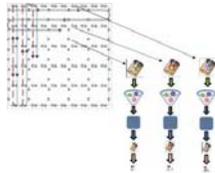


1

## Welcome – Thanks for joining this ITRC Training Class



### Soil Sampling and Decision Making Using Incremental Sampling Methodology (ISM) Part 2 – Implement, Assess, and Apply



Web-Based Document at:  
<http://www.itrcweb.org/ISM-1/>

### Incremental Sampling Methodology Technology Regulatory and Guidance Document (ISM-1, February 2012)

Sponsored by: Interstate Technology and Regulatory Council ([www.itrcweb.org](http://www.itrcweb.org))  
Hosted by: US EPA Clean Up Information Network ([www.cluin.org](http://www.cluin.org))

When sampling soil at potentially contaminated sites, the goal is collecting representative samples which will lead to quality decisions. Unfortunately traditional soil sampling methods don't always provide the accurate, reproducible, and defensible data needed. Incremental Sampling Methodology (ISM) can help with this soil sampling challenge. ISM is a structured composite sampling and processing protocol that reduces data variability and provides a reasonable estimate of a chemical's mean concentration for the volume of soil being sampled. The three key components of ISM are systematic planning, field sample collection, and laboratory processing and analysis. The adequacy of ISM sample support (sample mass) reduces sampling and laboratory errors, and the ISM strategy improves the reliability and defensibility of sampling data by reducing data variability.

ISM provides representative samples of specific soil volumes defined as Decision Units. An ISM replicate sample is established by collecting numerous increments of soil (typically 30 to 100 increments) that are combined, processed, and subsampled according to specific protocols. ISM is increasingly being used for sampling soils at hazardous waste sites and on suspected contaminated lands. Proponents have found that the coverage afforded by collecting many increments, together with disciplined processing and subsampling of the combined increments, yields consistent and reproducible results that in most instances have been preferable to the results obtained by more traditional (e.g. discrete) sampling approaches.

This 2-part training course along with ITRC's web-based [Incremental Sampling Methodology Technical and Regulatory Guidance Document \(ISM-1, 2012\)](http://www.itrcweb.org/ISM-1/) is intended to assist regulators and practitioners with the understanding the fundamental concepts of soil/contaminant heterogeneity, representative sampling, sampling/laboratory error and how ISM addresses these concepts. Through this training course you should learn:

- basic principles to improve soil sampling results
- systematic planning steps important to ISM
- how to determine ISM Decision Units (DU)
- the answers to common questions about ISM sampling design and data analysis
- methods to collect and analyze ISM soil samples
- the impact of laboratory processing on soil samples
- how to evaluate ISM data and make decisions

In addition this ISM training and guidance provides insight on when and how to apply ISM at a contaminated site, and will aid in developing or reviewing project documents incorporating ISM (e.g., work plans, sampling plans, reports). You will also be provided with links to additional resources related to ISM.

The intended users of this guidance and training course are state and federal regulators, project managers, and consultant personnel responsible for and/or directly involved in developing, identifying or applying soil and sediment sampling approaches and establishing sampling objectives and methods. In addition, data end users and decision makers will gain insight to the use and impacts of ISM for soil sampling for potentially contaminated sites.

**Recommended Reading:** We encourage participants to review the ITRC ISM document (<http://www.itrcweb.org/ISM-1/>) prior to participating in the training classes. If your time is limited in reviewing the document in advance, we suggest you prioritize your time by reading the Executive Summary, Chapter 4 "Statistical Sampling Designs for ISM," and Chapter 7 "Making Decisions Using ISM Data" to maximize your learning experience during the upcoming training classes.

ITRC (Interstate Technology and Regulatory Council) [www.itrcweb.org](http://www.itrcweb.org)

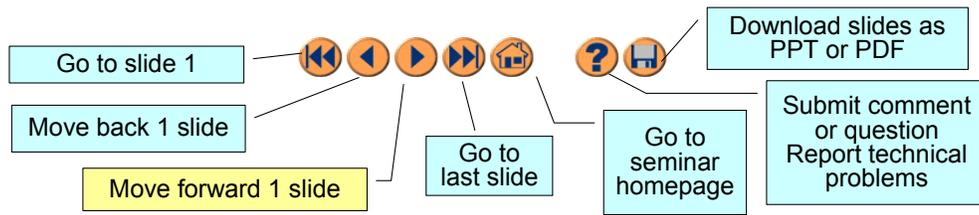
Training Co-Sponsored by: US EPA Technology Innovation and Field Services Division (TIFSD) ([www.clu-in.org](http://www.clu-in.org))

ITRC Training Program: [training@itrcweb.org](mailto:training@itrcweb.org); Phone: 402-201-2419

## Housekeeping



- ▶ Course time is 2¼ hours
- ▶ Question & Answer breaks
  - Phone - unmute \*6 to ask question out loud
  - Simulcast - ? icon at top to type in a question
- ▶ Turn off any pop-up blockers
- ▶ Move through slides
  - Arrow icons at top of screen
  - List of slides on left
- ▶ Feedback form available from last slide – **please** complete before leaving
- ▶ This event is being recorded



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

We have started the seminar with all phone lines muted to prevent background noise. Please keep your phone lines muted during the seminar to minimize disruption and background noise. During the question and answer break, press \*6 to unmute your lines to ask a question (note: \*6 to mute again). Also, please do NOT put this call on hold as this may bring unwanted background music over the lines and interrupt 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 using the ? icon. 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 1<sup>st</sup> 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 presentation overview, instructor bios, links to the slides and additional resources. Lastly, the button with a computer disc can be used to download and save today's presentation slides.

## ITRC ([www.itrcweb.org](http://www.itrcweb.org)) – Shaping the Future of Regulatory Acceptance



### ▶ Host organization



### ▶ Network

- State regulators
  - All 50 states, PR, DC
- Federal partners



- ITRC Industry Affiliates Program



- Academia
- Community stakeholders

### ▶ Disclaimer

- Full version in “Notes” section
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### ▶ Available from [www.itrcweb.org](http://www.itrcweb.org)

- Technical and regulatory guidance documents
- Internet-based and classroom training schedule
- More...

The Interstate Technology and Regulatory Council (ITRC) is a state-led coalition of regulators, industry experts, citizen stakeholders, academia and federal partners that work to achieve regulatory acceptance of environmental technologies and innovative approaches. ITRC consists of all 50 states (and Puerto Rico and the District of Columbia) that work to break down barriers and reduce compliance costs, making it easier to use new technologies and helping states maximize resources. ITRC brings together a diverse mix of environmental experts and stakeholders from both the public and private sectors to broaden and deepen technical knowledge and advance the regulatory acceptance of environmental technologies. Together, we’re building the environmental community’s ability to expedite quality decision making while protecting human health and the environment. With our network of organizations and individuals throughout the environmental community, ITRC is a unique catalyst for dialogue between regulators and the regulated community.

For a state to be a member of ITRC their environmental agency must designate a State Point of Contact. To find out who your State POC is check out the “contacts” section at [www.itrcweb.org](http://www.itrcweb.org). Also, click on “membership” to learn how you can become a member of an ITRC Technical Team.

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The information provided in documents, training curricula, and other print or electronic materials created by the Interstate Technology and Regulatory Council (“ITRC” and such materials are referred to as “ITRC Materials”) is intended as a general reference to help regulators and others develop a consistent approach to their evaluation, regulatory approval

## Meet the ITRC Trainers



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**Michael Stroh** is a senior project manager with the Missouri Department of Natural Resources in Jefferson City, Missouri. He joined the department in 1997 to oversee site assessment and cleanup activities in the state's Voluntary Cleanup Program before joining the state's Superfund Site Assessment group in 2002. Before joining MDNR, Michael worked as an analytical chemist and section supervisor for environmental laboratories in the private sector. He has also worked as a chemist for the US Geological Survey at a mobile laboratory on the Chesapeake Bay, and as a bihydrologist for The Nature Conservancy in Arlington, Virginia. Michael joined the ITRC Incremental Sampling Methodology (ISM) team at its inception in 2009 and has served as a chapter lead in drafting the technical and regulatory guidance document. Michael earned a bachelor's degree in biology from Truman State University in Kirksville, Missouri in 1985 and a master's degree in environmental science from the Indiana University School of Public and Environmental Affairs in Bloomington, Indiana in 1994.

**Jay Clausen** is a Physical Research Scientist with the US Army Corps of Engineers, Engineer Research and Development Center, Cold Regions Research and Engineering Laboratory (CRREL) in Hanover, New Hampshire. Since 2005, Jay has conducted research on the application of the Incremental Sampling Methodology (ISM) to military ranges containing energetics and metals in particulate form as well as the fate-and-transport of lead, tungsten, and energetics. Jay routinely presents at conferences and publishes peer-reviewed journal articles, and government reports. Prior to joining CRREL, Jay worked for 14 years in the environmental consulting field for AMEC as a Senior Hydrogeologist/Project Manager and Lockheed Martin focused on Department of Defense and Department of Energy issues. Jay earned a bachelor's degree in geology from the University of Nebraska in Omaha in 1987 and a master's degree in the geosciences from the University of Maine in Orono in 1990. Jay is currently working on PhD in the Natural Resources and Earth System Science program at the University of New Hampshire focused on the application of ISM techniques to soils containing metals. Jay is a certified professional geologist in the states of Kentucky, New Hampshire, Texas, and Washington and a certified professional hydrogeologist in the state of Washington.

**Mark Bruce** is a Corporate Technical Director for TestAmerica. He has experience in environmental monitoring of metals, wet chemistry, volatile and semivolatile organics including sample preparation and analysis since 1979. He has participated in the development of several EPA methods. His responsibilities include method development, evaluation, implementation and troubleshooting. He provides technical support to TestAmerica clients and internal staff. He has worked with incremental sample processing techniques since 2003. He has been active on the ITRC Incremental Sampling Methodology team since it started in 2009 and particularly involved with the development of the laboratory guidance. Mark earned a Ph.D. in Analytical Chemistry from the University of Cincinnati in 1984.

**Tim Frederick** is a human health risk assessor with EPA Region 4 in Atlanta, GA. He provides risk assessment, toxicology, and other technical support to EPA's project managers on a wide variety of sites in the Southeast. Tim has worked in the Region's Superfund Division since 2005 and serves on several national workgroups. Prior to joining EPA, Tim provided risk assessment and toxicology technical support to EPA as a contractor from 1996 to 2005, and he worked as an environmental consultant in Miami, Florida from 1990 to 1996. Tim has presented nationally at technical conferences, and he has provided guest lectures at local universities. Tim has been a member of the ITRC's Incremental Sampling Methodology team since it began in 2009. Tim earned a bachelor's degree in psychology from the University of Miami in Miami, Florida in 1989 and a master's of public health in environmental and occupational health from Emory University's Rollins School of Public Health in Atlanta, Georgia in 1996.

## Soil Sampling Data – What Do We All Need?

- ▶ Accuracy
- ▶ Reproducibility
- ▶ Defensibility



....but how do we get it?

**Incremental Sampling Methodology (ISM)**

.....may be your answer.....

Picture Reference: <http://www.swrcb.ca.gov/rwqcb2/brownfields.shtml>

Set stage – context with Day 1

## ISM Goal – A Representative Sample!



The primary objective of ISM is to obtain a representative sample having constituents in exactly the same proportions as the entire decision unit.



Representative subsampling



### Speaker Bullets

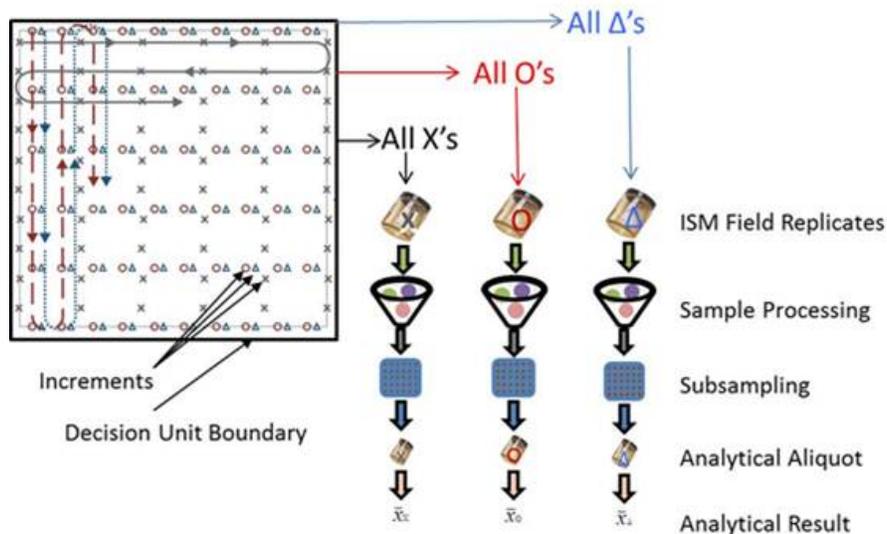
- Cannot analyze the whole decision unit, i.e. put the whole unit in a jar
- Sampling Error should be limited/managed to obtain sample representative of the field/DU
  - a representative subsample of the fraction of interest determined during systematic planning is collected in the laboratory and will be presented in the next module
- Sampling Error is managed with ISM through the collection of 30 or more increments of correct shape and adequate mass
- Field implementation and sampling activities should be performed by ISM trained personnel

### Supplemental Information

Section 5.1 Introduction

Picture Reference: <http://www.swrcb.ca.gov/rwqcb2/brownfields.shtml>

## Incremental Sampling Methodology (ISM) Process



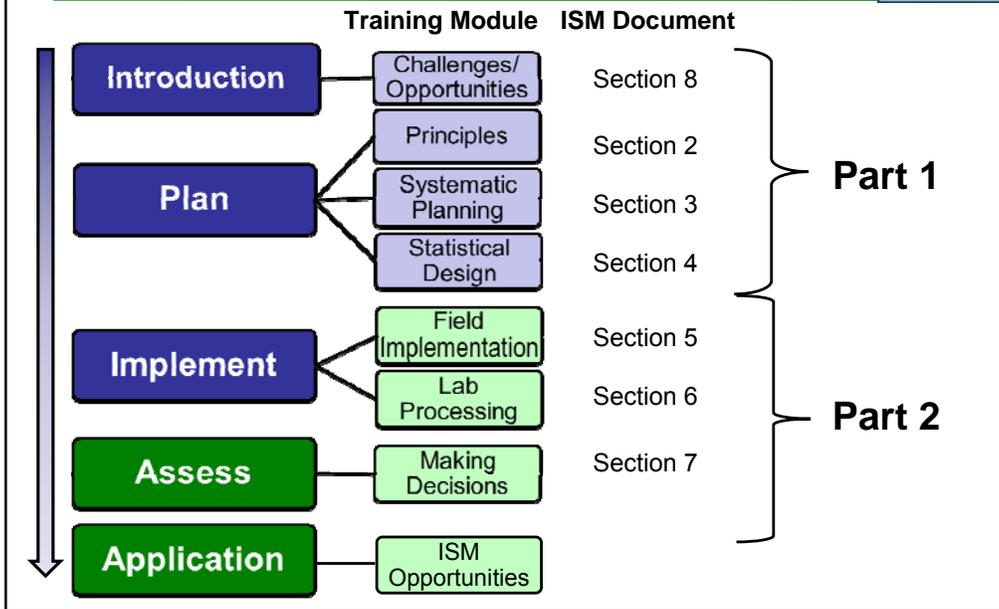
### WHAT IS ISM?

This slide shows quite succinctly all the steps involved in developing triplicate analytical results for a particular sampling unit. Triplicate results are what we are recommending for a number of reasons – largely related to addressing the heterogeneity we talked about earlier, and being able to make some statistical estimate of variability. Triplicates will allow a calculation of the 95% UCL that many of you – particularly risk assessors - are interested in.

Remember –

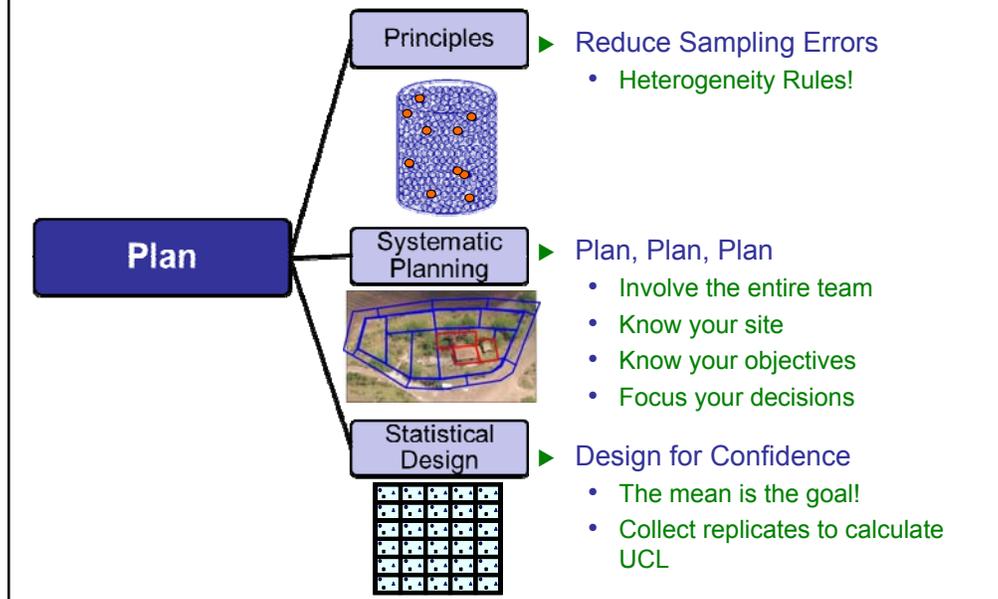
- minimum of 30 increments (20 – 60g/increment)
- 3 replicates in order to get a 95 UCL
- Sample size should be ~1 kg (600 – 2,500 grams).

# ISM Document and Training Roadmap



No associated notes.

## ISM Part 1 – Summary Principles, Systematic Planning, and Statistical Design



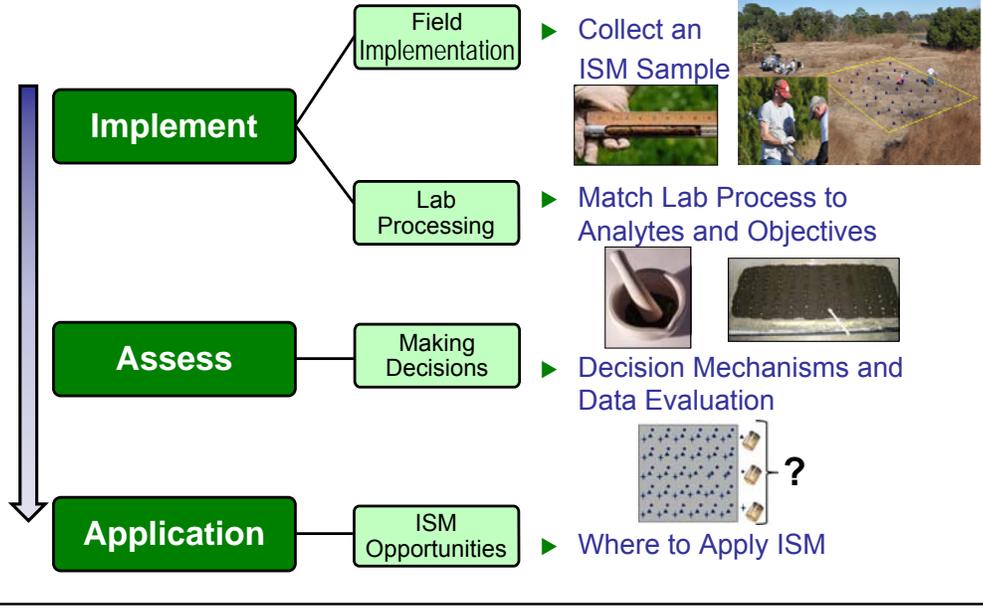
The last training included Modules on:

Soil and Principles – Be aware of issues related to heterogeneity and sampling errors

Systematic Planning - Involve the entire team, regulators, consultants, responsible parties in critical elements (e.g. conceptual site model, establish sampling objectives and decision units.) Sampling objectives should drive your sampling design, and the scale of decision making should align with sampling objectives.

Statistical Design - Provides the statistical foundation and describes why ISM provides a reasonable mean, describes a good ISM sampling design, and informs you how ISM provides 95% UCL.

# ISM Part 2 Preview Implement, Assess, and Apply



No associated notes.

## Field Implementation Learning Objectives

### Learn how to:

#### ► Collect an ISM sample

- Understand the similarities and differences between surface and subsurface ISM sampling
- Consider issues specific to non-volatile and volatile ISM sampling
- Implement and collect ISM replicate samples



ITRC, ISM-1, Section 5

### Speaker Bullets

With correct up front planning and coordination, ISM field implementation can be as simple as putting dirt in a bucket (or bag).

### Supplemental Information

ITRC, ISM-1, Section 5

## Key Presentation Topics

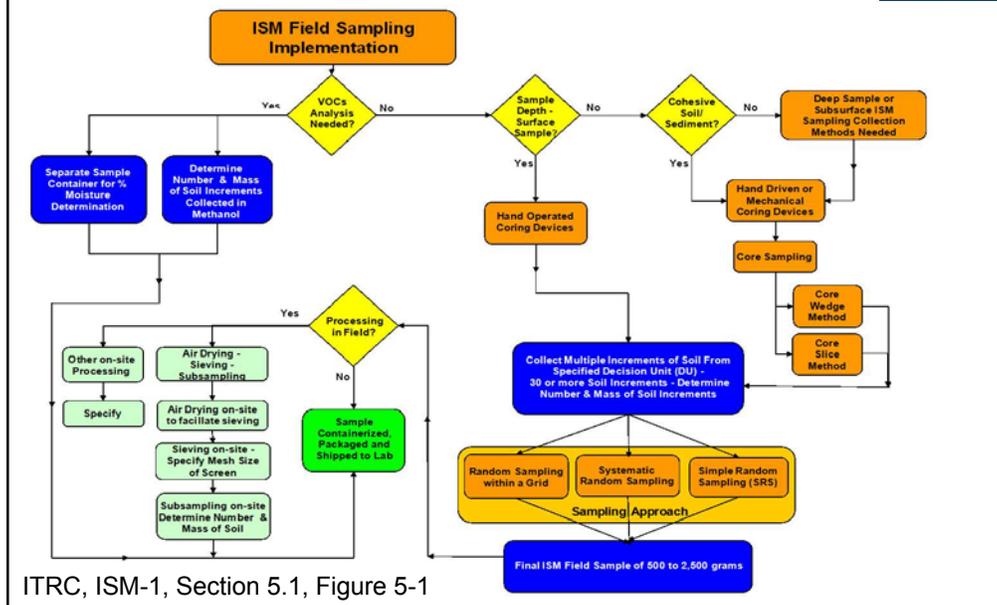
- ▶ Sampling design
- ▶ Sampling tools
- ▶ ISM surface/subsurface sampling
  - Cores and subsampling
- ▶ Specific contaminant of concern (COC) considerations
  - Non-volatile and volatile
- ▶ ISM replicates



### Speaker Bullets

- Brief review of sampling designs that were presented as part of Day 1, Section 4

# ISM Field Sampling Implementation



## Speaker Bullets

- Figure 5-1 from the technical regulatory document shown as an example only. The audience is not expected to read this flowchart (small font); provided only as an example to illustrate the multiple decisions that will need to be made and implemented.
- Multiple step process that all should be discussed/planned for during systematic planning
- Technical regulatory document will assist with these steps

## Supplemental Information

ITRC, ISM-1, Section 5.1, Figure 5-1

## Sample Collection Components

- ▶ Decision Unit (DU) sampling design
  - Simple random sampling
  - Random sampling within a grid
  - Systematic random sampling
- ▶ Sampling tools
  - Core shaped
  - Adequate diameter
- ▶ Mass
  - Increment mass
  - Sample mass



ITRC, ISM-1, Section 4.3.4.2

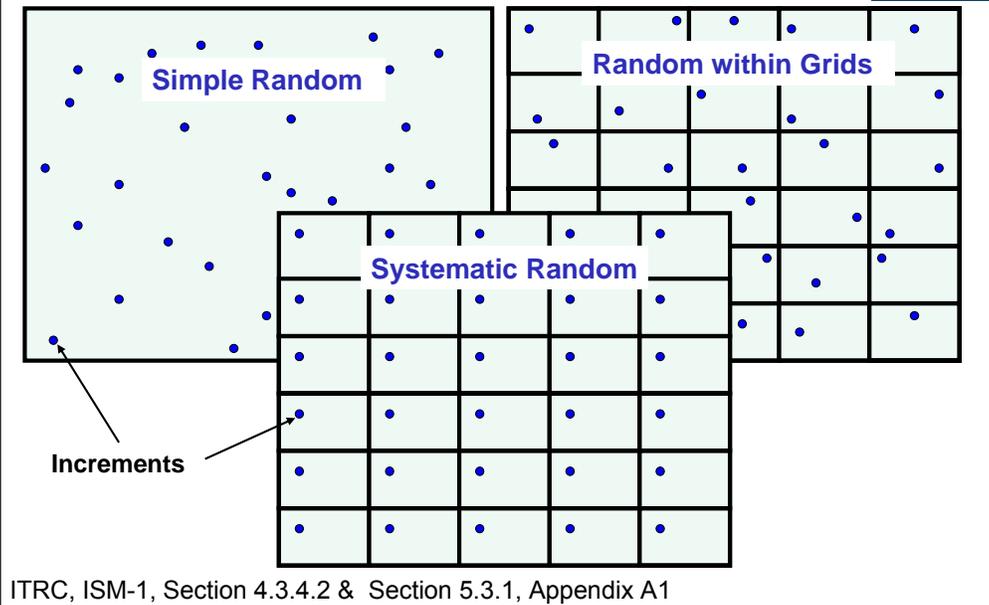
### Speaker Bullets

- Sampling Design - 3 basic sampling designs
- The appropriate sample support for tools to address soil sampling theory to limit error
- Adequate mass collected to limit error

### Supplemental Information

Section 4.3.4.2 Effects of sampling pattern

## Sampling Designs



### Speaker Bullets

- As a brief review, just as with discrete sampling, a variety of sampling methods may be implemented with ISM sampling.
  - The advantages/disadvantages were presented as part of the Section 4 of the Day 1 training. Additional information is found in Chapter 4 of the ISM document.
  - Simple Random Sampling, Systematic Random Sampling, and Systematic Grid Sampling all yield unbiased estimates of the mean. All are appropriate for ISM sampling, choice would be project specific
  - “unbiased” meaning every soil particle has an equal chance of being collected/analyzed
  - Note that “random” in this context does not mean wherever the sampling team feels like taking a sample, and that a formal approach to determining the random increment locations must be used
  - These are 2 dimensional examples, however, recall that DU’s have a depth/3 dimensional component
  - Recommended minimum 30 increments per DU regardless of sampling design
- 
- The systematic random sampling is generally more efficient to implement in the field; only one (initial) random location is determined (3 initial random locations for replicates); and
  - It ensures relatively even spatial distribution of increments across the DU, therefore avoids the appearance of over- or under-representation of sub-areas within a DU, (as may occur with SRS - Simple Random Sampling); reduce/control the size of any potential unsampled area (“hotspot”) to be detected/not detected.

### Supplemental Information

ITRC, ISM-1, Section 4.3.4.2 & Section 5.3.1, Appendix A1

## Florida Case Study: Decision Unit (DU) Identification



### ► Identify DU in the field

- Use typical environmental site investigation procedures
- Examples
  - Survey
  - GPS
  - Swing ties



ITRC, ISM-1, Section 9.3 & Appendix C, Section C.3

### Speaker Bullets

- DU(s) determined during systematic planning
- Standard environmental site investigation procedures employed to delineate DU in the field
- The photograph is from the Florida case study that was introduced as you recall during the ISM Day 1 training. Former golf course with arsenic as the contaminant of concern. The DU is a ¼ acre residential lot – assumed exposure area of potential future residential development.
- Chapter 9, Section 9.3 and Appendix C, Section C.3 for Former Golf Course Field Demonstration of ISM

### Supplemental Information

ITRC, ISM-1, Section 9.3 & Appendix C, Section C.3 for Former Golf Course Field Demonstration of ISM

## Increment Locations

- ▶ Identify increment locations in field
  - Utilize similar site investigation tools



ITRC, ISM-1, Section 5.3.1

### Speaker Bullets

- Sampling design, number of increments, increment locations are determined during systematic planning (Day 1 training)
- Typically 30 or more increments per DU
- Similar tools are utilized to identify increment locations; e.g. survey grid, GPS, sampling flags
- Example of transformer pad and residential backyard DUs
- Increment locations are flagged in these examples. May assist inexperienced ISM practitioners, more experienced ISM samplers it may not be necessary.

### Supplemental Information

ITRC, ISM-1, Section 5.3.1

## Florida Case Study: Increment Field Determination



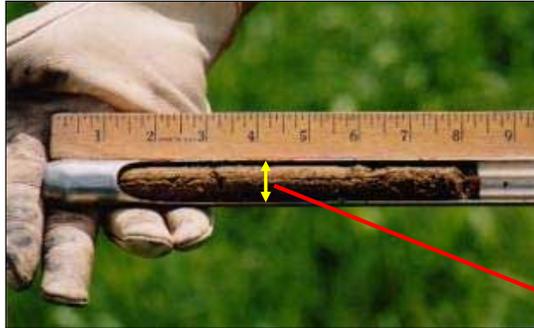
### Speaker Bullets

- It may or may not be necessary to determine/document the “exact” location of each increments, depending on criteria and DQOs specified during systematic planning. Check with regulatory authority.
- Generally not necessary, however regulations or policy, public perception, education, etc. may require.
- For the Florida Case study, specific increment locations were not marked/flagged. The locations (relative to the gridpoints, e.g., 2 ft east and 3.5 ft south) were recorded in the SAP (Sampling and Analysis Plan) that was prepared prior to the study. A tape measure was used to measure the first few locations exactly, the distance was converted to “heel-to-toe steps”, and then this method was used for all of the remaining increments.

## Sampling Tool Considerations

### ► Criteria - shape

- Cylindrical or core shaped increments
- Minimum diameter required – based on particle size (soil fraction) of interest



e.g., core diameter  
>16 mm

ITRC, ISM-1, Section 5.2

### Speaker Bullets

- Core increments should be collected
- Minimum diameter of core determined per fraction of interest; e.g. generally soil is considered <2mm fraction, minimum core diameter of 16 mm. See section 5.2 of ISM document for additional information on calculation/recommended core diameter.
- Larger fraction of interest (or particle size) requires a larger diameter core sampler
- Complete core increments should be collected across the depth of the DU (cohesive/noncohesive)
- Commercially available tools (next slides)

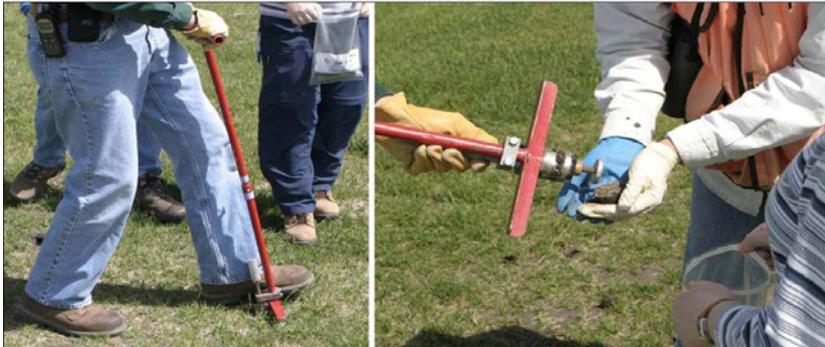
### Supplemental Information

ITRC, ISM-1, Section 5.2

## Additional Considerations



- ▶ Decontamination
  - Not necessary within DU (including replicates)
- ▶ Sampling tool
  - Appropriate for matrix and contaminant of interest



ITRC, ISM-1, Section 5.2

### Speaker Bullets

- Tools can be used without decontamination between increments within DU (including replicates); all increments are part of the same sample, cross contamination is not an issue.
- Collected directly into appropriate sample container (Ziploc lower right) not hands, only for example purposes.
- Example – Multi-Incremental Sampling Tool (MIST™)
- Other commercially available tools (next slides)

### Supplemental Information

ITRC, ISM-1, Section 5.2

## Sampling Tool Examples

### Soft Surface Soil



Source: Courtesy <http://www.jmcsoil.com/index.html>  
<http://fieldenvironmental.com/evc-incremental-sampler.php>

### Speaker Bullets

- Examples of commercially available tools.
- Other tools, e.g. agricultural soil samplers, may also be applicable to ISM.
- Top: EVC Incremental Sampler (kit); Bottom: JMC Backsaver Handle and Soil Tubes

## Alternate Sampling Tools



ITRC, ISM-1, Section 5.2; Figure 5-2b

### Speaker Bullets

- Alternate tools may be applicable depending on matrix

### Supplemental Information

ITRC, ISM-1, Section 5.2; Figure 5-2b

## Florida Case Study: Field Sampling



### Speaker Bullets

- Florida Golf Course Example – Every job needs at least one supervisor.
- Surface core increments collected in plastic 5 gallon bucket

## Florida Case Study: “Low Tech” Sampling Tools



### Speaker Bullets

- Low tech alternatives, if site conditions amenable
- Arsenic only COC; PVC would not interfere/contaminate analysis of COC (phthalates)
- Left pounding PVC section into ground; right PVC increments from DU, all increments then combined into 1 ISM sample

## Adequate Sample Mass

- ▶ Criteria – mass (non-volatile)
  - Recommended mass per increment: 20-60 grams
  - Final ISM samples: generally 600-2,500 grams

$$M_s = \rho \cdot n \cdot D_s \cdot \pi \cdot (q / 2)^2$$

$M_s$  – targeted mass of sample (g)

$D_s$  – increment length (cm)

$n$  – number of increments

$\rho$  - soil or sediment density (g/cm<sup>3</sup>)

$q$  - diameter of sample core (cm)



ITRC, ISM-1, Section 5.3.1

### Speaker Bullets

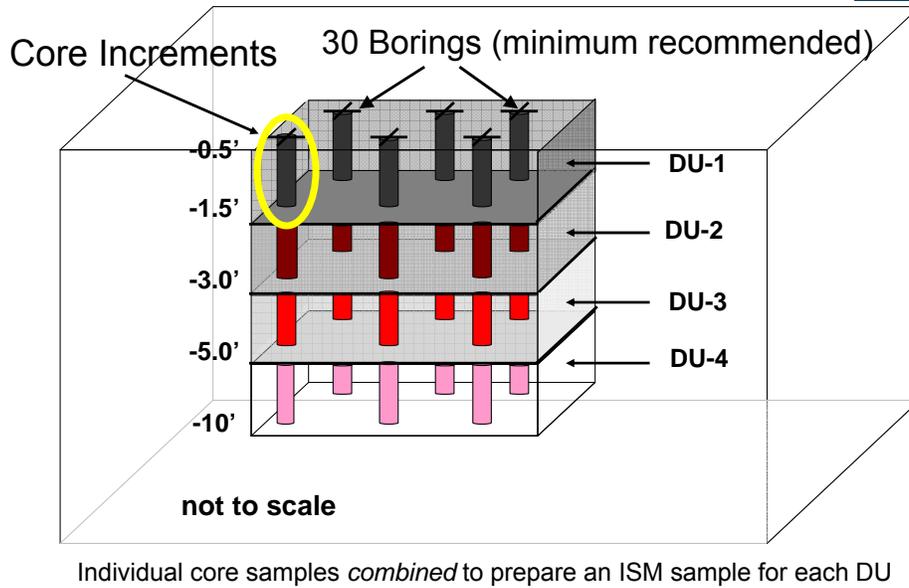
- All parameters should be determined during systematic planning
- Generally, a minimum of 30 increments should be collected for each DU

### Supplemental Information

ITRC, ISM-1, Section 5.3.1

Example of bulk ISM sample ~2 kg

## Subsurface Decision Units (DU)



### Speaker Bullets

- Same DU determination (3 dimensional volume), sample design, number of increments, etc. as surface sampling
- Subsurface DUs presented in Module 3
- DUs designated at depth to provide vertical resolution of contamination within DU and optimize remediation.

## Subsurface Sampling Considerations



- ▶ Preferred increment – entire core interval
- ▶ Core subsampling alternatives
  1. Core wedge
  2. Core slice



ITRC, ISM-1, Section 5.3.2

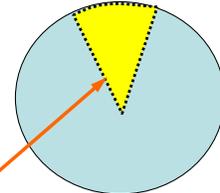
### Speaker Bullets

- Entire core increment preferred; logistically subsampling the cores may be necessary.
- Cores may be subsampled in field and combined to prepare ISM sample for designated DU layer

### Supplemental Information

ITRC, ISM-1, Section 5.3.2

## Core Wedge



e.g., wedge width  
>16 mm

**Continuous wedge removed from entire length of targeted DU interval for 100% coverage**

ITRC, ISM-1, Section 5.3.2.1

### Speaker Bullets

- “Wedge” as pictured is taken across entire core interval/DU depth
- The simplest approach is to split the core in half, vertically along the axis, reducing the increment mass by half. Alternatively, a single wedge of soil is taken from the entire length of the targeted depth interval.
- Wedge width requirement per fraction of interest (sample support) the same as core diameter (e.g. 16 mm for 2mm soil); larger fraction of interest requires wider wedge.

### Supplemental Information

ITRC, ISM-1, Section 5.3.2.1

## Core Slice



**Core Slice removed from randomly selected interval length of targeted DU depth**

ITRC, ISM-1, Section 5.3.2.1

### Speaker Bullets

- Randomly selected core “slice” collected from core interval as increment
- **Least preferred subsampling method**. Increased bias over previous options due to incomplete depth interval collection/representation

### Supplemental Information

ITRC, ISM-1, Section 5.3.2.1

## Field Processing for Non-Volatiles



- ▶ ISM sample processing in a controlled laboratory environment is recommended to reduce error
- ▶ Field processing may be applicable if project specific DQOs can be met



ITRC, ISM-1, Section 5.4.1

### Speaker Bullets

- Each processing step introduces some amount of sampling error; sample processing in a controlled laboratory environment recommended to reduce this error
- Generally follows same process as laboratory, i.e. sieving, spread evenly in thin layer, collect multiple systematic random increments to form subsample ( i.e., 2 dimensional (2D) Japanese slabcake subsampling)
- Subsampling not recommended for particulate contaminants (energetics, metals), entire sieved fraction should be ground and subsampled
- **Field subsampling triplicate samples recommended to evaluate error**

### Supplemental Information

ITRC, ISM-1, Section 5.4.1

## Florida Case Study: Non-Volatile ISM Sample Logistics



- ▶ Final ISM samples: typically 600-2,500 grams or more
  - Containers, storage, shipping
- ▶ Laboratory
  - Facilities and equipment for correct processing and subsampling



### Speaker Bullets

- All logistical considerations should be determined during site-specific systematic planning
- Large samples, alternate containers, temperature and preservation considerations, etc.
  - Samples were transferred from 5-gallon buckets to Ziploc bags, however, larger sample support may require storage and shipment of larger (5 gallon) samples to the laboratory.
- Laboratory coordination and communication essential

## ISM Volatile Sampling Tools



- ▶ Core type sampler
- ▶ Typical for VOC soil sampling per SW846 5035A



ITRC, ISM-1, Section 5.4.2

Source: Courtesy [www.ennovativetech.com](http://www.ennovativetech.com)

### Speaker Bullets

- Core increments on smaller (typical 5 gram) scale
- Standard VOC collection for high level, field preserved soil samples; SW846 Method 5035A, Section 8.2.2. Difference is multiple core increments placed into single adjusted volume of methanol

### Supplemental Information

ITRC, ISM-1, Section 5.4.2

Left) Core N' One; Center) Terra Core Sampler; Right) Easy Draw Syringe & PowerStop Handle

## ISM Volatile Samples – Subsurface

- ▶ Numerous increments collected across core/depth interval



### Speaker Bullets

- Core, wedge, slice increments impractical for VOCs
- Representative VOC sampling of subsurface core
- Potential VOC loss; increments should be collected and preserved as quickly as possible from opened core
- Photo on right is an example of a core with VOC plugs removed at ~2 inch intervals

## ISM Volatile Sample Logistics



### ► VOC preservation and analysis

- Increments are extruded from sampler directly into volume of appropriate container with predetermined methanol
- Methanol preserved sample submitted to laboratory
- Note shipping restrictions/requirements



ITRC, ISM-1, Section 5.4.2, Figure 5-11

### Speaker Bullets

- Close laboratory coordination required for the appropriate sample bottles and methanol volume
- Shipping logistics/restrictions may apply; Guidelines for the transportation of a solvent such as Methanol can be found in Department of Transportation (DOT), 49 Code of Federal Regulations (CFR), Part 172, List of Hazardous Substances (ground) and Reportable Quantities. Shipments via air transport may also be required to adhere to International Air Transport Association Dangerous Goods Regulations (IATA DGR) (air).

### Supplemental Information

ITRC, ISM-1, Section 5.4.2, Figure 5-11

See section 5.4.2 for additional information and options for methanol

## Replicates Recommended



- ▶ Increments collected from alternate random locations
  - Independent samples, not “splits”
- ▶ Minimum 3 replicate set for statistical evaluations
- ▶ Additional replicates may be necessary depending on contaminant heterogeneity and project specific DQOs

ITRC, ISM-1, Section 5.3.5

### Speaker Bullets

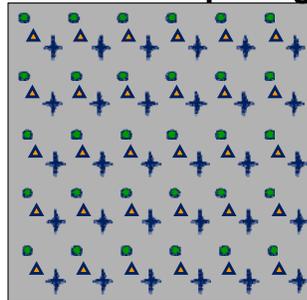
- Recommended to evaluate spatial heterogeneity and ensure reliable estimate of the mean
- Required for any statistical evaluation, e.g. 95% UCL of the mean
  - Refer to Day 1 training presentation of ISM replicates, estimation of mean and coverage
  - Refer to Chapter 4 of the ISM document for the statistical basis/evaluation of ISM replicates

### Supplemental Information

ITRC, ISM-1, Section 5.3.5

## Replicate Spacing and Collection

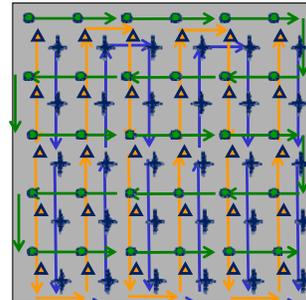
### Replicate Increment Spacing



### Decision Unit

R1  R2  R3 

### Sample Collection



### Decision Unit

Replicate 1 

Replicate 2 

Replicate 3 

ITRC, ISM-1, Section 5.3.5

### Speaker Bullets

- Field replicate example for systematic random sampling design; different random starting location, “serpentine pattern” if collected in succession
- The collection order (of increments/replicates) does not matter; what is most efficient in the field
- Submitted blind to laboratory

### Supplemental Information

ITRC, ISM-1, Section 5.3.5

## Field Replicates – Simple Example



### Speaker Bullets

- ISM replicate locations; stockpile sampling with grid demarcated with string

## Replicate/Sampling Reminders



- ▶ Replicates
  - What type
  - How many
  - Where/when will they be collected
  - How will they be evaluated
  
- ▶ “Homogenizing” or mixing not necessary
  - Laboratory processing and subsampling (following module) designed to attain representative analytical sample

### Speaker Bullets

- What type – DU and/or field subsampling replicates
- How many – 3 or more depending on DQOs
- Where – replicate increment location
- When – simultaneously within grid or consecutively

## Field Implementation Summary



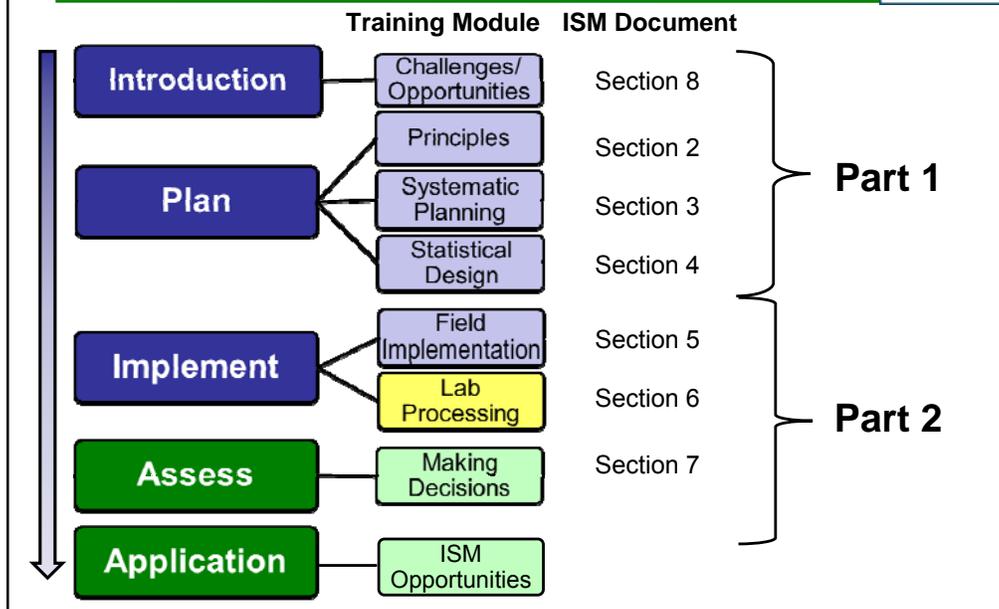
- ▶ Determined during Systematic Planning
  - Sampling design
  - Adequate sampling tools
  - ISM surface/subsurface sampling logistics
    - Subsurface cores and subsampling
  - Specific contaminant of concern (COC) considerations
    - Non-volatile and volatile
  - ISM replicates



### Speaker Bullets

- All items should be determined during systematic planning and implemented in the field to obtain representative samples to submit to the laboratory for additional processing and/or analysis.
- That concludes this module.

## ISM Document and Training Roadmap



### Speaker bullets

- Focus – Match lab process
- To analytes
- And end data use

### Narrative

Thank you Earl (or Jay). We have now learned how important planning is. Followed by diligent work to collect representative samples in the field. Now we need to maintain that level of effort at the laboratory. Next we are going to discuss how to match the right lab processes to the analytes of interest and the end uses of the data.

## Laboratory Processing Learning Objectives



### Learn how to:

- ▶ Match process options to analytes and data objectives
- ▶ Manage sample moisture
- ▶ Select/reduce particle size
- ▶ Collect subsamples for analysis
- ▶ Apply Quality Assurance
- ▶ Examine options for lab certification

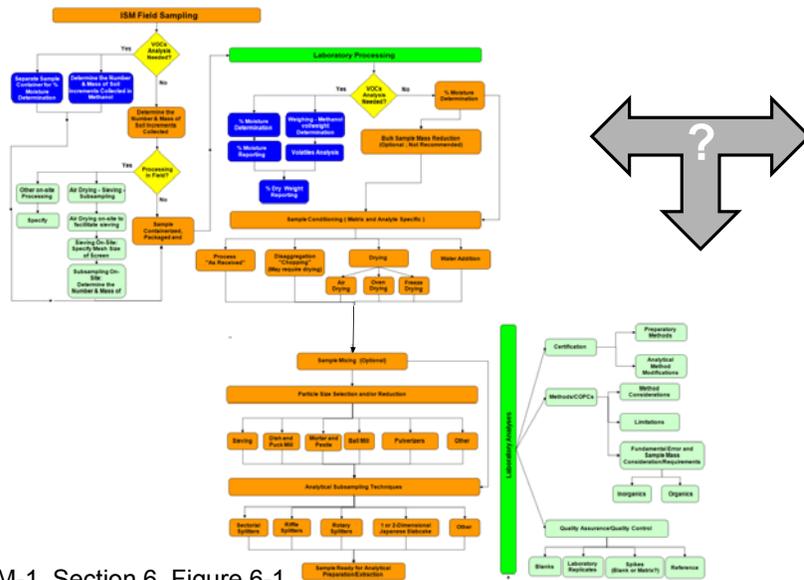
### Speaker bullets

- Focus – manage sample moisture, particle size selection and reduction and subsampling
- Ripple effects – digestion and extraction subsamples
- QC adaptations

### Narrative

We will focus on matching the processing options to the analytes and data needs. Laboratory support for ISM primarily focuses on the preliminary soil moisture management, particle size selection and particle size reduction and analytical subsampling. There are some ripple effects for sample digestion and extraction. Quality assurance processes should also be adapted to cover these new sample handling steps. Last we will examine the options for lab certification.

# Real Life ISM has Choices



ITRC, ISM-1, Section 6, Figure 6-1

**Speaker bullets**

- Many process choices

**Narrative**

Real life ISM processing has a variety of options. Sample processing decisions should be made upfront during project planning. We will be discussing those options and how to choose amongst them.

There is no universal process that is optimal for all analytes and matrices. Although the general dry, grind, subsample and analyze is the most common it is not appropriate in all cases. There are other options.

## Analyte-Matrix Driven Options



- ▶ Pick the right option
  - More representative subsamples
  - Better precision
  
- ▶ Pick the wrong option
  - Poor and unknown bias

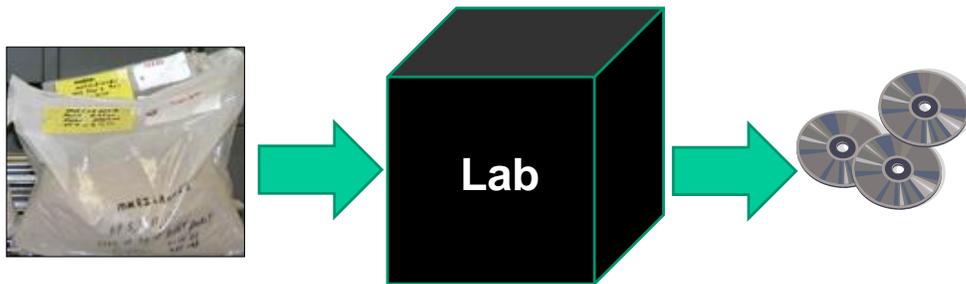
### Speaker bullets

- Pick the option based on analyte / matrix
- Pick right – representative subsample / results / better decisions
- Pick wrong – poor & unknown bias / risk wrong decision

### Narrative

The specific combination of analytes and matrix will determine which processing options are the best to meet the project specific data quality objectives. Pick the right option and the result is more representative subsamples, leading to more representative results with better precision to facilitate better decisions. Pick the wrong option and you end up with poor and unknown bias in the final results, potentially leading to the wrong decision.

## Include Lab Processing in Project Planning



ITRC, ISM-1, Section 6.1.1

### Speaker bullets

- Project planning, include lab processing

### Narrative

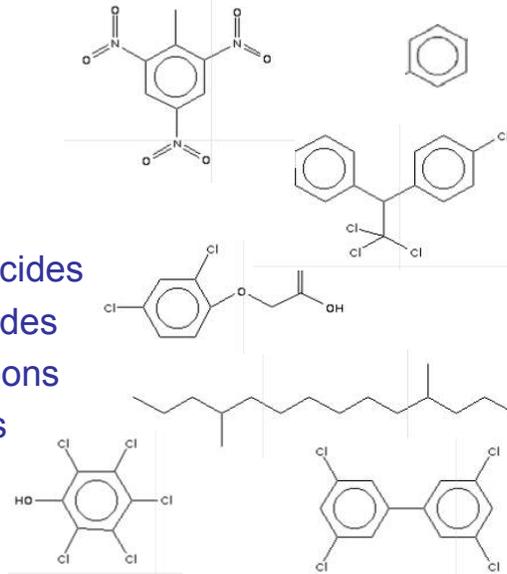
Include the lab processing options in the initial project planning discussions. Please don't assume that just feeding the lab dirt samples will automatically produce good data.

### Supplemental information

See Section 6.1.1

## Define the Analytes

- ▶ Volatile organics
- ▶ Energetics
- ▶ Metals, Hg
- ▶ PCBs
- ▶ Organochlorine pesticides
- ▶ Phenoxy acid herbicides
- ▶ Petroleum hydrocarbons
- ▶ Semivolatile organics
- ▶ Other



### Speaker bullets

- Determine analyte groups
- List individual analytes
- Energetics such as explosives and propellants
- Specific analytes >> optimal process

### Narrative

All analyte groups as well as the individual analytes within each group must be clearly defined and listed. The optimal sample handling options depend on the specific analytes. These differences will be highlighted in the discussion that follows.

## Coordinate VOC Sampling & Analysis



- ▶ Use methanol preservation
  - Methanol transport
  - Bottle sizes (large, medium, small)
- ▶ Analytical sensitivity limitations
  - Higher reporting limits
  - Selected Ion Monitoring GC-MS
    - Short analyte lists



ITRC, ISM-1, Section 6.2.1

### Speaker bullets

- 30 mL transport
- Bottle sizes
- Higher reporting limits
- SIM GC-MS for short lists

### Narrative

Careful coordination between lab and field crew is usually needed because the lab supplies custom methanol preserved bottles designed for the number of increments in each specific decision unit. Recall that Department of Transportation regulations restrict shipment of containers with more than 30 mL of methanol.

Use of the methanol extraction solvent facilitates combining many increments into a single analysis, but does limit the theoretical analytical sensitivity when compared to in-vial purge methods using aqueous solutions. Some data users consider the methanol extraction to be more efficient at recovering VOCs even though the calculated reporting limits tend to be higher.

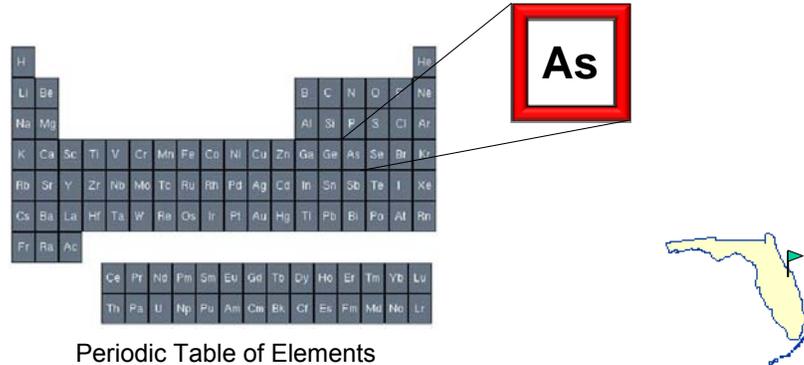
Selected ion monitoring GC-MS analysis can produce lower reporting limits when working with short analyte lists.

### Supplemental information

Section 6.2.1

## Florida Case Study: Contaminant of Concern

- ▶ Arsenic
  - From liquid applied pesticides



### Speaker bullets

- Arsenic
- Liquid pesticide

### Narrative

Arsenic was the contaminant of concern for the Florida golf course study. It had been applied in liquid form as a pesticide.

## Symbol Key



 ▶ Good effect

 ▶ Bad effect

 ▶ Result or statistic gets larger in value

 ▶ Result or statistic gets smaller in value

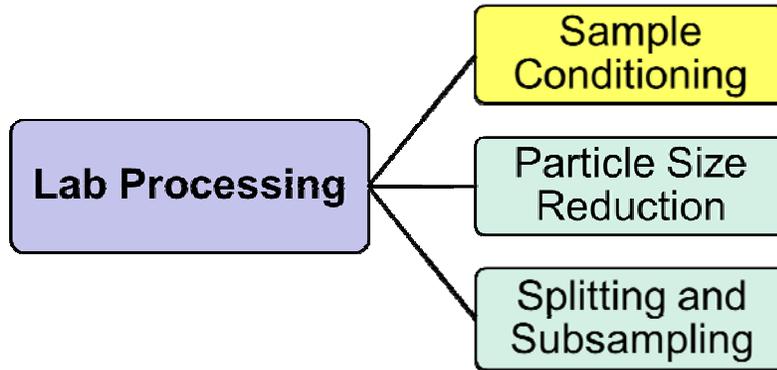
### Speaker bullets

- Coded arrows
- Solid green - good
- Striped red – bad
- Upward pointing - larger value
- Downward pointing - smaller value

### Narrative

Through the remainder of the lab processing section you will see several coded arrows. Solid green arrows mean the effect on the analytical results is good. Striped red arrows means the effect is bad. An upward pointing arrow means that the numerical statistic or result is larger. A downward pointing arrow means that the numerical statistic or result is smaller. The size of the arrow represents the magnitude of the change.

# Lab Processing Roadmap



No associated notes.

## Condition the Sample

### ► Air drying

- Room temperature – most common
- Ventilation hood
- Goal: Crushable agglomerates



### • Consider volatilization losses

- Boiling point
- Binding to soil particles
- Potential for Loss Table



– Naphthalene



– Acenaphthene



– Benzo[a]pyrene



### ► Use other options when drying not appropriate

ITRC, ISM-1, Section 6.2.2.3

### Speaker bullets

- Air dry
- Ventilation
- Low boiling – volatilization losses
- Binding to particles
- Loss risk table
- **Other conditioning options available when air drying not appropriate**

### Narrative

The most common sample conditioning step is air drying, usually at room temperature in a ventilation hood. The goal is to produce dry crushable dirt clods that work well with the mechanical processes that can follow. Air drying can produce low recovery of analytes with lower boiling points that are not strongly bound to the soil particles. The guidance has a loss risk table that summarizes the relative risk of losing various semivolatile organics and energetics during air drying. For example, the risk of losing naphthalene is large, acenaphthene is moderate and benzo[a]pyrene is small. Diesel and mercury have a wide variety of components or species with a range of volatilities. If lower boiling analytes are of primary interest, it might be necessary to avoid air drying.

When working with surface soils that have been exposed to air for many months or years after the potential contaminant release, the likelihood of volatilizing additional contaminants is small.

### Supplemental information

See Section 6.2.2.3

## Florida Case Study: Air Drying Samples



- ▶ Arsenic
  - High boiling arsenic species
  - Volatilization loss not expected



ITRC, ISM-1, Section 9, Appendix C

### Speaker bullets

- Case study example, look for this
- Highlight the process decisions for lab process module
- Data shown in next module
- Air drying

### Narrative

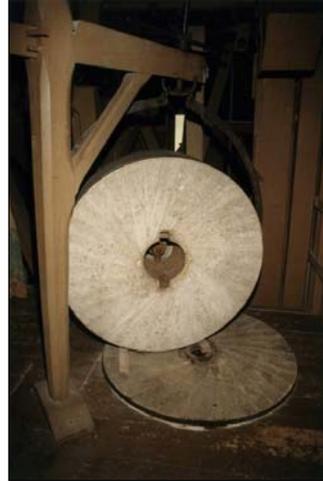
Since only high boiling arsenic species were expected in these surface soil samples for the golf course study, air drying was selected as the initial sample processing step.

### Supplemental information

Chapter 9 Appendix C

## Define Terms: Grinding

- ▶ Generic term for soil disaggregation or milling
- ▶ The grinding type or equipment must be specified to select a particular laboratory process



### Speaker bullets

- \* Grinding defined

### Narrative

Grinding is a generic term that includes soil disaggregation or milling. The specific grinding technique or equipment must be specified to select a particular lab process. By itself the word grinding is not specific enough for a lab to know what process choices are needed to support the project DQOs.

## Define Terms: Disaggregating

- ▶ Breaking all the soil clumps into individual small particles, but keeping the small pebbles and hard crystalline particles intact



ITRC, ISM-1, Section 6.2.2.3

### Speaker bullets

- Disaggregation defined
- Compared to milling
- Higher std dev of subsampling
- Lower measured metals concentration

### Narrative

Disaggregating means breaking the soil clumps into individual small particles but keeping the small pebbles and other hard crystalline materials intact. This can be accomplished using a bladed coffee chopper or gently crushing the dirt clods on a sieve. Even a hand mortar and pestle can be used gently to break up the clods without milling the stones.

### Supplemental information

See the end of Section 6.2.2.3

## Define Terms: Milling

- ▶ Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g.  $< 75 \mu\text{m}$ )



Picture from USACE-Alan Hewitt



ITRC, ISM-1, Section 6.2.2.5

### Speaker bullets

- Milling defined
- Compared to disaggregation
- Lower std dev
- Higher measured metals concentration

### Narrative

Milling on the other hand is complete particle size reduction of all soil components including the hard crystalline materials to a defined maximum particle size.

### Supplemental information

See Section 6.2.2.5

## Florida Case Study: Particle Size Reduction



- ▶ Disaggregation and sieving
  - Nugget effect expected to be small
    - Contaminant exposure sprayed as a liquid
- ▶ Mill
  - Puck mill
- ▶ Comparison study planned



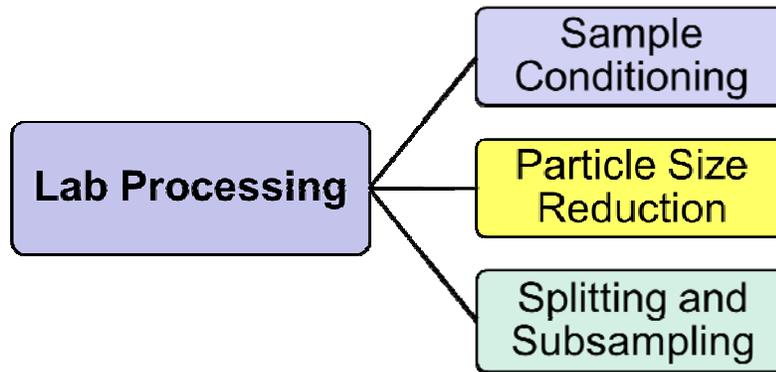
### Speaker bullets

- Disaggregate and #10 sieve
- Sample split for milled and unmilled processing

### Narrative

For the golf course study, after air drying the samples were disaggregated and sieved (#10, < 2mm). The nugget effect was expected to be small. Each sample was split with half for puck milling and the remainder kept unmilled for a comparison study.

## Lab Processing Roadmap



No associated notes.

## To Mill or Not to Mill? (Particle Size Reduction)

### ► Recommended

- Crystalline particles, fibrous threads, paint chips
- Energetics, metals

### ► Strengths

- Reduces variability
- Reduces subsampling error
- Facilitates mixing
- Improves precision



Picture from USACE-Alan Hewitt



ITRC, ISM-1, Section 6.2.2.5

### Speaker bullets

- Recommended
- When nugget effect is significant
- Energetics
- Metals

### Narrative

“To mill or not to mill” depends on the analytes, nugget effect and data quality objectives. Milling is recommended for energetics and metals when present in the sample as crystalline particles or fibrous threads. The particle size reduction associated with milling reduces variability. It reduces the subsampling error and improves precision.

### Supplemental information

See Section 6.2.2.5

## To Mill or Not to Mill

### ▶ Not recommended

- Volatile, thermally labile, increased “availability”



#### • Examples

- Monochloro PCBs, reactive SVOCs, decane, elemental mercury



#### • Limitations

- Analyte losses
- Metals contamination
- Potential high bias to metals risk assessment (pebbles)

**If uncertain,  
do milled & unmilled**

ITRC, ISM-1, Section 6.2.2.5

### Speaker bullets

- Not recommended
- Organics
- Thermal stability
- Low boiling
- Increase metal availability
- Metals contamination

### Narrative

Milling is generally not recommended for organics such as PCBs, Pesticides, hydrocarbons, semivolatile organics and some metals because some individual analytes in these groups can be lost through volatilization or thermal decomposition. Also, these analytes are less likely to be present as crystalline particles. The use of a metal mill can produce selected metal contamination of the sample. For some risk assessment purposes releasing the metals from inside the pebbles to produce higher measured concentrations is a potential high bias for the results.

### Supplemental information

See Section 6.2.2.5

## How Best to Mill

- ▶ Puck mill or ring and puck mill
  - “Stable” energetics

- ▶ Ball mill

- ▶ Mortar and pestle

- ▶ Consider

- Analytes
- Concentration of interest
- Mill materials
- Particle size needed



Picture from USACE-Alan Hewitt



Example mills, other types are possible as well

ITRC, ISM-1, Section 6.2.2.5

### Speaker bullets

- Puck mill
- Ball mill
- Mortar and pestle
- Compare analyte to mill materials

### Narrative

If one chooses to mill a soil sample there are several options. The puck mill has been demonstrated to be effective for energetics (Method 8330B) and some metals. The puck mill produces the most consistently small particles among the options, thus reducing the standard deviation of replicate results the most. The ball mill is a less expensive and less rigorous option that is still suitable for many applications. It produces a moderate improvement in precision. The mortar and pestle, either automated or manual, typically produces some precision improvement. When choosing the milling equipment consider the analytes, concentration of interest, mill materials and particle size needed. This is not an exclusive list of mills, other equipment can be used, if it meets data quality needs.

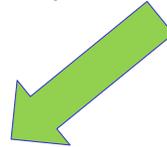
### Supplemental information

See Section 6.2.2.5

## Florida Case Study: Results Confirm Milling Not Needed



- ▶ Disaggregation and sieving
  - Nugget effect expected to be small
    - Contaminant exposure sprayed as a liquid
- ▶ Mill
  - Puck mill
- ▶ Results confirm milling not needed for this part of site
  - Small precision improvement with milling
  - No change in mean concentration



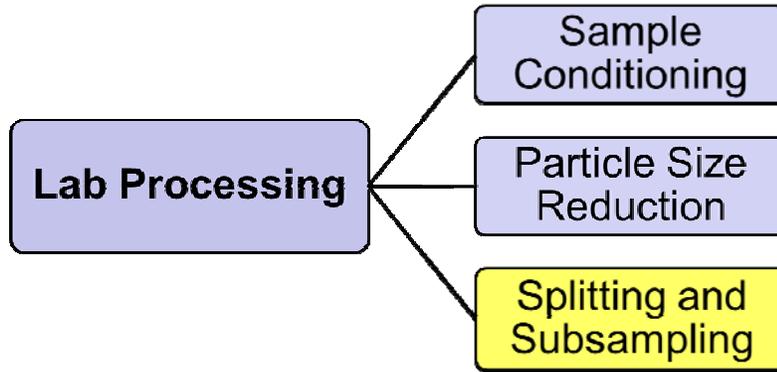
### Speaker bullets

- Only a small difference between milled and unmilled results.
- Milling not needed
- Due to liquid applied COC

### Narrative

For the golf course, the results from the comparison of unmilled and milled sample results showed that there was essentially no change in the mean concentration results and only a small improvement precision. These results confirm that milling was not needed for this site where the arsenic contaminant of concern was distributed as a liquid and the nugget effect was small.

# Lab Processing Roadmap



No associated notes.

## Dry Splitting Options



### ▶ Rotary sectorial splitter



ITRC, ISM-1, Section 6.2.2.7

#### Speaker bullets

- Rotary sectorial splitter
- Best precision
- More expensive
- Less available

#### Narrative

The automated rotary sectorial splitter produces the most consistent and representative sample splits, thus providing the best precision. In this technique the sample flows from the hopper on top of the machine and is equally distributed into the small sample containers on the rotating sample collector.

Sectorial splitter subsampling can substantially minimize bias and improve precision.

#### Supplemental information

See Section 6.2.2.7

## Subsampling Options

### ↓ ▶ 2-Dimensional Japanese Slabcake



Dry

Wet



ITRC, ISM-1, Section 6.2.2.7

#### Speaker bullets

- 2 D slabcake
- Lower cost than sectorial splitter
- Pretty good representativeness
- Wet or dry sample
- Systematic Random design
- All increments combined = analytical subsample

#### Narrative

The 2 dimensional Japanese slabcake frequently provides acceptable subsample representativeness at a lower cost than the sectorial splitter. This process is a miniaturized version of what takes place in the systematic random field sample collection process. The wet or dry processed sample is spread evenly in rectangular slabcake and divided into grids as determined in project planning. The default is 30. The analyst removes a small increment from a random location in the first grid. Subsequent increments are collected from the same location in the other grids. All increments are combined to form the subsample for digestion or extraction so the size of the increment must be appropriate for the number of increments and the target subsample size.

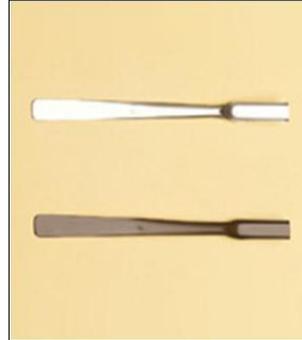
2D slabcake subsampling can minimize bias and improve precision.

#### Supplemental information

See Section 6.2.2.7

## Subsampling Tools

- ↓ ▶ Square straight-sided scoops for dry non-cohesive soil



### Speaker bullets

- Non-cohesive soil
- Blunt scoop with sides
- Not round scoop

### Narrative

Proper increment collection from the 2 dimensional slabcake should use a blunt end scoop with straight sides so that the particles from the top, middle and bottom of the slabcake are all sampled equally. Rounded spoons, scoops and spatulas should not be used because they discriminate against the particles on the bottom of the slabcake and can bias the subsample. The proper tool improves subsample representativeness and reduces bias.

## Florida Case Study: Choose Subsampling Process

- ▶ 2-D Slabcake Subsampling
  - Lower cost than sectorial splitter
  - More representative than “dig a spot”



### Narrative

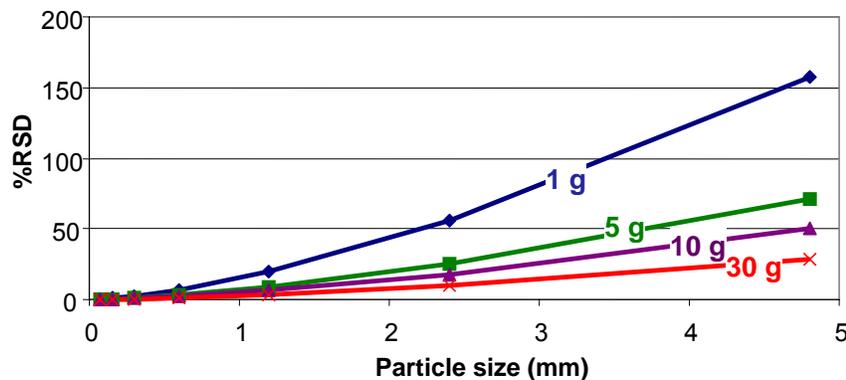
The 2 dimensional slabcake was chosen as the best balance between cost and representativeness for the golf course study.

## Why Use Large Subsamples?

### ► Larger particles



- Produce larger errors or require larger subsamples



ITRC, ISM-1, Section 6.3.3

### Speaker bullets

- Large particles >> more variable results
- Reduce variability – larger subsamples
- See Day 1 training about the heterogeneity of soil for the causes

### Narrative

When particle size reduction through milling is not appropriate or available, the use of larger subsample sizes can reduce the variability and produce better precision for replicate samples. For example, with metals analysis of 1 g samples, %RSD > 100% is expected when large particles are present. Precision can be improved significantly without milling by increasing the subsample size to 5 or 10 g. During the Part 1 training the heterogeneity of soil touched on this correlation between variability and particle size.

### Supplemental information

See Section 6.3.3 and ASTM Standard D6323 Sec A1.1

## Florida Case Study: Nugget Effect Minimal

- ▶ 2 g subsamples on disaggregated aliquots
- ▶ 2 g subsamples on milled aliquots
  
- ▶ Low heterogeneity expected
  - Confirmed through replicates



### Narrative

The golf course study used 2 g subsamples for both the disaggregated and milled soil samples since the nugget effect was expected to be small. The results from this case study will be discussed in the next module.

## Laboratory Quality Control Measures



- ▶ Laboratory equipment blanks
  - Limited clean matrices
- ▶ Laboratory control samples (LCS) and matrix spikes
  - Practicality of large scale spiking in kg samples
    - High cost
    - Limited availability
  - Introduced post ISM processing into subsample
- ▶ Subsampling replicates

### Speaker bullets

- QC
- Blanks – no single clean matrix for all analytes
- LCS – matrix + cost
- MS - cost
- Replicates - triplicates

### Narrative

Quality control measures include equipment or method blanks, laboratory control samples, matrix spike samples and subsampling replicates. There is no one single clean matrix for use with all analytes that mimics the characteristics of soil. Large scale LCSs and matrix spikes currently have significant cost associated with them and may not always be practical. Currently it is more common to introduce these QC measures after the preliminary ISM processing is complete. Subsampling replicates of field samples are suggested as a good way to assess laboratory reproducibility. Triplicates are the most common choice.

### Supplemental information

See Section 6.4

## Florida Case Study: Challenges with “Blank” Samples



- ▶ Ottawa sand method blank attempted for milling
  - Metals content of the sand was too variable
- ▶ Standard preparation batch QC
  - No laboratory control sample or matrix spike through ISM processes



### Narrative

The Florida golf course study attempted to use Ottawa sand as a milling equipment blank material, but in the end it was determined that the arsenic content in the as-received material was too variable. Standard preparation batch QC was used. No laboratory control samples or matrix spikes were run through the various ISM processes.

## Verify Laboratory Certification



- ▶ National Environmental Laboratory Accreditation Program (NELAP)
- ▶ Non-NELAP state accreditation
- ▶ Agency-specific accreditation
  - DoD Environmental Laboratory Approval Program



ITRC, ISM-1, Section 6.4.1

### Speaker bullets

- Certification options from these
- NELAP
- State
- Agency (e.g. DoD)

### Narrative

Lab certification is available through the usual avenues; the National Environmental Laboratory Accreditation Program run by the NELAC Institute, non-NELAP states and agency specific accreditation such as the DoD Environmental Laboratory Approval Program.

### Supplemental information

See Section 6.4.1

## Cite Reference Methods



- ▶ Collecting and Processing of Representative Samples For Energetic Residues in Solid Matrices from Military Training Ranges
  - USEPA SW-846 Method 8330B, Appendix A  
<http://www.epa.gov/osw/hazard/testmethods/pdfs/8330b.pdf>
  
- ▶ Metals in Solid Matrices
  - USACE research effort
  - Planned SW-846 Method 3050 - Update V?

ITRC, ISM-1, Section 6.4.1

### Speaker bullets

- Reference methods
- 8330B
- 3050

•Certification usually based on analysis method, secondarily extraction of digestion and subsampling ignored, but now covered for ISM

### Narrative

At this point there are few references methods to cite. EPA Method 8330B for explosives is the best known and most widely used and recognized. The US Army Corps of Engineers is working on a modification of EPA metals digestion Method 3050. Hopefully this will make it into Update V for SW-846.

### Supplemental information

See Section 6.4.1

## Use Alternate References

- ▶ ASTM D6323 Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities
  - ASTM 2003
  
- ▶ Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples
  - Gerlach 2003
  
- ▶ Laboratory Standard Operating Procedure

ITRC, ISM-1, Section 6.4.1

### Speaker bullets

- General references
- ASTM
- EPA
- Lab SOP

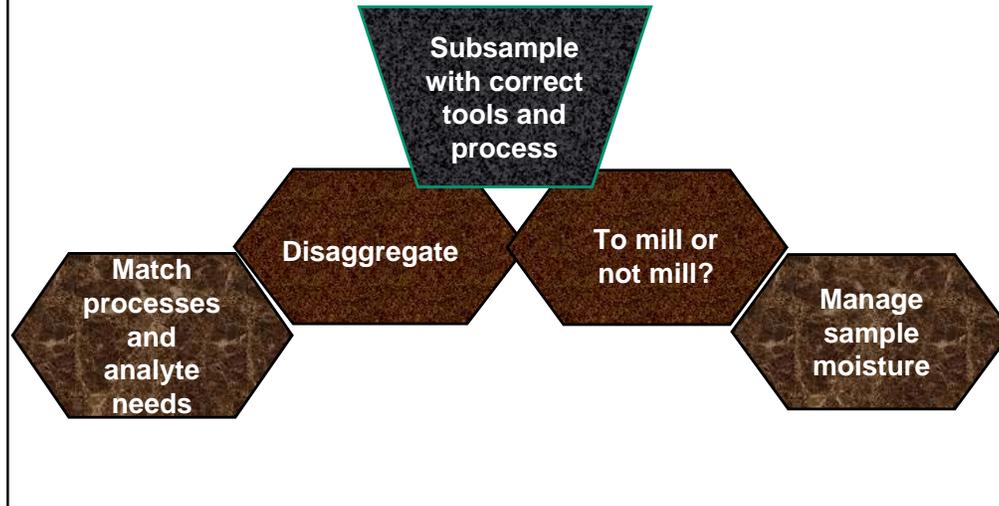
### Narrative

There are two general references related to subsampling and particle size reduction, one from ASTM and the other from the EPA. In the absence of a specific reference method some states will certify based on a specific laboratory standard operating procedure.

### Supplemental information

See Section 6.4.1

## Lab Process “Big Rocks”



### Speaker bullets

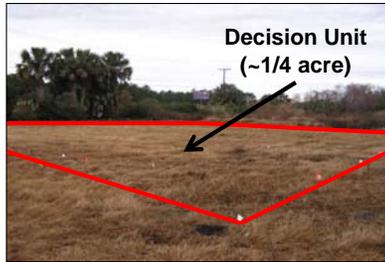
- Big Rocks
- Match analyte and processes
- Manage moisture
- Disaggregate
- Mill or not
- Subsample

### Narrative

In closing, there are five foundational “big rocks” that must be addressed for lab processing of ISM samples. First, match the processes and the analyte needs. Second, manage sample moisture according to these analyte needs. Third, disaggregate samples by breaking up the soil clods. Fourth, answer “To mill or not to mill?” and choose the appropriate process. Fifth and last use the right subsampling process with the right tool to collect the most representative subsample to run through the sample digestion or extraction process.

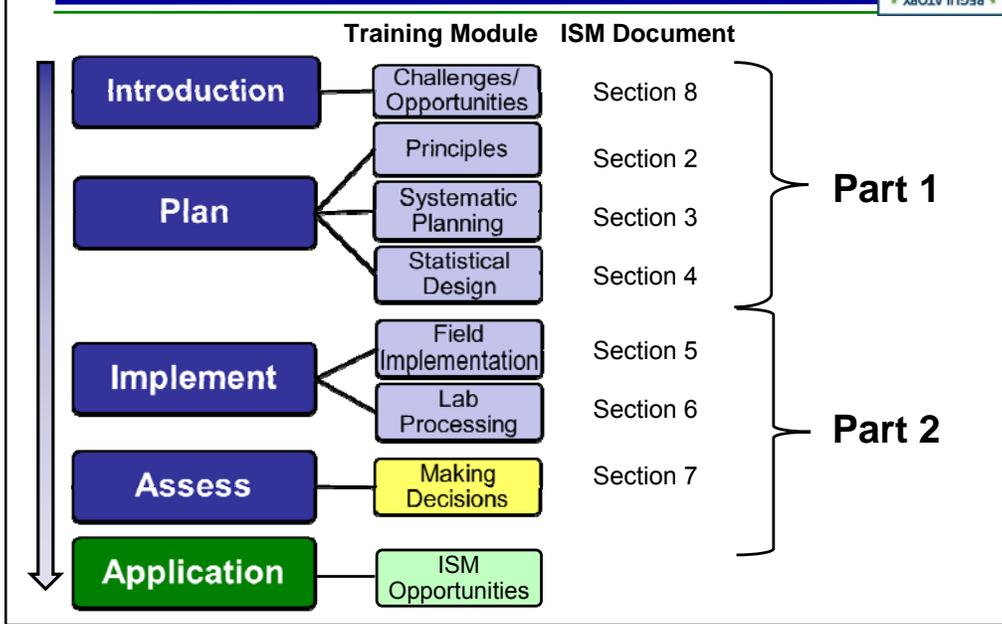
The next module will be presented by Tim Frederick from the EPA Region 4 (Jay Clausen from the US Army Corps of Engineers)

# Question and Answer Break



No associated notes.

# ISM Document and Training Roadmap



No associated notes.

## Making Decisions: Learning Objectives

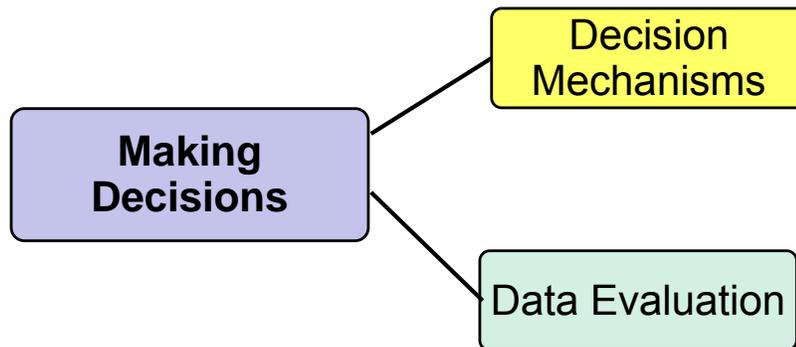
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### Learn how to:

- ▶ Use ISM data to make decisions
- ▶ Evaluate data
  - Identifying sources of error
  - Quantify error
  - Interpret error
  - Isolate sources of error

No associated notes.



### Speaker bullets

- Introduce decision mechanisms (not necessarily different from the way we make decisions with discrete data, but some wrinkles with ISM data that must be considered).
- Decision mechanisms chosen BEFORE data collection
- Data Evaluation – tools we use to assess whether we have adequately addressed project goals
- Data evaluation occurs AFTER data collection

## Making Decisions



- ▶ Decision Mechanism (DM)
  - Structured approach to making decisions
  - Identified and agreed upon during Data Quality Objective (DQO) process
  - 6 common types of DM

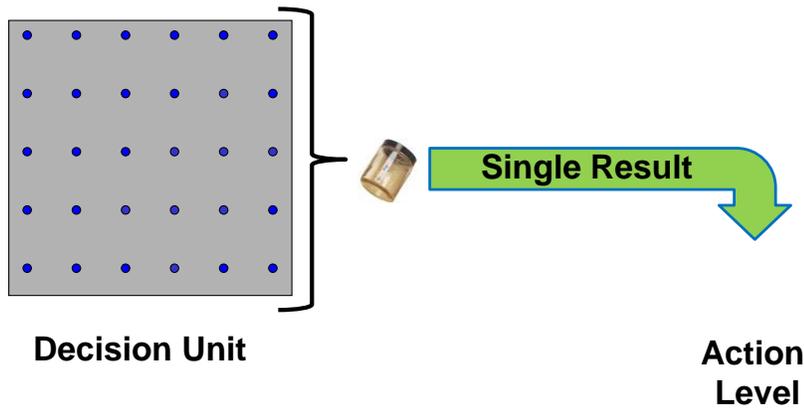
Bullet 1: ITRC Definition: an algorithm or protocol that results in the decision about a potential contaminant of concern or for a decision for a volume of media.

Each decision mechanism has strengths, weaknesses, and assumptions. In some cases, agency requirements will dictate the approach to be used – not unique to ISM, but there are some wrinkles to ISM that should be considered

Bullet 2: understand what the decision is and how it will be determined; avoid confusion – make a priori decisions; It is critical during strategic planning that parties agree up front;

Bullet 3: indicate that these are examples; you should come up with site-specific criteria (not all DMs will be acceptable in all states, scope before hand)

## DM 1: Compare One ISM Result to Action Level



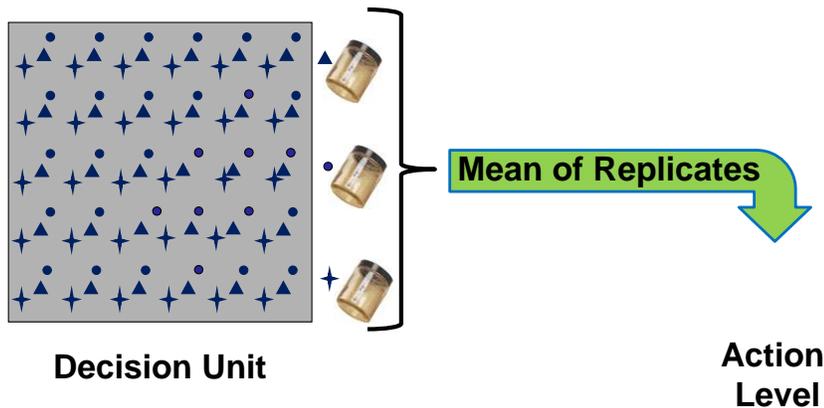
ITRC, ISM-1, Section 4.2.1 and Section 7.2.1

**Speaker Bullets** - The simplest decision mechanism is to compare a single ISM result to an Action or Screening level. There are some limitations to this approach, however. -- verifying something that you expect (well above or below) – Refer to Module 4 (4.2.1) and Module 7 (7.2.1)

### Narrative -

This approach might be acceptable when the predicted mean concentration is much greater than, or much less than, the action level. In this situation, the ISM sample provides confirmation of what is already strongly suspected—that the DU clearly passes or fails. However, when the ISM sample result is close to the action level, this decision mechanism is unreliable and decision errors in both directions are possible (i.e., concluding that the DU fails when the average concentration is in fact below the action level, or vice versa). This approach may not be acceptable to regulatory agencies for decision making. Acceptability of this approach will depend largely upon the objectives of the sampling.

## DM 2: Compare Average ISM Result to Action Level



ITRC, ISM-1, Section 7.2.2

**Narrative** - In this decision mechanism, a number of replicate ISM samples are collected in the field from the same DU. These replicates provide a measure of the variability of the entire sampling, preparation, and analytical process. The mean concentration of the replicates is calculated and compared to the action or screening level identified during systematic planning.

The mean concentration from replicate samples is likely to be closer to the true mean of the DU than the result from a single ISM sample (see Ch 4), and could, therefore, be considered to provide a more reliable estimate of the mean. There is no assurance, however, that the actual mean concentration has not been underestimated.

Consequently, this decision mechanism would not be useful in circumstances where project objectives dictate that the mean concentration estimate must be conservative (e.g., EPC values in most human health risk assessments). \*\*\*\*\*Some regulators may ask that error be calculated\*\*\*\*\*accuracy vs precision\*\*\*\*\*

## Florida Case Study: Decision Mechanism (DM) 2



Mean arsenic concentrations (mg/kg)

	Discrete <i>n</i> = 30	Incr-30 <i>n</i> = 3	Incr-100 <i>n</i> = 3
DU 2	4.2	5	5.2
DU 3	7.5	10.5	9.5

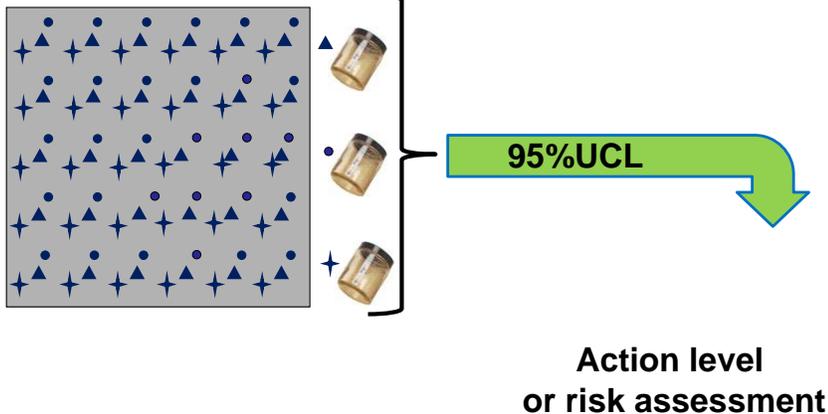


**Narrative** – This table presents the data from two DUs from the Florida pilot project (see Appendix C for more information). Please note, however, that the 30 discrete samples collected in the quarter-acre DU is more than would be generally collected on a typical site.

In this investigation, incremental sampling methods resulted in higher contaminant concentrations – this is a noteworthy example for those who worry that incremental sampling systematically dilutes concentrations

## DM 3: Calculate 95%UCL then Compare to Action Level or Use for Risk Assessment

### Decision Unit



ITRC, ISM-1, Section 4.2.2 and Section 7.2.3

- Typically for comparison of a 95% UCL to an action level, use in a risk assessment, or wherever a conservative estimate of the mean is required (where 95% UCL would be used as an exposure point concentration)
- Need at least three replicates – results improve (closer to the mean) w/ more replicates

**Narrative** - Project objectives may specify that the estimate of the mean concentration provided by ISM sampling must be health protective, meaning that there is low chance of underestimating the actual mean concentration within the DU, such as for use in a baseline risk assessment.

Calculation of a 95% UCL for ISM data requires a minimum of three ISM samples. This is fewer than is required for discrete data sets to yield reliable 95% UCL values. Additional replicates increase the performance of the mean estimate (i.e., provides a 95% UCL closer to the actual mean), and although this increases the cost, it may be worthwhile if the site is relatively heterogeneous and the result is anticipated to be close to the action level.

Section 4.2.2 link to the calculator

## Florida Case Study: Decision Mechanism 3: (DU 1)



### Arsenic Data (mg/kg)

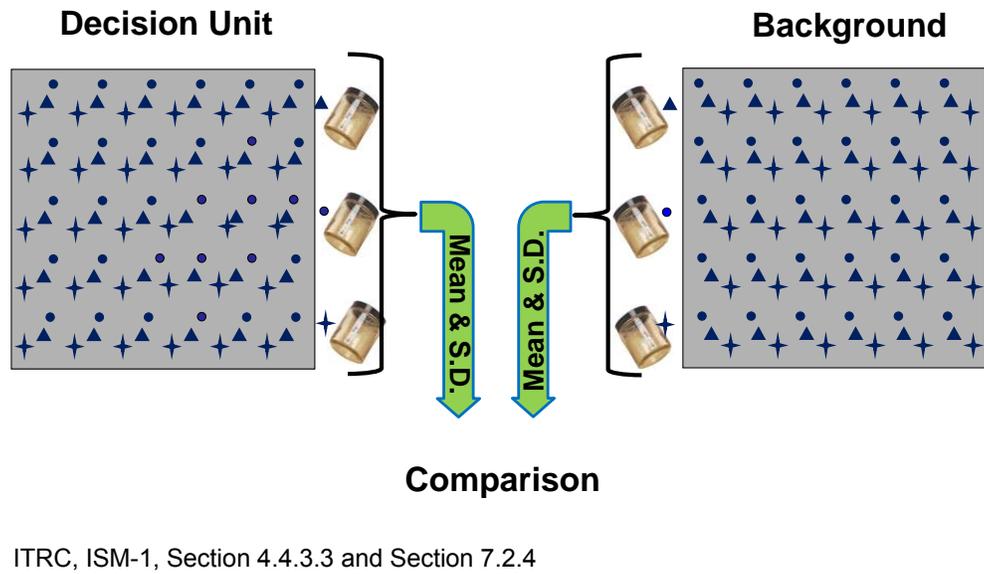
	Discrete n = 10 (mg/kg)	Incr-30 n = 3 (mg/kg)	Incr-100 n = 3 (mg/kg)
Mean	2	1.8	1.7
Std Dev	1.4	0.08	0.03
<b>95UCL</b>	<b>3.0</b>	<b>2.0</b>	<b>1.8</b>

Florida Action Level: 2.1 mg/kg



**Speaker Bullets** - Means are similar; variability (std dev) much lower for incremental samples; variability affects 95% UCL; in this case – method used determines the decision to be made

## DM 4: Compare to Background

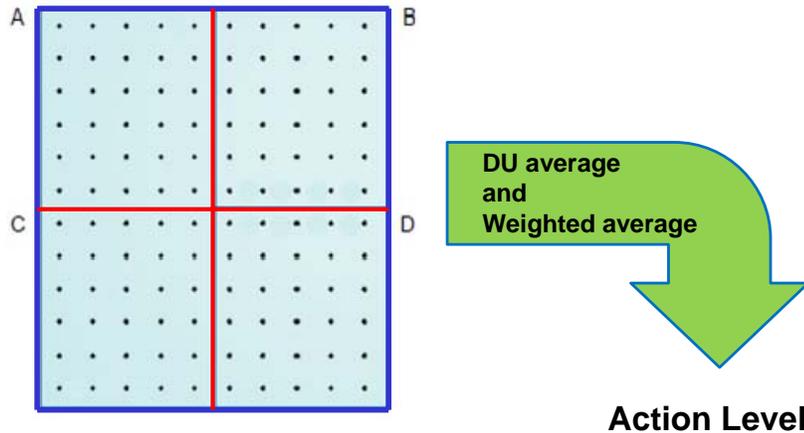


### Narrative:

ISM results are not suitable for point-by-point comparison with UTLs generated from discrete sample background data because ISM and discrete datasets have fundamentally different characteristics. If background and site data are both generated using ISM, comparisons of central tendencies (e.g., medians) can be made using hypothesis testing, but statistical power to detect differences will be low due to the limited number of replicates in most ISM data sets. Therefore caution is urged when comparing site to background. The best approach is to use graphical analysis to compare the ISM site data and ISM background data.

See Section 7.2.4 for more information

## DM 5: Combining Decision Units

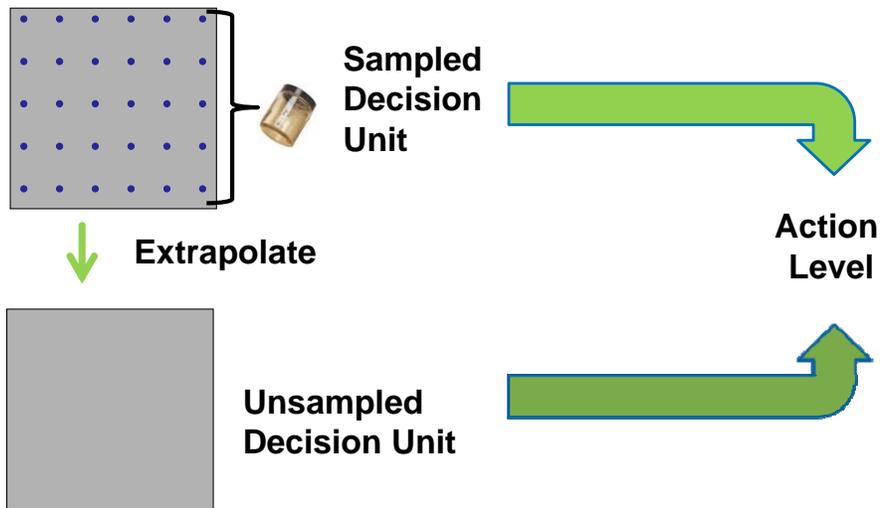


ITRC, ISM-1, Section 4.4.1 and Section 7.2.5

Combining results from two or more small DUs to estimate the overall mean concentration in a larger combined DU is advantageous when the data must support more than one decision (e.g., overlapping exposure units for ecological and human health receptors). In this example, The decision Units A, B, C, and D (red squares) represent 1/4 –acre residential exposure units. A decision must be made on each of these units. However, an ecological receptor may range over the entire site, and a separate decision may need to be made on the larger “super DU” (blue square) Both of these project goals could be accomplished by pooling the data of the smaller DUs to calculate a weighted average for the “super DU.”

The formulas for calculating weighted averages are presented in Chapter 4 of the ITRC Tech Reg. These formulas take into account the size of the smaller DUs, weighting their contribution to the larger DU accordingly. The ability to combine DUs extends vertically as well as horizontally; that is results from DUs from different soil depths can be combined if needed to meet sampling objectives

## DM 6: Extrapolation to Unsampled Areas



ITRC, ISM-1, Section 4.4.4.2 and Section 7.2.6

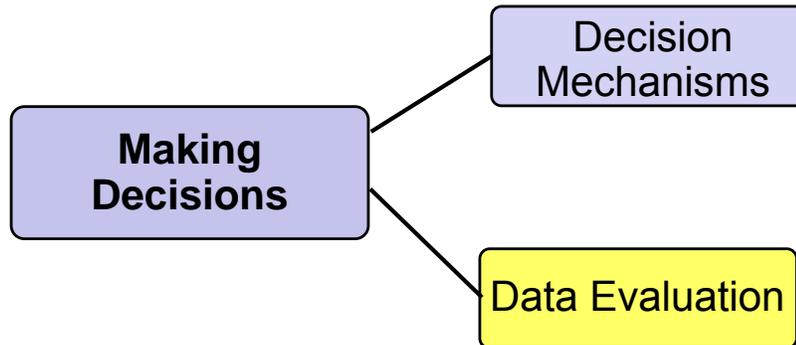
**Speaker Bullets – Not unique to ISM** ---fundamental assumption made with this mechanism is that the distributions of contaminant concentrations in the unsampled areas are essentially the same as in the sampled areas, in the absence of data to verify the assumption, the confidence in the assumption is subjective.

This DM (decision mechanism) involves extrapolating data from a sampled DU to an unsampled DU. The results from the sampled DU are assumed to also apply to the unsampled DU and decisions are made accordingly. \*\*\*\*This approach may not be acceptable to individual states or other regulatory agencies based on feedback received during the comment period and the possible introduction of decision errors (See Section 4.4.4.2)\*\*\*\*

### Narrative

- Frequently proposed for large tracts of land or large volumes of soil to reduce costs.
- May be necessary when funds or other constraints prohibit sampling all DUs
- ISM has no unique capability to enable extrapolation or reduce the uncertainty involved.
- The issue is the same whether the DU is sampled by ISM or with discrete sampling – is there sufficient basis to assume that the mean concentration estimated there is the same (or higher) than the mean concentration in unsampled DUs.
- Must assume that both the mean and the distribution of contaminants are essentially equivalent across DUs

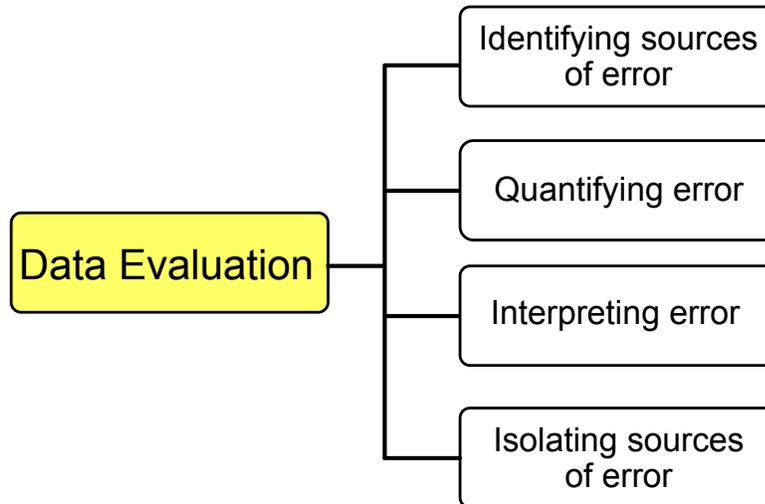
## Making Decisions Using ISM Data



**Speaker bullets – decision mechanisms decided upfront / data evaluation**

The purpose of this section is to describe how to evaluate the data to ensure that sampling objectives have been met.

## Data Evaluation Components



Data evaluation can include several steps that we will discuss in the following slides

## Identifying Sources of Error

### Field

- ▶ Number of increments
- ▶ Increment collection
- ▶ Field processing
- ▶ Field splitting
- ▶ DU size and shape

### Laboratory

- ▶ Lab processing
- ▶ Subsampling
- ▶ Extraction
- ▶ Digestion
- ▶ Analysis

Error (Variability) – not “mistake”

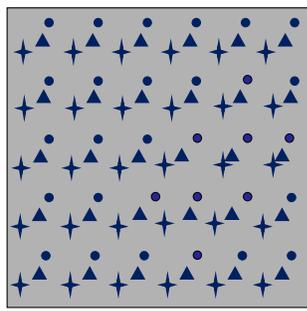
Speaker Bullets – not comprehensive; theoretical sources of error discussed in Chapter 2 (Section 2.2) ISM controls these better than discrete

It is useful when evaluating data to consider the steps in the process where errors (variability) may have been introduced

If error is determined to be unacceptable in a given data set – one or more of these sources may be at fault

## Quantifying Error

$$\text{RSD} = \text{CV} = \text{standard deviation} / \text{arithmetic mean}$$



**Decision Unit**



**Data includes all sources of error**

ITRC, ISM-1, Section 4.3.1.3 and Section 7.3

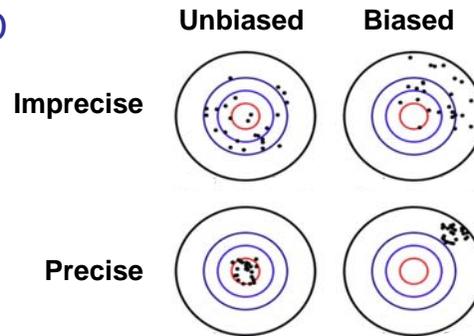
**Narrative** - Evaluation of overall error can be accomplished by using replicate data to calculate a relative standard deviation (RSD) for the replicates. The RSD is a statistical measure of variance that quantifies the precision of the data, although not the accuracy of the estimate. RSD is a measure of reproducibility of estimates of the mean provided by replicates. It provides a measure of the total error associated with the replicates. (see Section 4.3.1.3 for more info)

In order to calculate appropriate statistics, at least three field replicate samples are needed.

## Interpreting Error



- ▶ “Unacceptable” RSD
- ▶ Low RSD
- ▶ High RSD



ITRC, ISM-1, Section 4. 3.4.4 and Section 7.3

- While there are rules of thumb provided in other guidance documents (CHECK WITH YOUR STATE REGULATORY AGENCY), the ITRC offers no prescribed definition of “acceptable” RSD (should be evaluated on a site-by-site basis) – See Section 7.3 for more info

- Low RSD – Generally you don’t need to go back and identify sources of error. While a low RSD may indicate that the field replicates are providing reproducible estimates of the average, this is not always the case -- is not always an indication that the mean is accurate or that the 95% UCL exceeds the population mean (unless the distribution can be reasonably assumed to be relatively homogeneous) \*\* An estimate of the average from replicates with a low RSD is not necessarily close to the actual mean. The opportunity for significant error is greatest when the DU is relatively heterogeneous and the replicates by chance give similar results.

- High RSD may indicate errors introduced through lab or field activities or may be attributable to heterogeneity in site concentrations. Generally leads to the need to isolate potential sources of error – discussed on the earlier slide “Identifying Sources of Error” (see Section 4.3.4.4 for more info)

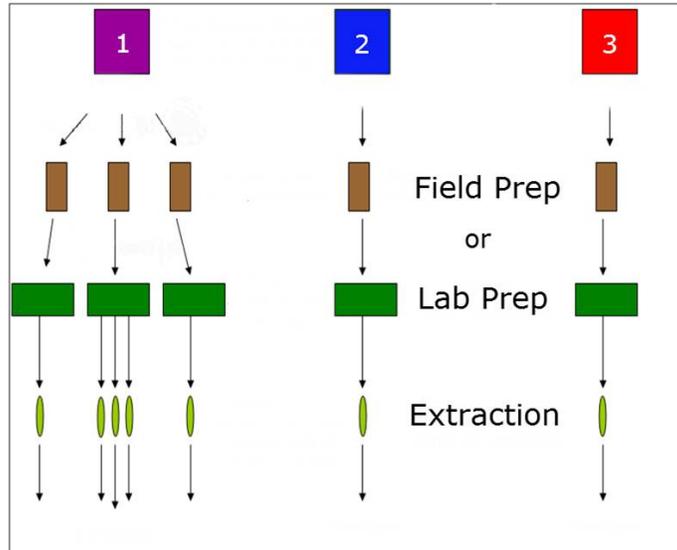
- Lab

- Field

RSD measures reproducibility, not accuracy of the results (see Section 4 for additional discussion of these concepts).

If the goal is to make sure that the mean is not underestimated, a 95UCL should be calculated regardless of whether the RSD is high or low.

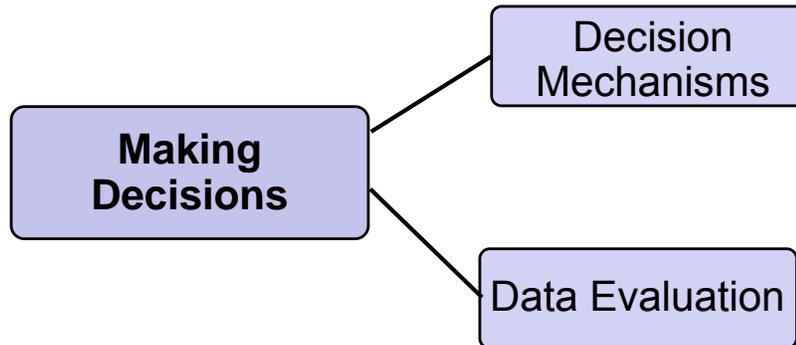
## Isolating Sources of Error



Adapted from EPA 2011, page 38: <http://go.usa.gov/EAE>

Ideally, the project team will then designate one of these replicates for separation into laboratory replicates.) Replicate RSD data is intended to quantify the total error of the measurement system; and if one sample is split into laboratory replicates then the total error can be attributed to either field sampling or laboratory procedures.

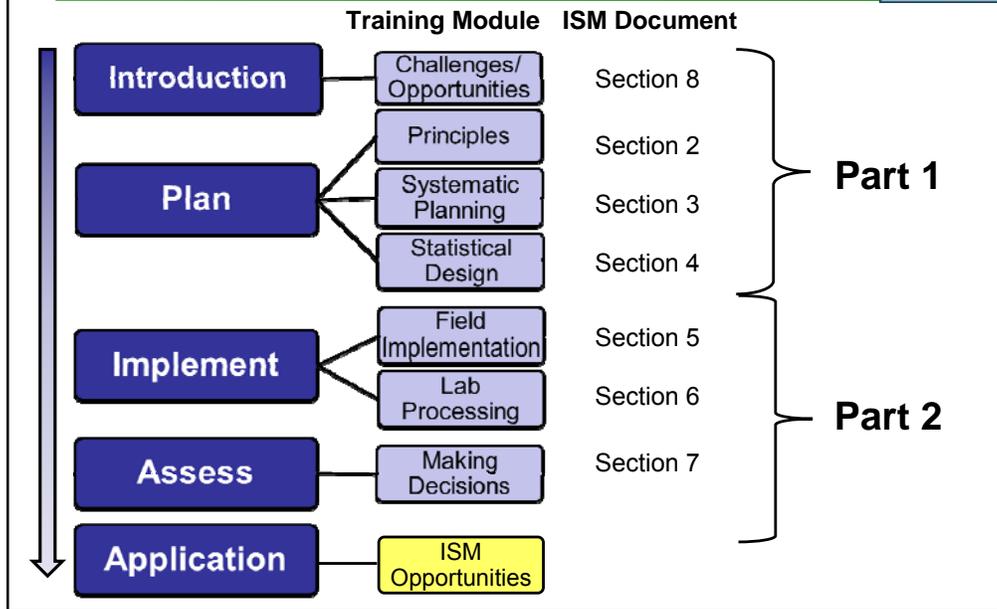
## Making Decisions Using ISM Data



**Speaker bullets** –The purpose of this section is to describe how to use the data generated through ISM samples to make decisions regarding a DU and how to evaluate the data to ensure that sampling objectives have been met.

**Narrative:** ISM provides estimates of the mean concentration of the contaminant in one or more SUs that can be used to make a decision regarding the DU. Since the data may inform one or more decision objectives, it is helpful to establish a structured approach to making decisions, referred to here as *decision mechanisms*. Systematic evaluation of the data can determine if sampling errors have been adequately addressed for the decisions being made.

# ISM Document and Training Roadmap



In the next few slides we will look at some of the types of sites and applications where ISM may be a useful tool to consider, briefly touch on cost considerations and then we'll recap some of the take home messages about ISM that we'd like for you to leave with today.

## ISM Applications



- ▶ Regulated sites
- ▶ Residential yards
- ▶ Former pesticide-applied orchards
- ▶ Stockpiled soil
- ▶ Post-soil treatment sampling
- ▶ Dredged materials



Residential Yard



Soil Stockpile

### Other Applications

We recognize that there are many potential applications for ISM in the work we do. We have focused on regulated sites – and there are already examples of where ISM has been applied successfully, with much thanks to members of the ITRC's ISM Technical Team.

However, we recognize that there can be reluctance and resistance to implementing ISM on regulated sites, so perhaps you can think of other opportunities – other circumstances and types of sampling – where there may be an easier 'point of entry' for demonstrating ISM in your state/region/jurisdiction. These (applications) are just some of the opportunities identified by members of the ISM Team - instances where the mean is appropriate & ISM makes sense (more so than discrete sampling).

Applications are grouped together in the following manner – evenly dispersed discharges, mixed/less variable soils, large areas, etc., and continue on to the next slide.

In NJ and many other states, there are restrictions (regulations) which limit the use of composite soil sampling as well as the averaging of soil sample results. Please refer to Table 8-1 in Section 8 of the ISM document for a complete listing of all states with restrictions on compositing. In NJ, however, there is a process whereby one can petition for a "variance" from the technical requirements and guidance.

The rationale for varying from the requirement needs to include documentation that the work conducted has achieved the objectives of the rule requirement or guidance from which it varied. Thus ISM could be utilized in NJ, and possibly other states, by providing the technical justification that it not only meets but most likely exceeds the discrete soil sampling requirements as well as its objectives.

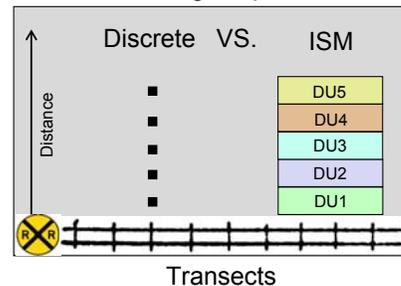
Speaker Note: We realize there are concerns.....others have had them too.....but open yourself to an opportunity for better decision- making through ISM

## ISM Applications (continued)

- ▶ Large Areas
  - Tailings impoundments
  - Agricultural fields
  - Floodplain soils
- ▶ Transects
- ▶ Dredged materials



Mine Tailings Impoundment



### Other Applications

We recognize that there are many potential applications for ISM in the work we do. We have focused on regulated sites – and there are already examples of where ISM has been applied successfully, with much thanks to members of the ITRC's ISM Technical Team.

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Speaker Note: We realize there are concerns.....others have had them too.....but open yourself to an opportunity for better decision- making through ISM

## ISM Applications (continued)

- ▶ Firing Ranges
- ▶ Confirmatory sampling
- ▶ Background
- ▶ Other
  - Fill material
  - “Rail to trail” sites



Small Arms Firing Range



Post-Excavation Confirmatory Sampling

### SLIDE on ISM Applications (continued)

So maybe you have already seen some opportunities – either on regulated sites or some other circumstance – where ISM might be a good fit. Or maybe you could bring up some other possibilities not listed here during the question/answer period that’s coming up.

**Another tool in the toolbox.** Remember ISM, although an improvement in regards to environmental sampling doesn’t invalidate what we have been doing...

...but we think it will make life a whole lot easier (and better) in many, many circumstances.

## How Does ISM Cost Compare?



### Elements

- ▶ Planning
- ▶ Field Collection
- ▶ QA/QC Samples
- ▶ Sample Transport
- ▶ Sample Processing/Conditioning
- ▶ Lab Analysis
- ▶ Overall Sampling/Analysis Portion of Project

- It depends
- We can pull out each element of a sampling/analysis project and take a look at how ISM might compare
- Planning – about the same, maybe a little more for those less familiar with systematic planning and DU concept
- Field Collection – Often ISM costs more
- QA/QC – can be fewer QA/QC samples with ISM since fewer overall samples collected
- Sample Transport can be higher with ISM since samples are so much larger
- Sample Processing/Conditioning – since there often is none of this with discrete, ISM will add to cost here.
- Lab analysis – per sample can be higher due to subsampling protocol; but fewer samples submitted can mean lower lab bill
- Overall – often the effect of fewer samples collected for analysis means a lower overall cost
- But remember, these are only sampling/analysis costs; as we'll see in a couple slides that does not really tell the whole story
- But first, let's look at a specific cost comparison example

## A Cost Comparison Example



### US Army Corps of Engineers Study Metals in Soil

Activity	Per Sample Cost (\$)		Total Project Sampling/Analysis Cost (\$)		
	ISM	Discrete	ISM <sup>1</sup>	Discrete <sup>2</sup>	Discrete <sup>3</sup>
Field Sampling	35-50	10-15	105-150	70-105	150-225
Lab Prep	40-60	0-10	120-180	0-70	0-150
Analysis	225-275	125-135	675-825	875-945	1,875-2,025
<b>Total</b>	<b>300-385</b>	<b>135-160</b>	<b>945-1,155</b>	<b>945-1,120</b>	<b>2,025-2,400</b>

<sup>1</sup> Based on 3 replicate 100-increment ISM/DU

<sup>2</sup> Based on collection of 7 discrete samples/DU

<sup>3</sup> Based on collection of 15 grab samples/DU

Source: US Army Corps, Cost and Performance Report of Incremental Sampling Methodology for Soil Containing Metallic Residues, ERDC TR-13-10, September 2013

- Two case studies in guidance doc looked at cost - both showed 50%-80% savings vs. discrete; both looked at energetics
- This table is from a recent USACE cost comparison study looking at metals in soil
- Included two discrete sampling scenarios; 7 samples/DU and 15/DU
- Found that although per sample cost 35-53% higher, overall project cost was up to 40% lower.
- Again want to emphasize these are just the sampling analysis project costs

## Bottom Line on Cost Comparisons



Measuring the cost difference  
between ISM and discrete sampling.



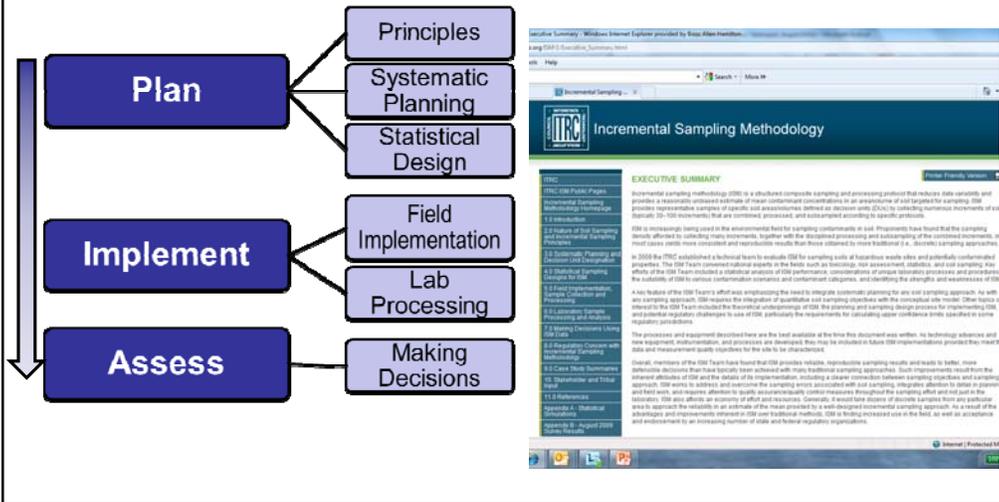
Measuring the cost of making a  
wrong decision.

- Sampling/analysis usually leads to some decision
- Quality of the decision is only as good as quality of the data on which it is made
- We've seen how ISM can provide a very high quality estimate of mean concentration
- So while it is relatively easy to compare cost of ISM vs. discrete sampling/analysis plans,
- Ultimately what matters is the cost of making a decision error – how expensive is it to unnecessarily excavate 100 yd<sup>3</sup> soil? How expensive is it to walk away from a site that actually poses a risk to human health?

# ITRC's ISM Solution



Web-Based Document at:  
<http://www.itrcweb.org/ISM-1/>



Here's our answer to how to get a representative sample.

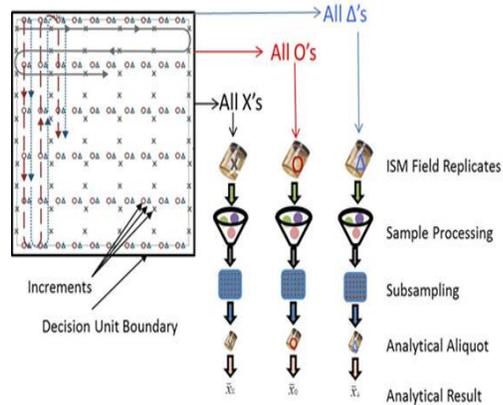
This flow chart/illustration provides a snapshot of the contents and structure of our two-day training – and of our Technical and Regulatory Guidance Document on incremental sampling methodology. From planning through assessment we've developed a process for considering all the important aspects of getting that 'representative sample' we've been talking about. And remember – all the elements we have displayed here in this figure would pertain to other types of sampling – but it turns out that incremental sampling does a better job for many circumstances, and that is the focus of our document and training.

The Technical and Regulatory Guidance Document is over 400 pages (web-based) – so hopefully this 2-session training has given you the comfort - and motivation - to dive in to the more detailed presentation in the actual document.

## Overview & Wrap-up

### ISM Provides:

- ▶ Unbiased estimate of the mean
- ▶ Improved spatial coverage
- ▶ Increased sample representativeness
- ▶ Control over most common sources of sampling error
- ▶ Reduced data variability
- ▶ Ability to calculate 95% UCL



No associated notes.

## Thank You for Participating



- ▶ 2nd question and answer break
- ▶ Links to additional resources
  - <http://www.clu-in.org/conf/itrc/ISM/resource.cfm>
- ▶ Feedback form – *please complete*
  - <http://www.clu-in.org/conf/itrc/ISM/feedback.cfm>

Need confirmation of your participation today?

Fill out the feedback form and check box for confirmation email.

Links to additional resources:

<http://www.clu-in.org/conf/itrc/ISM/resource.cfm>

Your feedback is important – please fill out the form at:

<http://www.cluin.org/conf/itrc/ISM/feedback.cfm>

### The benefits that ITRC offers to state regulators and technology developers, vendors, and consultants include:

- ✓ Helping regulators build their knowledge base and raise their confidence about new environmental technologies
- ✓ Helping regulators save time and money when evaluating environmental technologies
- ✓ Guiding technology developers in the collection of performance data to satisfy the requirements of multiple states
- ✓ Helping technology vendors avoid the time and expense of conducting duplicative and costly demonstrations
- ✓ Providing a reliable network among members of the environmental community to focus on innovative environmental technologies

### How you can get involved with ITRC:

- ✓ Join an ITRC Team – with just 10% of your time you can have a positive impact on the regulatory process and acceptance of innovative technologies and approaches
- ✓ Sponsor ITRC's technical team and other activities
- ✓ Use ITRC products and attend training courses
- ✓ Submit proposals for new technical teams and projects