the decision unit (DU).
  - Determine whether data goal is only to determine DU average, or also to preserve spatial information.
- **Sample Processing and Subsampling**
  - Sample processing may begin in the field and finish in the lab, or all be done in the lab.
  - Goal: maintain chain of sample representativeness for each step thru subsampling.
  - As important for discrete designs as for ICS!

• In the case of ICS designs, collecting multiple increments of uniform mass/ volume in an unbiased manner within the decision unit involves 1 of 2 strategies: Collect an incremental sample across the entire decision unit so that a single incremental sample represents the entire decision unit, or collect a composite sample that represents the average concentration across a smaller area within the decision unit. For the latter case, the entire area of the DU is split between several areas called sampling units (SUs), each of which is represented by a composite sample. SUs preserve spatial information about contaminant concentrations, which can be accessed later should the need arise (such as the DU average exceeds a decision threshold such that cleanup is needed).

• Maintaining the chain of sample representativeness at each step of sample/ increment collection may require sieving, grinding and/or increasing the mass of the analytical subsample. This is as critical for discrete samples as for incremental-composite samples. If representativeness is lost at the subsampling stage, the work that went into ensuring representativeness as the field level is wasted.



- ASTM D6323 (2003) Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities (www.astm.org)
- The graph shows that as particle size gets smaller, the mass of the subsample needed to obtain good precision gets smaller. The reason why grinding to small particle sizes (like 75 microns) is important for metals is that very small analytical subsamples are used, usually between 0.5 to 2 grams of soil. Even if sieving to 2 mm is performed, when only 1 gram is used (blue curve), the minimum variability in the data (given as %RSD) is extreme. (recall that as %RSD increases, variability increases and precision decreases)
    - As an example, say you have a sample with a true Pb concentration of 100 ppm. The soil was sieved through a 10-mesh sieve (2 mm screen size) and everything larger than 2 mm was removed from the sample. Great care was taken to subsample in a representative way, but only 1 gram of that sieved soil is taken for the analytical subsample. The BEST precision that can be expected (no matter how carefully the tech performs the sampling) for the 100 ppm sample is for results to fall within the range of 12 to 188 ppm 95% of the time. That is a very wide range.
    - From this minimum starting point, the variability only gets worse if sloppy subsampling techniques are used.
- Increasing the analytical subsample mass to 10 grams (purple curve) significantly improves the minimum %RSD (i.e., the best possible precision) even if the largest particle size stays at 2 mm. If the true concentration of the sample is 100, and great care is exercised during subsampling, 95% of the time 10 gram subsamples will provide results in the range of 70 to 130 ppm.
- On the other hand, if the particle size is reduced to less than 1/10th of a mm, the minimum variability for a 1-gram subsample is around 5 %RSD. This means that results for a 100 ppm sample could fall, at best, between 90 and 110 ppm 95% of the time.

Sample Processing Prior to Subsampling

- Obviously depends on soil type, moisture, etc.)
  - Drying: oven or air-drying
  - Disaggregation: breaking up clods
  - Sieving: separate out particle size fraction of interest
  - Grinding: mills & grinders (under some circumstances)

Gy theory guides sample handling & subsampling to produce the analytical sample

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Off-the-shelf coffee grinders and mortar/pestles are examples of tools that can aid disaggregation.

Heavy-duty grinders/mills that can reduce particle size to <100 microns include: rotary pulverizers, ball mills, and puck mills. For more information on sample processing, see ITRC ISM-1 Section 6.2.2.5

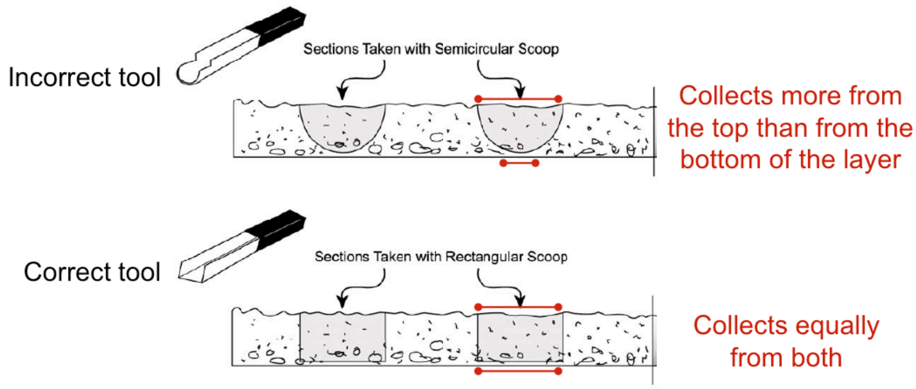
Gy theory is the theory of sampling particulate materials. More information concerning Gy theory can be found in the ITRC ISM-1 document, EPA/600/R-03/027 (subsampling guidance) and EPA/530-D-02-002 (SW-846/RCRA sampling guidance).

EPA/600/R-03/027
November 2003

Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples

http://www.clu-in.org/download/char/epa_subsampling_guidance.pdf

“Incorrect” vs. “Correct” Subsampling



Subsampling with the rounded scoop preferentially gathers particles from the top and discriminates against those at the bottom. The correctly shaped tool is shown in the bottom panel. (Adapted from EPA/600/R-03/027)

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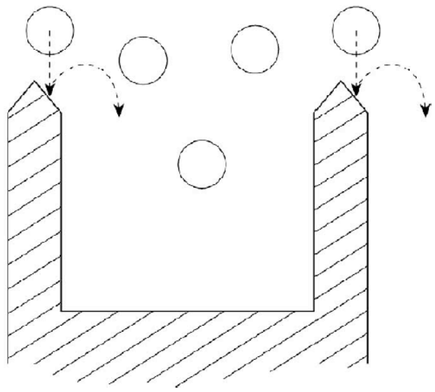
Ref: Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples EPA/600/R-03/027 Nov 2003

• An incorrect subsampling tool allows some portions of the soil sample population to have a greater probability than others of inclusion in the analytical subsample. The soil sample population is the portion of the matrix that has been defined as the target, such as all particles smaller than 2 mm diameter. If all particles less than 2 mm is the target population, the subsampling tool cannot discriminate against (for example) particles in the 1- to 2-mm range vs. particles smaller than 1 mm.

• One example of discrimination can happen when the soil sample has segregated in the jar so that the larger particles are predominately at the top of the jar. Then when the soil in the jar is poured out into a “slab” for subsampling, the larger particles end up at the bottom of the slab. As depicted in the figure, the larger particles are less likely to be chosen by the rounded scoop.

• Another example of discrimination against larger particles can occur if the subsampling tool is shallow and narrow (as are some spatulas), so that larger particles roll off the tool.

• An example of discriminating against smaller particles can occur when soil is scooped off the top of jar contents which have segregated such that finer particles have mostly settled to the bottom out of reach of the sampling tool.



The 2003 EPA Subsampling Guidance

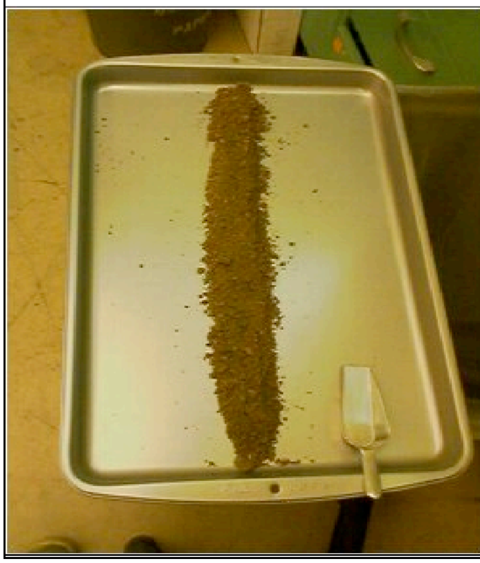
The sampling tool "mouth" should be at least 3 times the size of the largest particle + 10 for particles <3 mm.
For 2-mm particles, a "mouth" of 16 mm is recommended.

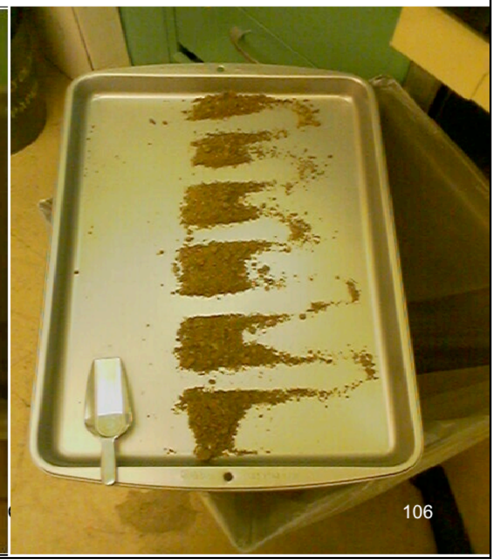
Vertical view of a correct sampling tool that gives all particles equal chance of being included in the subsample depending only on where their center of gravity lies with respect to the cutting edge of the tool. (From EPA/600/R-03/027)

Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples EPA/600/R-03/027 Nov 2003

- A tool is "correct" if, as the tool is pushed forward, particles are included or excluded with equal probability.

1-D Slab Cake Sample Mass Reduction





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2-D Slab Cake Sample Mass Reduction

Field subsampling from core to sample jar

See ITRC ISM-1, Section 2.6.6.7

Lab subsampling to reduce sample volume for further processing

Photo courtesy of Mark Bruce, TestAmerica

From EPA 600/R-03/027

From Table 8. Subsampling methods that are NOT recommended based on experimental evaluation

Method	Typical Increment Size	Sensitivity to Grouping & Segregation	Moisture Content	Correct Sampling Possible	Agreement with Calculated s_{FE}^2	Comments
Degenerate Fractional Shoveling	Medium to Large	Moderate to High	Dry to Moist	Yes, if Careful	Unlikely	Performance Tied to Lot Mass; Subject to Bias; N.R.
Rolling and Quartering	Large	High	Dry	Yes, if Careful	Usually Not Close	Highly Variable; NR
Coning and Quartering	Large	High	Dry	Yes, if Careful	Usually Not Close	Usually Biased; NR
V-Blender	N.A.	High	Dry	N.A.	Very Unlikely	Problems with GE; N.R.
Vibratory Spatula	Small	Very High	Dry	No	Not Close	Problems with GE; NR
Grab Sampler	Variable	Very High	Dry to Moist	No	Not Close	Biased and Variable; N.R.

Any Questions?

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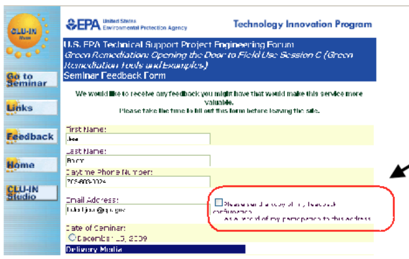
Resources & Feedback

- To view a complete list of resources for this seminar, please visit the **Additional Resources**
- Please complete the **Feedback Form** to help ensure events like this are offered in the future



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