

Soil Sampling and Decision Making Using Incremental Sampling Methodology (ISM)



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► What is the ITRC?

- Introduction to Incremental Sampling Methodology (ISM)
 - Systematic planning
 - Field sample collection
 - Laboratory processing and analysis
- ► Where to get more information



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and approaches







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> New Technology

Better Environmental Protection

> Environmental Regulations

translates

good science

into better

decision

making



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ITRC Purpose

To advance innovative environmental decision making



ITRC Mission





Develop information resources and help break down barriers to the acceptance and use of technically sound innovative solutions to environmental challenges through an active network of diverse professionals

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Conduct

Training

and

Implement **Solutions**



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9



Link to ITRC Document on Incremental Sampling:

http://www.itrcweb.org/ism-1/

Next Internet Course on ISM:

September 20, 2016 (Tuesday) 1:00 PM - 3:15 PM EASTERN TIME

www.itrcweb.org/Training

Soil Samples – Desirable Properties?



Representative Data:

- Accurate
- Reproducible
- Defensible



....but how do we get it? Incremental Sampling Methodology (ISM)might be the answer.....

Are Your Samples Representative?

How fully do you plan your sampling event?

- Are you confident in your sample results?
- How representative are your samples?

- Do you understand the distribution?
- How reproducible are your data?



What Does the Sample Represent?







¹³ What Do These Environmental Criteria Have In Common?



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- Soil screening levels
- Regional screening levels
- Site-specific cleanup levels
- Exposure point concentrations

Uncertainty Sources





Instrument analysis

- Sample preparation
- Laboratory sub-sampling
- Field sample collection







Uncertainty Sources





- Instrument analysis
- Sample preparation
- Laboratory sub-sampling
- Field sample collection



¹⁶ What is Incremental Sampling Methodology (ISM)?



ISM Objective: To obtain a single sample for analysis that has the mean analyte concentration representative of the decision unit

- Structured composite sampling and processing protocol
- Reduces data variability
- Provides a reasonably unbiased estimate of mean contaminant concentrations in a volume of soil targeted for sampling

Decision Unit (DU): the smallest volume of soil (or other media) for which a decision will be made based upon ISM sampling

ISM Document and Training Roadmap

17

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¹⁸ Our ITRC Solution: *ITRC ISM-1 Technical and Regulatory Guidance Document*



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

sivi-1/executive_summary.num				
Help				
	✓ Search ▼ More ≫			
Incremental Sampling	. x	🚵 🔻 🔂 👻 🖃		
Incremental Sampling Methodology				
ITRC	EXECUTIVE SUMMARY	Printer Friendly Version		
ITRC ISM Public Pages Incremental Sampling Metholodolgy Homepage 1.0 Introduction	Incremental sampling methodology (ISM) is a structured composite sampling and procet provides a reasonably unbiased estimate of mean contaminant concentrations in an are provides representative samples of specific soil areas/volumes defined as decision units (typically 30–100 increments) that are combined, processed, and subsampled according	ssing protocol that reduces data variability and avolume of soil targeted for sampling. ISM s (DUs) by collecting numerous increments of soil j to specific protocols.		
2.0 Nature of Soil Sampling and Incremental Sampling Principles	ISM is increasingly being used in the environmental field for sampling contaminants in so density afforded by collecting many increments, together with the disciplined processing most cases yields more consistent and reproducible results than those obtained by mor	oil. Proponents have found that the sampling and subsampling of the combined increments, in e traditional (i.e., discrete) sampling approaches.		
3.0 Systematic Planning and Decision Unit Designation 4.0 Statistical Sampling Designs for ISM	In 2009 the ITRC established a technical team to evaluate ISM for sampling soils at haza properties. The ISM Team convened national experts in the fields such as toxicology, risk efforts of the ISM Team included a statistical analysis of ISM performance, considerations the suitability of ISM to various contamination expension and contaminant categories and the suitability.	irdous waste sites and potentially contaminated cassessment, statistics, and soil sampling. Key s of unique laboratory processes and procedures, d identifying the strengths and weaknesses of ISM		
5.0 Field Implementation, Sample Collection and Processing	A key feature of the ISM Team's effort was emphasizing the need to integrate systematic planning or any soil sampling approach. SM requires the integration of quantitative soil sampling objectives with the conceptual site model. Other topics of interest to the ISM Team included the theoretical underpinnings of ISM, the planning and sampling design process for implementing ISM, and potential regulatory challenges to use of ISM, particularly the requirements for calculating upper confidence limits specified in some regulatory jurisdictions. The processes and equipment described here are the best available at the time this document was written. As technology advances and new equipment, instrumentation, and processes are developed, they may be included in future ISM implementations provided they meet the data and measurement quality objectives for the site to be characterized. Overall, members of the ISM Team hore found that ISM provides reliable, reproducible sampling results and leads to better, more defensible decisions than have typically been achieved with many traditional sampling approaches. Such improvements result from the inherent attributes of ISM and the details of its implementation, including a clearer connection between sampling objectives and sampling approaches. Such improvements result from the IsM provide with soil sampling approaches of discretions and sampling approaches. Clear and sampling approaches a distributes of ISM and the details of its implementation, including a clearer connection between sampling objectives and sampling approaches and overcome the sampling proxis as advances and sampling approaches. Such improvements result in the IsM provide that are eccomered of the advantages and improvements and sampling approaches. Such and not just in the IsManni sampling approaches. Clearer and sampling objectives and sampling approaches and better more defined works and actionation and the sampling approaches. Clearer and sampling objectives approvide to the alloss to the sampline cleare sam			
6.0 Laboratory Sample Processing and Analysis				
7.0 Making Decisions Using ISM Data				
8.0 Regulatory Concern with Incremental Sampling Metholodolgy				
9.0 Case Study Summaries				
10. Stakeholder and Tribal Input				
11.0 References				
The References				
Appendix A - Statistical Simulations	advantages and improvements innerent in ISM over traditional methods, ISM is finding in-	area and and an area and a and a a and a a a a a a a a a a		

- Fundamental understanding of how and why ISM works
- Detailed instructions for design and implementation
- Addresses potential regulatory concerns
- Provides case studies and simulations

What is Incremental Sampling Methodology (ISM)?





Advantages and Limitations of ISM



Advantages of ISM	Effect
Improved spatial coverage (increments x replicates)	Sample includes high and low concentrations in proper proportions
Higher Sample Mass	 Reduces errors associated with sample processing and analysis
Optimized processing	 Representative subsamples for analysis
Fewer non-detects	 Simplifies statistical analysis
More consistent data	More confident decision

Limitations of ISM	Effect
Small number of replicates	 Limits Upper Confidence Limit calculation methods
No spatial resolution within Decision Unit	 Limits remediation options within Decision Unit Limits multivariate comparisons
Assessing Acute Toxicity	 Decision Unit has to be very small

ISM – What's In It For YOU?



- Fewer analyses but a more representative sample
- ► High quality data leads to a more confident decision
- Potential for cost savings

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²³ ISM Part 1 – Principles, Systematic Planning, and Statistical Design



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²⁴ Principles Learning Objectives



Learn how to use basic principles to improve planning, implementation and decision-making:

- Soil heterogeneity at 2 spatial scales makes it difficult to correctly interpret data results
 - Those spatial scales are micro-scale and short-scale
 - Heterogeneity at these scales can cause data variability → costly decision errors
- Micro-scale heterogeneity is managed by increasing sample mass and *improving lab sample processing* (required by ISM)
- Short-scale spatial heterogeneity is managed by the field incremental sampling of ISM

ITRC, ISM-1, Sections 2 and 5.3.1

²⁵ How Soil Heterogeneity Can Cause Decision Errors: Navigation Pane



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- Heterogeneity: the condition of being non-uniform
- The heterogeneous nature of contaminants in soils increases the chances of decision error

ITRC, ISM-1, Section 2.1

²⁶ Soil is a Complex Particulate Material





► All soil is heterogeneous in composition

Typical mixing/stirring cannot make soil uniform

ITRC, ISM-1, Section 2.2

²⁷ Micro-Scale Variation in a Homogeneous-Looking Soil





A sandy soil, showing variation in particulate size and mineral content (10X magnification)

Soil Particle Composition





- Many contaminants adhere to the surfaces of certain minerals
- Organic carbon is composed of complex molecules that act as molecular sponges

ITRC, ISM-1, Section 2.2

"Sticky" Minerals





Electron microscope photograph of smectite clay – magnification 23,500

- Contaminant molecules/atoms "stick" well to certain particles
- Smallest particles usually the stickiest
 - Clays (see photo)
 - Iron (hydr)oxides
- Stickiness mechanisms
 - (-) and (+) charges
 - Surface area

ITRC, ISM-1, Section 2.2.1.1

Photo credit: USGS, 2006

Particles with High Loadings are Called "Nuggets"



 Contaminants adsorbed to distinct particles form "nuggets" of high concentration

30

"the iron in a cubic yard of soil [1-1.5 tons] is capable of adsorbing 0.5 to 5 lbs of soluble metals ...or organics" (Vance 1994). Arsenic (whitish color) sorbed to iron hydroxide particles



Photo courtesy of Roger Brewer, HDOH

ITRC, ISM-1, Section 2.2 hyperlinks

Key Point: Contaminants Often Exist or Behave as Particles





Photo courtesy of Alan Hewitt (USACE)

Tiny chunks of pure TNT-based explosive compound isolated from a soil sample

Particulates in Solid Matrices Create "Micro-Heterogeneity"



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- Micro-heterogeneity" is non-uniformity within the sample jar
- Important because contamination is heterogeneous at the same spatial scale as sample analysis

ITRC, ISM-1, Section 2.5.2

³³ Micro-Heterogeneity Makes Contamination Hard to "Read"





- Micro-heterogeneity interferes with interpreting analytical results
- If contaminant distribution is not uniform in the sample jar, how sure that analytical data represent the contents of the jar, much less the field?
 - Huge mismatch between scale of decision-making and scale of sample analysis

ITRC, ISM-1, Section 2.4

³⁴ Metals Analysis on 1 Gram of Soil Guides Decisions on Tons



VS.



Photo credits: Roger Brewer, HDOH



Short-Scale Field Heterogeneity: Co-located Samples

- Shortest spatial scale in the field measured by "co-located samples" (inches to a few feet apart)
- Samples anticipated to be "equivalent," but often give very different results
- Chance governs exact location where soil is scooped
 - Therefore, *chance* can determine decision outcome!
- ISM addresses the problems of both micro- and short-scale heterogeneity

35



1 ft



Arsenic in residential yard transect (mg/kg)



⁵Long-Scale Heterogeneity is Generally at the Scale of Decision-Making



Figure credit: Roger Brewer, HDOH

Results for an actual sampled property. Green circles denote concentrations below the action level; red circles are above the action level.
Heterogeneity Causes Sampling Errors

37



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- Sampling error occurs when samples fail to mirror (represent) the original targeted population
- Need the concept of "sample support" (the physical dimensions and mass of the sample)

ITRC, ISM-1, Section 2.3.2, 2.4.1.1 and 2.2 hyperlinks

Concentration is a Function of Sample Support and Nugget Mass



Common assumption

38

The amount of soil analyzed makes no difference to what results are obtained.



Concentration (mg/kg) = contaminant mass (mg) / the soil mass (kg)

Assumption wrong for solids

Can have the **same** contaminant nugget mass (blue), BUT in different **sample** masses (white)...



...get different concentration results

³⁹Smaller Sample Supports More Prone to Sampling Error than Larger Ones





Illustration of sampling error: For the blue and green samples, the proportion of nuggets in the samples do not represent the nugget proportion of the population (the large container)

⁴⁰ Change the Sample Support and Change the Concentration



Concentration (mg/kg) = contaminant mass (mg) / the soil mass (kg)

Arsenic <u>mass</u> of 5 ng in a sample support of 1 µg of other soil minerals: arsenic <u>conc</u> = 5000 mg/kg

Analyze an As-Fe-OH grain . by itself and arsenic <u>conc</u> might be 100,000 mg/kg (10%) or more. Arsenic (As) sorbed to iron hydroxide (Fe-OH) mineral grains



Figure courtesy of Roger Brewer

ISM Addresses Sample Support



Same As-Fe-OH grains in 1 gram of other minerals: arsenic conc = 0.005 mg/kg



Photo credit: Deana Crumbling

A lack of control over sample support during lab subsampling and in the field is a primary cause of sampling error and data variability.

ISM explicitly manages sample support!

ITRC, ISM-1, Sections 5 and 6

Ways to Reduce Sampling Error When Sampling a Jar



- ISM stresses the importance of sample support and techniques to reduce sampling error
 - Reduce particle size (grinding)

42

- Increase sample support (i.e., extract a larger analytical sample mass)
- Take many increments to make up the analytical subsample ("incremental subsampling")
- Use equipment like rotary splitters —



ITRC, ISM-1, Table 3-1 and 6.2.2.5 to 6.2.2.7

Reducing Short-scale Sampling Error

- Goal is to get THE concentration for a target soil volume, so...
 - IDEAL: analyze whole volume as a single sample
 - PRACTICAL: Increase sample support and sampling coverage by taking many small increments across the area and pooling them

This is what ISM does

#2 496 #1 30 116 Set of co-located

1 ft

49

samples for uranium



Sampling Error Causes Data Variability





Sampling errors contribute to data variability

ITRC, ISM-1, Sections 2.4.1.3

Study Data for Pb: 5 Laboratory Replicate Subsamples from Same Jar





Lab Replicate Number

Same Soil Sample After Grinding

46



~5000 ppm

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Sample Support Influences Statistical Distributions





Concepts Underlying ISM: Avoiding Decision Error



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- Decision Error: a decision that would have been made differently if the true condition were known
- Can occur when conclusions are based on data that were significantly influenced by heterogeneity

ITRC, ISM-1, Section 2.4.1.3 and 2.4.2

Skewed Data Distributions Promote Decision Errors



Suppose 3 is an action level. The likelihood of single data points exceeding 3 depends on the sample support. True mean of large batch = 1.92Density Function f(x) 100 0 g q 88 5 Am-241 Activity Result, nCi/g(x)



- Pay attention to QC results in the data package!
 - Suspect sampling error due to micro-scale withinsample heterogeneity when
 - Lab duplicates do not "match"
 - Matrix spikes/matrix spike duplicates do not "match"
 - Suspect sampling error due to short-scale betweensample heterogeneity when
 - Co-located samples do not "match"



- Be wary of making decisions based on a single data point
 - Especially when traditional sample collection and handling is used
- ► Use ISM in field and lab!

Ensure ISM work plans spell out procedures to detect and control sampling error



- Inadequate management of soil heterogeneity produces highly variable data sets
- The "maximum concentration" notion is meaningless
- Chance data variability can be misinterpreted to represent the "true" condition for large soil volumes
- Misinterpreting data, especially single data points, can lead to costly decision errors
- The "nuts and bolts" of managing sampling error in the field and lab will be presented in Part 2



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⁵⁵ Systematic Planning Learning Objectives



Learn how to:

- Conduct systematic planning steps important to ISM
 - Conceptual Site Model (CSM)
 - Risk pathways and contaminants of concern
 - Project objectives (Sampling and Data Quality Objectives (DQOs))
- Determine Decision Units (DUs)
 - Information used to develop DUs
 - Why DUs are important
 - Types of DUs
 - Real world examples (i.e., case studies)



ITRC, ISM-1, Section 3

⁵⁶ No Data Quality Objective (DQO)/Decision Units? Bad Data!



- Decision Units (DUs) The smallest volume of soil for which a decision will be made based on ISM sampling
- Designating DUs arguably most important aspect of ISM from a regulatory perspective
 - Selection of DUs determines
 - Where samples are being collected
 - How many
 - DU selection determines whether the data are able to satisfy the project objectives, both sampling objectives and data quality objectives

⁵⁷ Systematic Planning and Implementation



- Develop Conceptual Site Model (CSM)
- Identify contaminants and project objectives
- Identify data needed and how it will be used
- Define Decision Units (DUs)

Key Step of ISM

- Develop decision statements
- Collect samples to characterize DUs
- Evaluate data

ITRC, ISM-1, Table 3-1

Conceptual Site Model (CSM)





ITRC, ISM-1, Figure 3-2

Data/Information Needs

59



- What receptors and pathways are being evaluated?
- What are your sampling objectives?
- Are there multiple sampling objectives that must be met?
- What is the scale of decision making?
- What population parameter is of interest?

The key is the volume over which the mean should be estimated.

Example Sampling Objectives



- Estimate the mean concentration of contaminants in a pre-determined volume of soil (i.e., DU)
- Delineate the extent of contamination above screening levels
- Estimate the potential risk to receptors posed by the soil contamination
- Evaluate background metals concentrations in soil
- Confirmation sampling following remediation

Designating Decision Units (DUs)



- Information used to develop DUs
- Why DUs are so important
- Types of DUs
- Examples

61



Stakeholder Agreement

Decision Units (DUs)



The <u>volume</u> of soil where samples are to be collected and decisions made based on the resulting data.

Source Areas

62











Size, shape and type of DU are an outcome of systematic planning and depend on site specific data quality objectives.

ITRC, ISM-1, Section 3.3

Why ISM Is Important





⁶⁴ Traditional Site Investigation Approach





Potential Concerns

- Inadequate number of sample points to define outward boundaries
- High risk of False Negatives and False Positives
- Confusion over single point "hot spots"
- Cost of 30 analyses
- Sample points should be randomly located for estimation of exposure point concentration (EPC)

ISM Approach (Option 1)

65





Advantages

- More representative
- Risk evaluation objective identified up front
- Increments randomly and evenly spaced to minimize size of hot spot missed
- Quick and cheap if minimal contamination suspected

Disadvantages

 Additional sampling required if DU fails

ISM Approach (Option 2)





Advantages

- Addresses both source area and perimeter as well as directional variability if an exceedance is found
- Best approach to minimize additional sampling
- Will minimize remediation volumes if DU exceeds screening level
- If increments are collected using cores, vertical delineation is easily done with stacked DUs

⁶⁷ Suspected Lead Paint and Pesticides Around House and in Yard





Do lead or pesticides exceed action levels around the house or in the yard?

⁶⁸ Former Pesticide Mixing Area (0.5 acres)





Suspected heavy contamination with arsenic, dioxins (from PCP) and leachable pesticides

⁶⁹ Source Area and Exposure Area DU Designation





Primary objective is to delineate the source area and the extent of contamination.

Former Pesticide Mixing Area

70





Source Area DUs: Heavy contamination + leaching

Exposure Area DUs: Maximum 5,000 ft²

⁷¹ Former Power Plant Proposed Community Center





Primary objective is to identify and delineate source area and extent of contamination that exceeds action levels. **100'**

⁷² Former Power Plant Decision Unit Designation





*Assuming 3' depth

100**′**
⁷³ Really Big Decision Units (DU)! (400-acre former sugarcane field)



Source Area DU (investigated separately)

Initial Screening DU

- Residual pesticide levels?
- OK for residential development?

Lot-Scale Resolution

- Hypothetical lots
- 5,000 ft² Exposure Area
- May also be required



Primary objective is to determine if property can be developed for residential use.

⁷⁴ Really Small Decision Units??? What about the Sandbox!?





- Yard-size DUs are most often appropriate
 - If acute hazards or intense exposure are being evaluated, smaller DUs may be necessary
 - Not typical
 - Investigate known or suspected source areas separately
 - Remember: As sampling objectives change, so must the sampling design

⁷⁵ Why DUs (and ISM) are Important (Discrete Sample Data)







PCB sample aliquot = 30 grams (one spoonful of soil)

100**′**

⁷⁶ Why DUs (and ISM) are Important (ISM Sample Data)







Why ISM Is Important

77





⁷⁸ Why Discrete Samples Miss Contamination in the Field



Area average PASSESArea average FAILS(Isolated False Positives)(Majority False Negatives)

Area average FAILS (Isolated False Negatives)



Excavation Decision Units



Floor and sides tested as separate DUs



X - Increment Sampling Locations

ITRC, ISM-1, Section 3.3.6 and Figure 3-11

Stockpile Decision Units





ITRC, ISM-1, Section 3.3.5 and Figure 3-10

Subsurface Decision Units





ITRC, ISM-1, Section 3.3.4 and Figure 3-8

ISM Case Study – Florida Golf Course





82

Decision Unit (DU) Highlights

83



Determining DU size and location

- Use all available information
- Determine Data Quality Objectives
- Establish DUs with risk assessment and remedial goals in mind from the start
- Many random increments required (30 to 50+)
 - Capture the effects of heterogeneity
 - Characterize a DU

Decision Unit Highlights (continued)

ISM samples

84

- More efficient and cost effective method
- Minimize the chance of missing hot spots in the DU

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- Represent larger volumes, i.e., DUs
- Tight grids of screening data can be useful to locate suspected source areas for better DU designation, if needed

Summary: Systematic Planning



Conduct Systematic Planning

85

- It's important to develop a CSM before beginning a sampling design
- Be sure that your sampling design will achieve your sampling objectives
- Be certain that your sampling design will provide the kind of data necessary to fulfill the sampling objectives

Decision Unit designation

- Make sure that all site information has been used to develop your DUs
- Be sure that your scale of decision making aligns with your sampling objectives

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⁸⁷ Statistical Design Learning Objectives



Learn how to

- Answer common questions about ISM related to
 - Sampling design
 - Data analysis
- Expand your understanding of
 - Statistical theory
 - Simulation studies conducted by the ITRC ISM Team



95UCL = 95% Upper Confidence Limit of the mean

88

Questions – Sampling Design



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Sections 4.4.3.3 and 7.2.4

89

⁹⁰ **1. Does a single ISM provide a reasonable estimate of the mean?**

Answer:

- It depends how much error we are willing to accept. Under some circumstances, one ISM sample can substantially underestimate the actual mean concentration.
- ► Why would someone collect just 1 ISM?
 - UCL not required by regulator
 - Save time and expense
 - Assumption that more sampling wouldn't change the decision. For example
 - Variance among individual increments is low
 - Mean of DU is far above or below an action level

1(b). How "badly" might I underestimate the mean?



Underestimate of Mean

C۷	Frequency	Magnitude	True Mean	Estimate
1	33%	10%	400 ppm	≤ 360 ppm
2	33%	20%	400 ppm	≤ 320 ppm
3	25%	30 - 60%	400 ppm	160 - 280 ppm

*Coefficient of variation (CV) = St Dev / mean

ITRC, ISM-1, Section 4.2.1, Figure 4-2

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2. Can a 95UCL be calculated?

Answer:

92

- Yes, even with as few as 3 ISM samples (replicates).
- ▶ Need at least 3 replicates $(r \ge 3)$
- Supported by theory and statistical simulations
- Fewer methods are available than we are used to with discrete sampling:
 - Chebyshev
 - Student's-t
- Each ISM result provides an estimate of the mean ("x-bar")

Parameter estimates are calculated directly from ISM data

2(b). How do I choose a UCL method?

- Consider performance measures (informed by simulation study)
 - Coverage (probability UCL > mean)
 - Magnitude of difference between UCL and mean



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- Recognize the key to performance is <u>variability</u>
 - Distribution of discretes ≠ Distribution of ISM results
 - CV = standard deviation / mean = 3.0 refers to distribution of discretes
 - With only ISM results, assumptions about variability are very uncertain

93



- ► ISM distribution variance is smaller
- ISM distribution shape becomes more non-normal with increasing CV of discrete distribution

ITRC, ISM-1, Figure 4-3

95



	Dispersion Among Individual Increments			
UCL Method	Low (CV <1.5 or GSD <3)	Medium (1.5 < CV < 3 or 3 < GSD < 4.5)	High (CV >3 or GSD >4.5)	
Student's-t	Yes	No	No	
Chebyshev	Yes	Yes	Maybe	

CV based on underlying distribution of increments

- Both methods provide desired 95% coverage when variability is low
- Chebyshev has more consistent 95% coverage for medium and high variability
- Increasing r (>3) and n (>30) provides marginal improvement in coverage for Chebyshev, but no improvement for Student's-t

ITRC, ISM-1, Table 4-4, Sections 4.3; Appendix A

How much higher is Chebyshev?

- Chebyshev will tend to yield 10-45% higher UCLs than Student's-t depending on the CV of 3 replicates
- Example: Student's-t = 100 ppm, Chebyshev = 110 145 ppm

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96

⁹⁷ 2(c). Can I use ProUCL to calculate the 95UCL?



Answer:

- EPA is updating ProUCL to include an ISM module. Visit the ITRC website for additional tools.
- ProUCL was originally designed to work with discrete sample data, but is being updated to include an ISM module.
- Only Chebyshev and Student's-t UCLs are implemented for ISM datasets.
- ITRC guidance has calculator tools that work for ISM data (see ISM-1, Sections 4.2.2 at http://www.itrcweb.org/ism-1/4_2_2_UCL_Calculation_Method.html).

ITRC, ISM-1, Sections 4.2.2

Bias and Precision

98





Accuracy reflects both bias and precision (reproducibility)

These are metrics of the performance on average. They can only be assessed through simulation of many hypothetical sampling events – not by the results of any single ISM sampling event

ITRC, ISM-1, Section 4.3.1, Figure 4-6 and Appendix E

Components of the RSD



Field

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

Laboratory

- Lab processing
- Subsampling
- Extraction
- Digestion
- Analysis
- Simulations used to explore alternative sampling designs did not attempt to isolate sources of error
- In Day 2 of ISM training, methods will be presented for quantifying and reducing relative errors associated with field and lab practices that contribute to RSD

¹⁰⁰ **2(d). What can we infer from the RSD?**

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Answer:

- Without data to quantify sources of lab and field error that contribute to RSD, it is difficult to be conclusive. We expect that low RSD is an indication that steps to reduce error are successful. The 95UCL coverage does not depend on the RSD.
- RSD is the ratio of statistics calculated from ISM replicates
 - RSD = SD / mean
- 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

ITRC, ISM-1, Section 4.3.4.4

¹⁰¹3. Is there a preferred ISM sampling design?





¹⁰²3. Is there a preferred ISM sampling design?





Simple Random



Systematic

ITRC, ISM-1, Section 4.3.4.2

•	•	•	•	•
•	•	•	•	•
•	•	•	•	•
•	•	•	e	•
•	•	•	•	•
•		•	•	•

Random within Grid

• ▲	• ▲	• ▲	• * *	• * *
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• * •	• *	• * •	• * •	• * •
*	*	• *	*	• *

Systematic (3 replicates)

¹⁰³3. Is there a preferred ISM sampling design (continued)?



Answer:

• Each random sampling design yields unbiased estimates of the mean and is an acceptable approach in most situations.

Systematic random sampling is most often used because it is the easiest to implement



ITRC, ISM-1, Section 4.3.4.2

3(b). How many increments?



Answer:

- n = 30: generally, 30 increments per ISM sample provide good results. Lower numbers are discouraged and higher numbers provide diminishing improvement in statistics.
- As the number of increments increases:
 - spatial coverage improves (greater sample density)
 - lower variability in ISM results (smaller standard deviation)
 - 95UCL will tend to be closer to the mean
- Size of DU can be a consideration large DUs may require more increments

10 20 **30** 40 50 60 70 80 90 100

ITRC, ISM-1, Sections 4.3.4.1 and 5.3.1

3(c). How many replicates?

Answer:

• r =3 : for most DUs, three replicates is sufficient.

- Minimum number to calculate standard deviation (and 95UCL) of ISM results
- More replicates will produce a 95UCL closer to the actual mean, but may not be cost-effective unless the result is near the action level







A. n = 30, r = 1
B. n = 90, r = 1
C. n = 30, r = 3 (so 30 x 3 = 90)



Scenario	Spatial Coverage	Analysis Cost	Estimate of Mean	Estimate of Variance
А	Low	Low	Yes	No
В	3 x A	А	Yes	No
С	3 x A	3 x A	Yes	Yes

¹⁰⁷4. Can I extrapolate results across DUs?



- Unsampled DU extrapolate estimate of mean
- DU with 1 ISM extrapolate estimate of variability
 - Standard deviation (SD)
 - Coefficient of variation (CV)







ITRC, ISM-1, Section 4.4.2

4(b). Extrapolation of the Mean

<u>Answer:</u>

• You are assuming that the mean concentration in the unsampled DU(s) is the same as in the sampled DU.

$DU-1 \stackrel{?}{=} DU-2$



▶ DU-1:

- Mean = 100
- SD = ?
- DU-2:
 - Extrapolation: Mean = 100



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¹⁰⁹ **4(c). Extrapolation of the Variance**

<u>Answer:</u>

• You are assuming that the heterogeneity in contaminant concentrations is similar in all of the DUs.

$DU-1 \stackrel{?}{=} DU-2$



► DU-1:

- Mean = 100
- SD = 50
- CV = SD/mean = 50/100 = 0.5

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CAUTION

- DU-2:
 - Mean = 400
 - Extrapolation:
 - CV = 0.5 = x / 400
 - therefore
 - SD = x = 200

ITRC, ISM-1, Section 4.4.2

¹¹⁰5. Can background and site ISM data be compared?



Answer:

• Yes, but statistical tools for comparison are limited.



Each data sets consists of ISM samples, preferably generated with similar sampling designs

ITRC, ISM-1, Section 4.4.3.3

¹¹¹5. Can background and site ISM data be compared?



Answer:

• Yes, but statistical tools for comparison are limited.

Background





- Equal central tendency (mean, median) ?
- Equal upper tails ?
- Hypothesis testing is limited to parametric tests of the mean:
 - Assume distribution shape
 - Use estimates of mean, SD, and number of replicates
- Cannot test upper tails with ISM data

ITRC, ISM-1, Section 4.4.3.3

5. Example Background Comparison

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ITRC, ISM-1, Section 7.2.4, Figure 7-1

Recap of Learning Objectives



113



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¹¹⁴ Summary: Statistical Design



- Mean or 95UCL from ISM data may be used to make decisions about a site
- 3 replicate samples provide adequate information to calculate a 95UCL
- Systematic random sampling is most commonly used
- About 30 increments per ISM sample is usually sufficient
- Extrapolation of the mean or variance can be very uncertain
- Comparisons between ISM data (e.g., site vs. background) are possible, with caution

¹¹⁵ ISM Part 1 – Summary Principles, Systematic Planning, and Statistical Design







¹¹⁷ ISM Part 1 Summary and Part 2 Preview





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

118

- Increment mass
- Sample mass

¹¹⁹ Sampling Designs





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

¹²⁰Florida Case Study: Decision Unit (DU) Identification

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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

121 Increment Locations



Identify increment locations in field

• Utilize similar site investigation tools





ITRC, ISM-1, Section 5.3.1

¹²²Florida Case Study: Increment Field Determination







¹²³ Sampling Tool Considerations



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



e.g., core diameter >16 mm

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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

¹²⁵ Sampling Tool Examples



Soft Surface Soil







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

Alternate Sampling Tools





¹²⁷Florida Case Study: Field Sampling





¹²⁸Florida Case Study: "Low Tech" Sampling Tools



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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

 ρ - soil or sediment density (g/cm³)

q - diameter of sample core (cm)



ITRC, ISM-1, Section 5.3.1

¹³⁰ Subsurface Decision Units (DU)



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Individual core samples *combined* to prepare an ISM sample for each DU

Subsurface Sampling Considerations



Preferred increment – entire core interval

- Core subsampling alternatives
 - 1. Core wedge
 - 2. Core slice

131



ITRC, ISM-1, Section 5.3.2

¹³² Core Wedge





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

ITRC, ISM-1, Section 5.3.2.1







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

ITRC, ISM-1, Section 5.3.2.1

¹³⁴ **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

Final ISM samples: typically 600-2,500 grams or more

- Containers, storage, shipping
- Laboratory
 - Facilities and equipment for correct processing and subsampling







136 ISM Volatile Sampling Tools



Core type sampler

Typical for VOC soil sampling per SW846 5035A



ITRC, ISM-1, Section 5.4.2

Source: Courtesy <u>www.ennovativetech.com</u>

¹³⁷ **ISM Volatile Samples – Subsurface**



- Each core represents an *increment* from the DU Layer
- Subsamples of increment collected from each core to prepare bulk sample (e.g., 5g plug every 5cm, total subsample mass 20-60g to produce bulk ISM sample)



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

138

 Note shipping restrictions/ requirements



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

¹³⁹ **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

Replicate Spacing and Collection





ITRC, ISM-1, Section 5.3.5

140

Field Replicates – Simple Example

141





¹⁴² **Replicate/Sampling Reminders**



- 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

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¹⁴³ 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



¹⁴⁴Laboratory 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
Analyte-Matrix Driven Options

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Pick the right option

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

¹⁴⁶Include Lab Processing in Project Planning





ITRC, ISM-1, Section 6.1.1

¹⁴⁷ **Define the Analytes**



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

HO-

- Semivolatile organics
- Other



¹⁴⁸ 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



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ITRC, ISM-1, Section 6.2.1

¹⁴⁹Florida Case Study: Contaminant of Concern



► Arsenic

From liquid applied pesticides



Periodic Table of Elements





¹⁵² 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



¹⁵³Florida Case Study: Air Drying Samples



► Arsenic

- High boiling arsenic species
- Volatilization loss not expected



ITRC, ISM-1, Section 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



¹⁵⁵ **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

¹⁵⁶ **Define Terms: Milling**



Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. < 75 µm)</p>



Picture from USACE-Alan Hewitt



ITRC, ISM-1, Section 6.2.2.5

¹⁵⁷Florida 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



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¹⁵⁹To 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

Not recommended Volatile, thermally labile,

- increased "availability"
- Examples

160

 Monochloro PCBs, reactive SVOCs, decane, elemental mercury

Limitations

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

ITRC, ISM-1, Section 6.2.2.5

To Mill or Not to Mill

60

If uncertain, do milled & unmilled



ITRC, ISM-1, Section 6.2.2.5

How Best to Mill

- Puck mill or ring and puck mill
 - "Stable" energetics
- Ball mill

161

- Mortar and pestle
 - Consider
 - Analytes
 - Concentration of interest
 - Mill materials
 - Particle size needed
- Example mills, other types are possible as well







¹⁶²Florida 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





¹⁶⁴ **Dry Splitting Options**



Rotary sectorial splitter



ITRC, ISM-1, Section 6.2.2.7

¹⁶⁵ Subsampling Options



2-Dimensional Japanese Slabcake



ITRC, ISM-1, Section 6.2.2.7

¹⁶⁶ Subsampling Tools



Square straight-sided scoops for dry non-cohesive soil





¹⁶⁷Florida Case Study: Choose Subsampling Process

2-D Slabcake Subsampling

- Lower cost than sectorial splitter
- More representative than "dig a spot"



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¹⁶⁸ Why Use Large Subsamples?



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Larger particles

• Produce larger errors or require larger subsamples



ITRC, ISM-1, Section 6.3.3

¹⁶⁹Florida Case Study: Nugget Effect Minimal



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



Laboratory Quality Control Measures

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- 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



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



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¹⁷² Verify Laboratory Certification



 National Environmental Laboratory Accreditation Program (NELAP)



- Non-NELAP state accreditation
- Agency-specific accreditation
 - DoD Environmental Laboratory Approval Program



¹⁷³ 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

¹⁷⁴ 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

Lab Process "Big Rocks"



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ISM Document and Training Roadmap

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¹⁷⁷Making Decisions: Learning Objectives

Learn how to:

Use ISM data to make decisions

Evaluate data

- Identifying sources of error
- Quantify error
- Interpret error
- Isolate sources of error



179 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

¹⁸⁰DM 1: Compare One ISM Result to Action Level





ITRC, ISM-1, Section 4.2.1 and Section 7.2.1
¹⁸¹DM 2: Compare <u>Average</u> ISM Result to Action Level



Level

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ITRC, ISM-1, Section 7.2.2

¹⁸²Florida Case Study: Decision Mechanism (DM) 2



Mean arsenic concentrations (mg/kg)

	Discrete n = 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

¹⁸³DM 3: Calculate 95%UCL then Compare to Action Level or Use for Risk Assessment Decision Unit

95%UCL

Action level or risk assessment

ITRC, ISM-1, Section 4.2.2 and Section 7.2.3

¹⁸⁴Florida 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
95UCL	3.0	2.0	1.8

Florida Action Level: 2.1 mg/kg





185 DM 4: Compare to Background





Comparison

ITRC, ISM-1, Section 4.4.3.3 and Section 7.2.4

186 DM 5: Combining Decision Units



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ITRC, ISM-1, Section 4.4.1 and Section 7.2.5



ITRC, ISM-1, Section 4.4.4.2 and Section 7.2.6



¹⁸⁹ **Data Evaluation Components**



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Identifying Sources of Error



Field

190

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

Laboratory

- Lab processing
- Subsampling
- Extraction
- Digestion
- Analysis

¹⁹¹ Quantifying Error



RSD = CV = standard deviation / arithmetic mean



Data includes all sources of error

Decision Unit

ITRC, ISM-1, Section 4.3.1.3 and Section 7.3

¹⁹² Interpreting Error





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

¹⁹³ Isolating Sources of Error





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

How Does ISM Cost Compare?

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Elements

194

- Planning
- Field Collection
- QA/QC Samples
- Sample Transport
- Sample Processing/Conditioning
- Lab Analysis

Overall <u>Sampling/Analysis Portion of Project</u>

A Cost Comparison Example

195



US Army Corps of Engineers Study Metals in Soil

	Per Sample Cost (\$)		Total Project Sampling/Analysis Cost (\$)		
Activity	ISM	Discrete	ISM ¹	Discrete ²	Discrete ³
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
Total	300-385	135-160	945-1,155	945-1,120	2,025-2,400

¹ Based on 3 replicate 100-increment ISM/DU

² Based on collection of 7 discrete samples/DU

³ Based on collection of 15 grab samples/DU

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

¹⁹⁶ Bottom Line on Cost Comparisons





Measuring the cost difference between ISM and discrete sampling.



Measuring the cost of making a wrong decision.

¹⁹⁷ Overview & Wrap-up



ISM:

- Specifically designed to address short comings of discrete sampling methods
- Provides a systematic, science-based approach to site investigation
- Increases data representativeness
- Provides greater confidence in decision making









Links to additional resources

- <u>http://www.clu-in.org/conf/itrc/ISM/resource.cfm</u>
- Link to ITRC Document on Incremental Sampling:

http://www.itrcweb.org/ism-1/

Next Internet Course on ISM:

September 20, 2016 (Tuesday) 1:00 PM - 3:15 PM EASTERN TIME