

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) 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

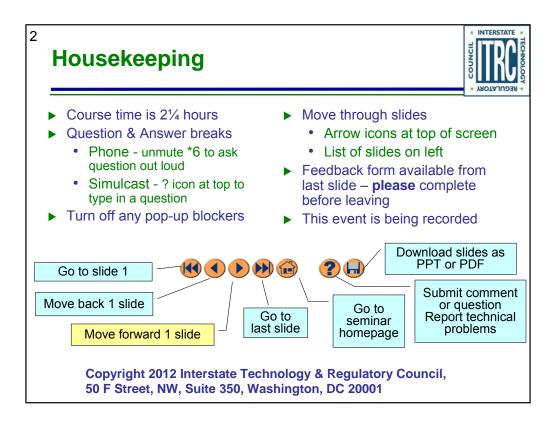
In addition this ISM training and guidance provides insight on when and how to apply ISM at a contaminated site, and will aid in developing or reviewing project documents incorporating ISM (e.g., work plans, sampling plans, reports). You will also be provided with links to additional resources related 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 (<u>http://www.itrcweb.org/ISM-1/</u>) 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

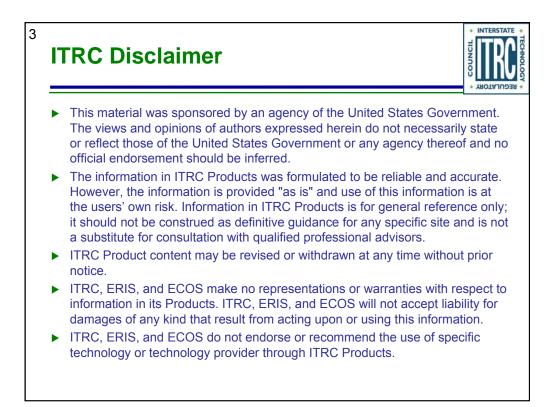
Training Co-Sponsored by: US EPA Technology Innovation and Field Services Division (TIFSD) (<u>www.clu-in.org</u>) ITRC Training Program: training@itrcweb.org; Phone: 402-201-2419



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 1st and last slides respectively. You may also advance to any slide using the numbered links that appear on the left side of your screen. The button with a house icon will take you back to main seminar page which displays our 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.



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Meet the ITRC Instructors





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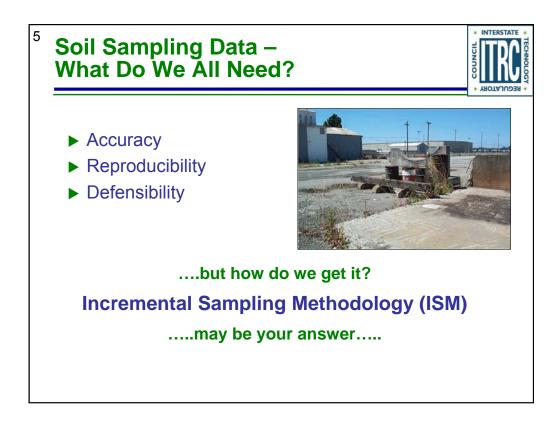
Tim Frederick USEPA Region 4 Atlanta, GA 404-562-8598 frederick.tim@epa.gov

Paul Hadley is a Senior Hazardous Substances Engineer with the California Department of Toxic Substances Control in Sacramento, California. Since 1983 he has been an environmental engineer focusing on hazardous waste site cleanups. Paul has been an active member of ITRC since the organization's inception in 1995. He has participated on or led teams on diverse subjects such as bioremediation of chlorinated solvents, risk assessment, and most recently incremental sampling, and served as the state-lead on the ITRC's first classroom and Internet-based Training courses, both on the natural attenuation of chlorinated solvents. He has authored several papers on topics related to these and other subjects addressed by various ITRC technical teams. Paul earned a bachelor's degree in biochemistry in 1977 and master's degree in civil/environmental engineering in 1983, both from the University of California in Davis. Paul is a Registered Civil Engineer in California.

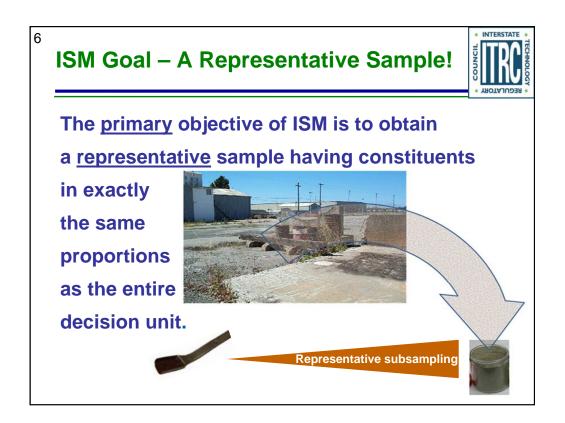
Jay Clausen is a Physical Research Scientist with the US Army Corps of Engineers, Engineer Research and Development Center, Cold Regions Research and Engineering Laboratory (CRREL) in Hanover, New Hampshire. Since 2005, Jay has conducted research on the application of the Incremental Sampling Methodology (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 the Technical Director for TestAmerica in North Canton, Ohio. 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. Mark has been presenting at conferences and workshops since 1990. 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.



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



•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

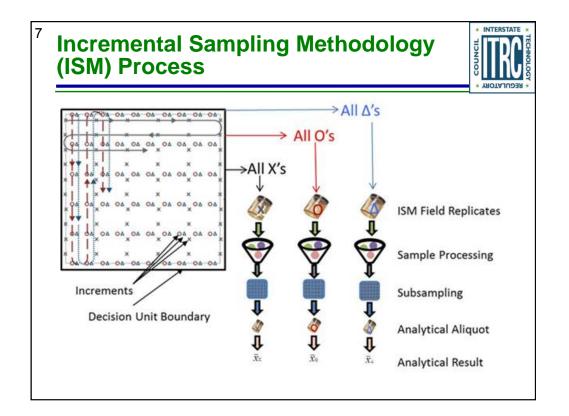
•Sampling Error is managed with ISM through the collection of 30 or more increments of correct shape and adequate mass

•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



WHAT IS ISM?

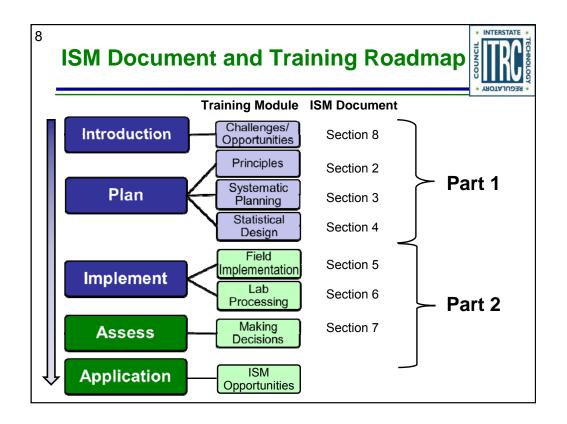
This slide shows quite succinctly all the steps involved in developing triplicate analytical results for a particular sampling unit. Triplicate results are what we are recommending for a number of reasons – largely related to addressing the heterogeneity we talked about earlier, and being able to make 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 –

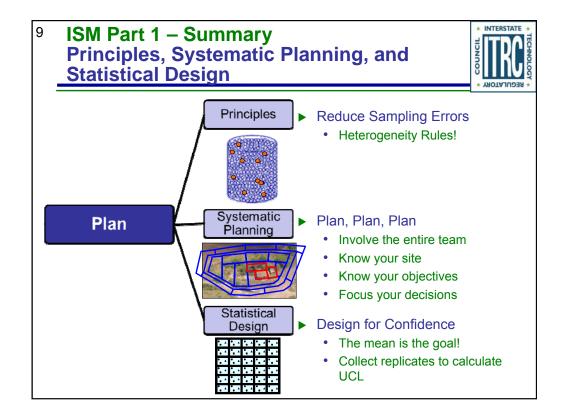
•minimum of 30 increments (20 - 60g/increment)

•3 replicates in order to get a 95 UCL

•Sample size should be ~1 kg (600 – 2,500 grams).



No associated notes.

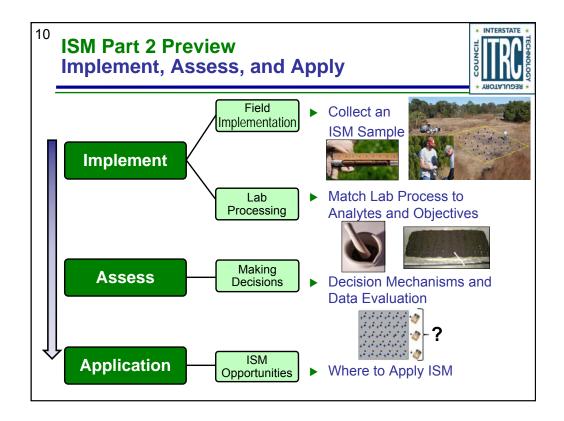


The last training included Modules on:

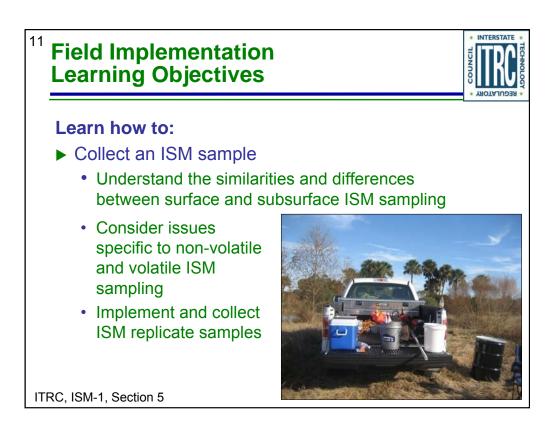
Soil and Principles - Be aware of issues related to heterogeneity and sampling errors

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.



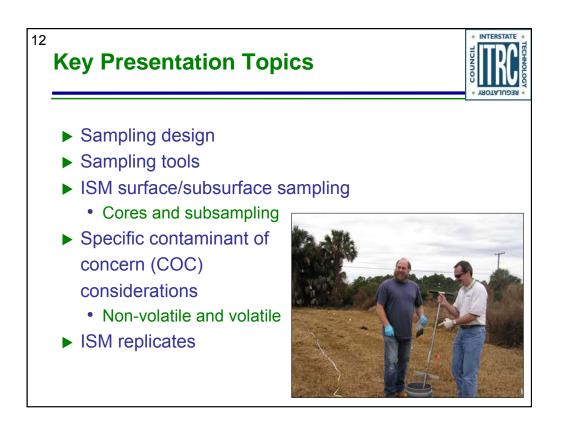
No associated notes.



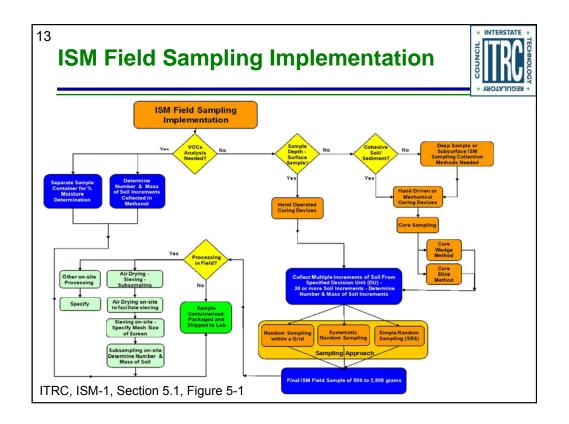
With correct up front planning and coordination, ISM field implementation can be as simple as putting dirt in a bucket (or bag).

Supplemental Information

ITRC, ISM-1, Section 5



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



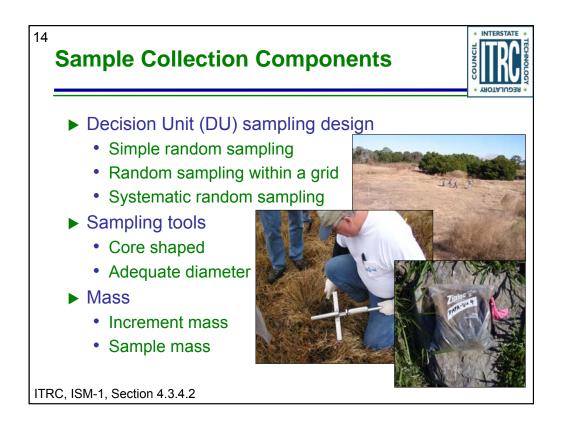
•Figure 5-1 from the technical regulatory document shown as an example only. The audience is not expected to read this flowchart (small font); provided only as an example to illustrate the multiple decisions that will need to be made and implemented.

•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

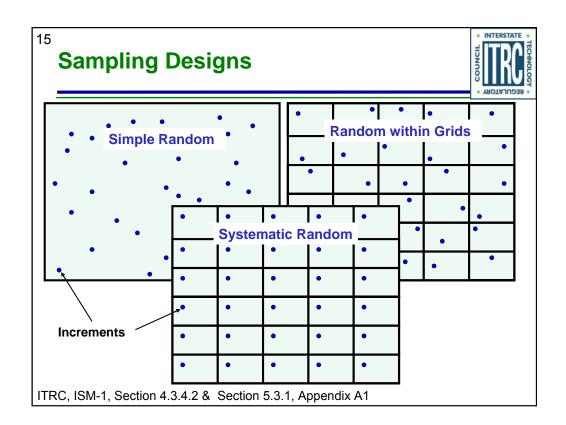


•Sampling Design - 3 basic sampling designs

•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



•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

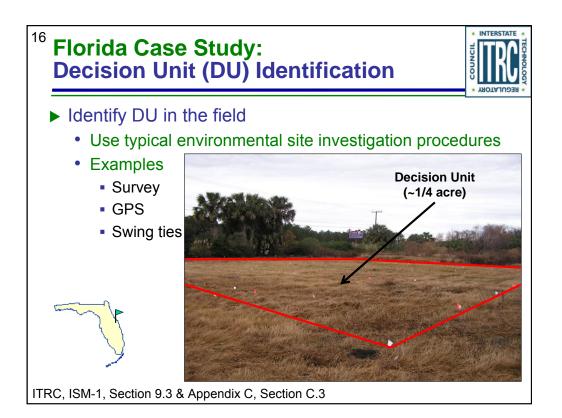
•Recommended minimum 30 increments per DU regardless of sampling design

•The systematic random sampling is <u>generally</u> 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



•DU(s) determined during systematic planning

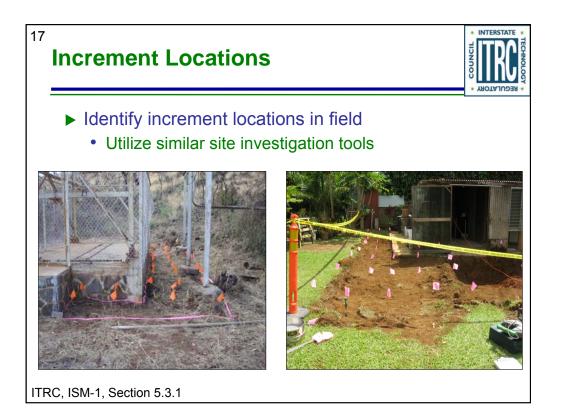
•Standard environmental site investigation procedures employed to delineate DU in the field

•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 ¹/₄ 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.3for Former Golf Course Field Demonstration of ISM



•Sampling design, number of increments, increment locations are determined during systematic planning (Day 1 training)

•Typically 30 or more increments per DU

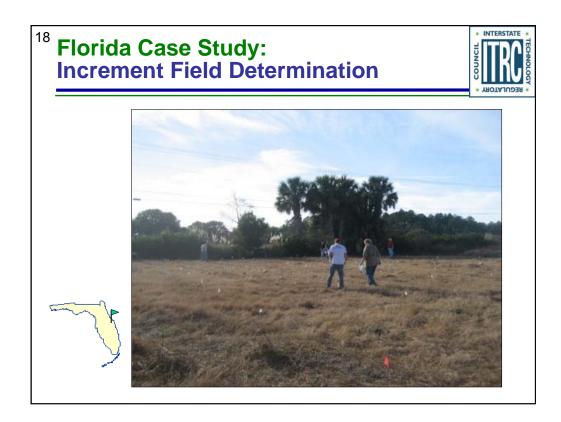
•Similar tools are utilized to identify increment locations; e.g. survey grid, GPS, sampling flags

•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

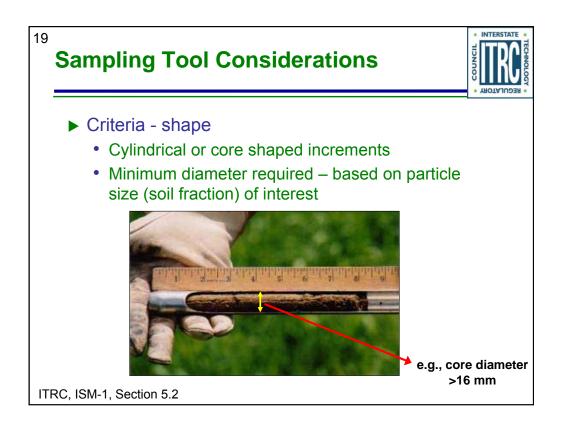
ITRC, ISM-1, Section 5.3.1



•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.



•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.

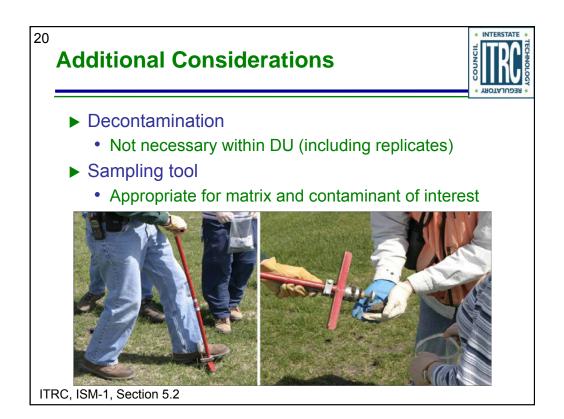
•Larger fraction of interest (or particle size) requires a larger diameter core sampler

•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



•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 (MISTTM)

•Other commercially available tools (next slides)

Supplemental Information

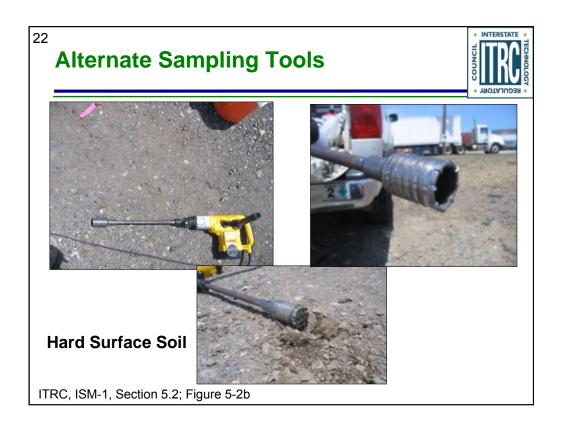
ITRC, ISM-1, Section 5.2



•Examples of commercially available tools.

•Other tools, e.g. agricultural soil samplers, may also be applicable to ISM.

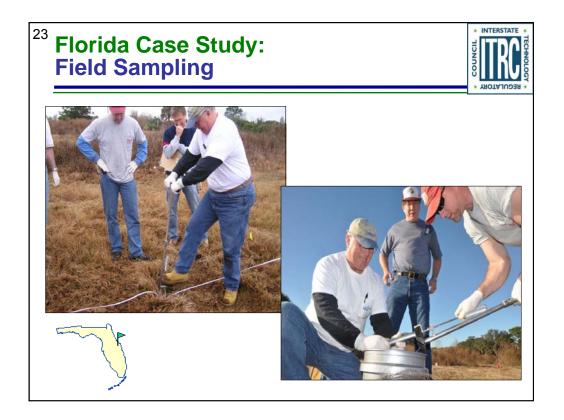
•Top: EVC Incremental Sampler (kit); Bottom: JMC Backsaver Handle and Soil Tubes



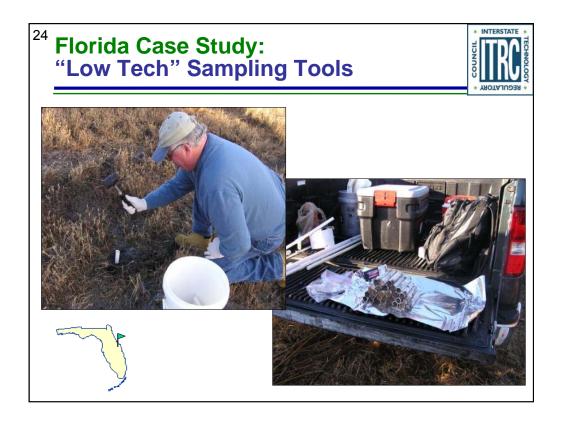
•Alternate tools may be applicable depending on matrix

Supplemental Information

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



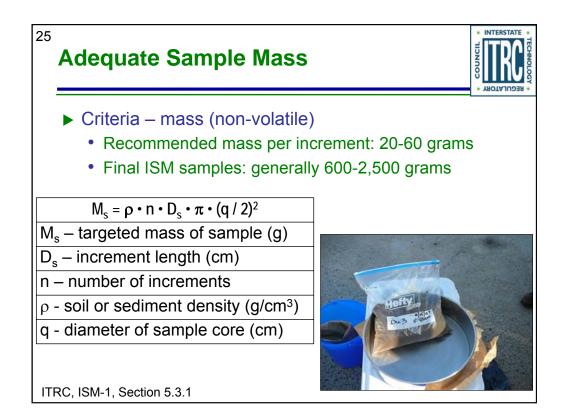
Florida Golf Course Example – Every job needs at least one supervisor.
Surface core increments collected in plastic 5 gallon bucket



Low tech alternatives, if site conditions amenable

Arsenic only COC; PVC would not interfere/contaminate analysis of COC (phthalates)

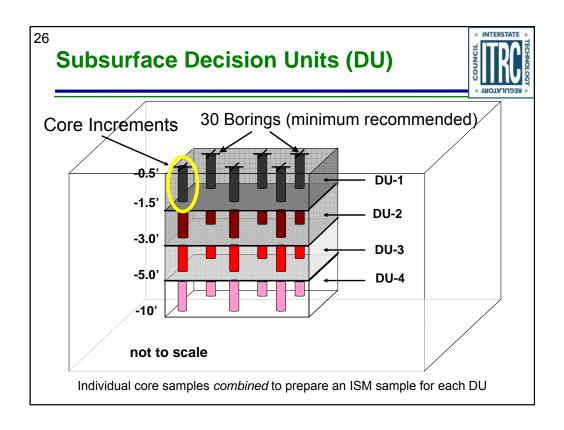
•Left pounding PVC section into ground; right PVC increments from DU, <u>all increments</u> then combined into 1 ISM sample



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

Supplemental Information

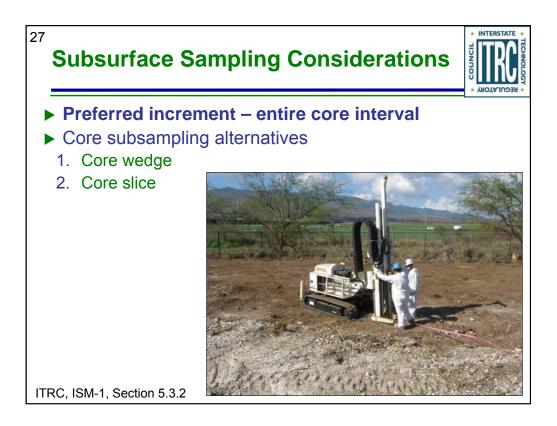
ITRC, ISM-1, Section 5.3.1 Example of bulk ISM sample ~2 kg



•Same DU determination (3 dimensional volume), sample design, number of increments, etc. as surface sampling

•Subsurface DUs presented in Module 3

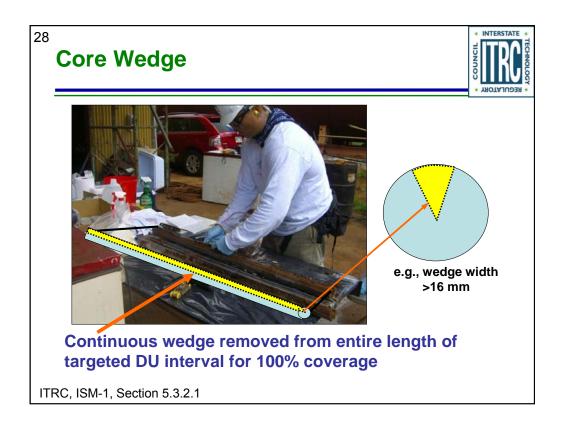
•DUs designated at depth to provide vertical resolution of contamination within DU and optimize remediation.



•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



•"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

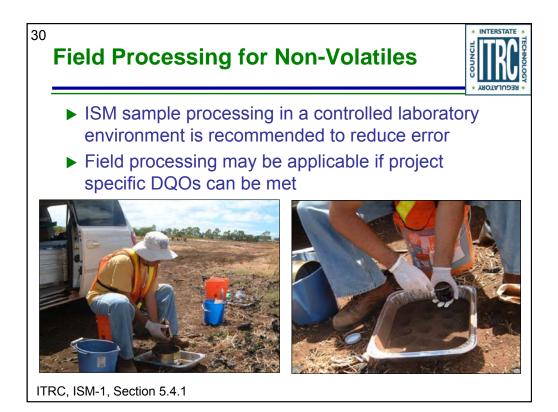


•Randomly selected core "slice" collected from core interval as increment

•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



•Each processing step introduces some amount of sampling error; sample processing in a controlled laboratory environment recommended to reduce this error

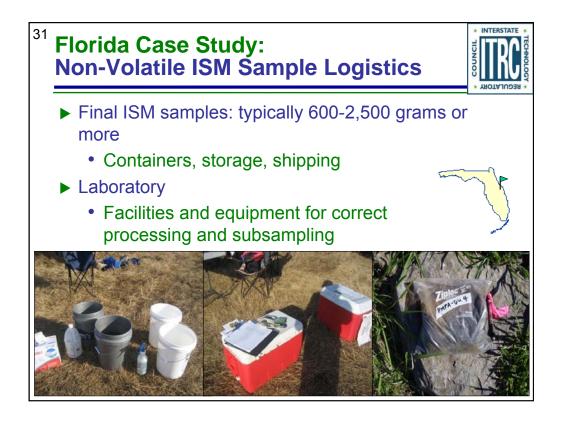
•Generally follows same process as laboratory, i.e. sieving, spread evenly in thin layer, collect multiple systematic random increments to form subsample (i.e., 2 dimensional (2D) Japanese slabcake subsampling)

•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

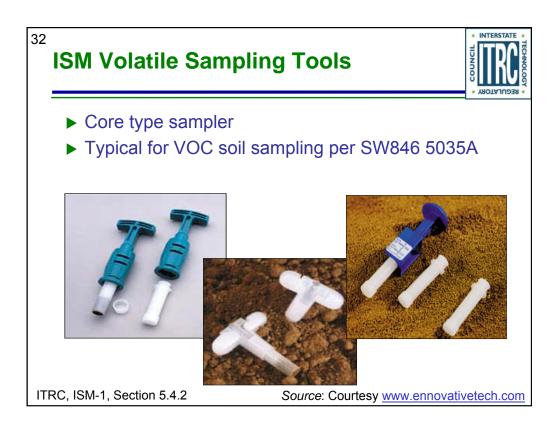
ITRC, ISM-1, Section 5.4.1



•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



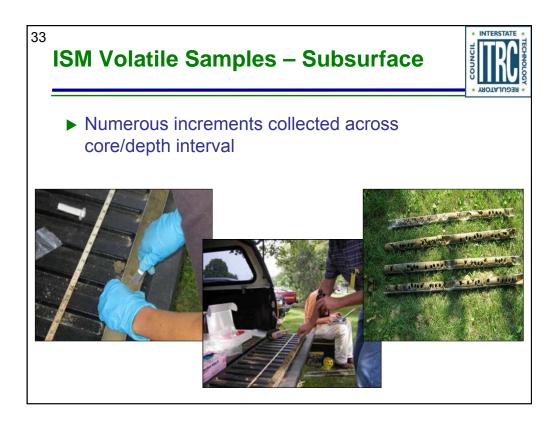
•Core increments on smaller (typical 5 gram) scale

•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

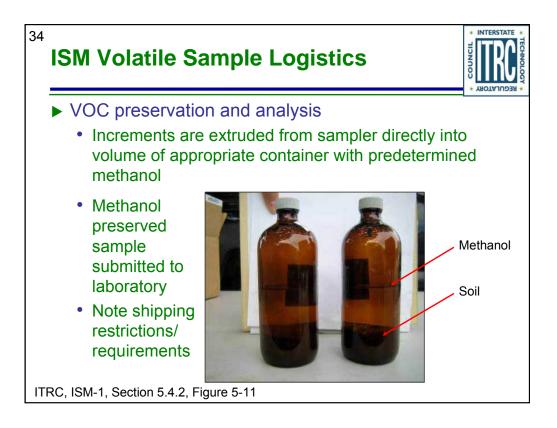


•Core, wedge, slice increments impractical for VOCs

•Representative VOC sampling of subsurface core

•Potential VOC loss; increments should be collected and preserved a quickly as possible from opened core

•Photo on right is an example of a core with VOC plugs removed at ~2 inch intervals

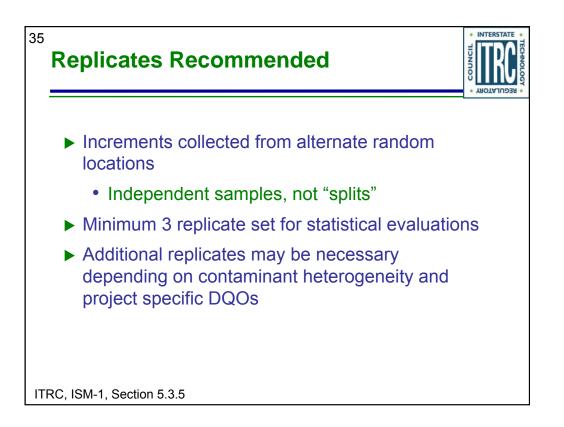


•Close laboratory coordination required for the appropriate sample bottles and methanol volume

•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

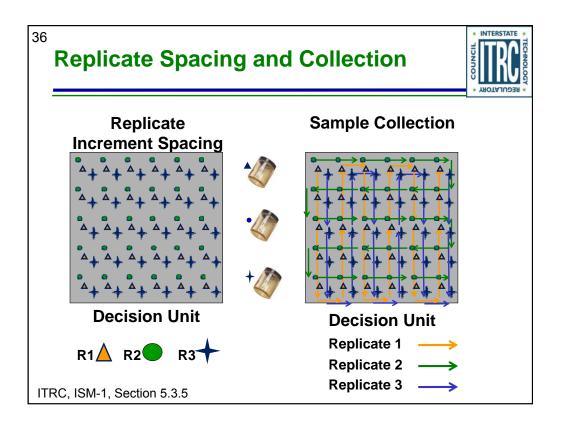
ITRC, ISM-1, Section 5.4.2, Figure 5-11 See section 5.4.2 for additional information and options for methanol



- 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



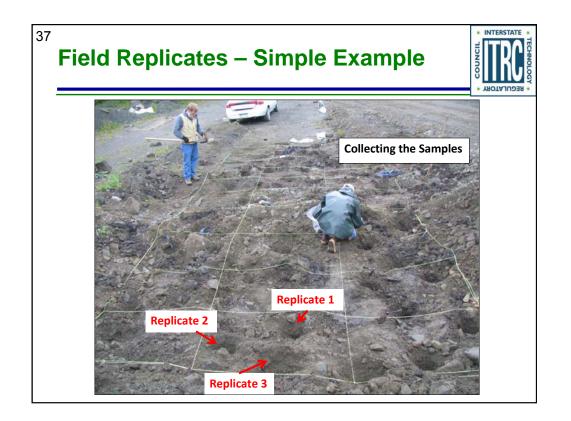
•Field replicate example for systematic random sampling design; different random starting location, "serpentine pattern" if collected in succession

•The collection order (of increments/replicates) does not matter; what is most efficient in the field

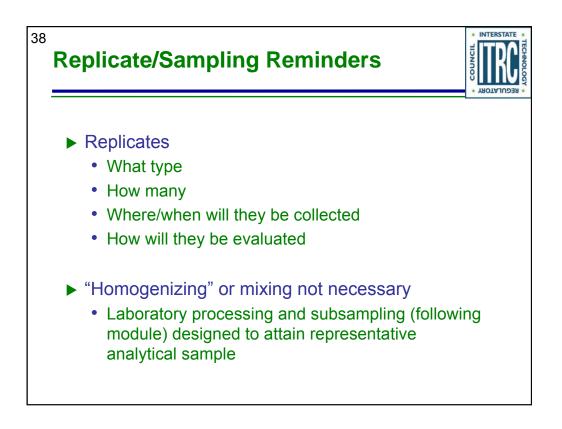
•Submitted blind to laboratory

Supplemental Information

ITRC, ISM-1, Section 5.3.5



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

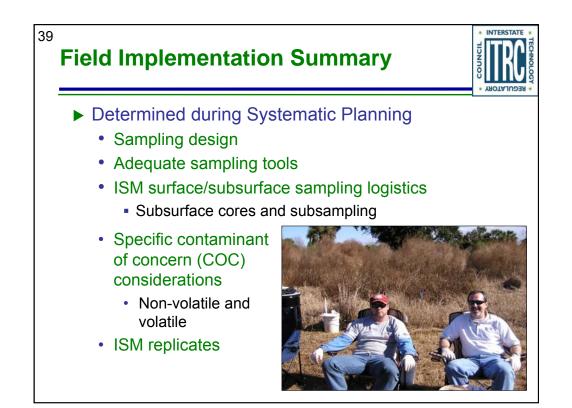


•What type - DU and/or field subsampling replicates

•How many – 3 or more depending on DQOs

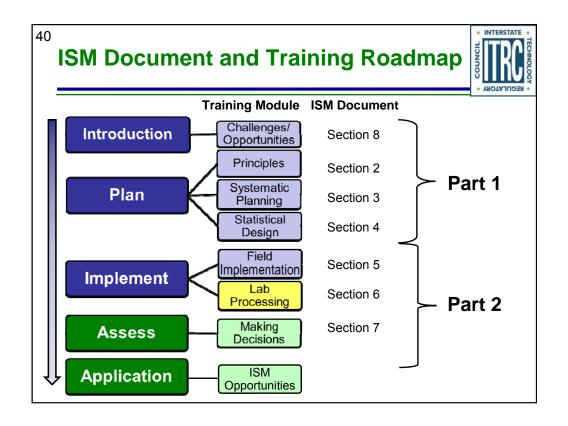
•Where - replicate increment location

•When – simultaneously within grid or consecutively



•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.



Focus – Match lab processTo analytesAnd 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.





Learn how to:

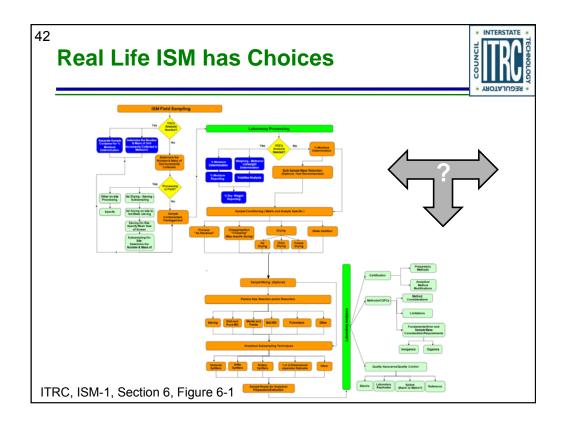
- Match process options to analytes and data objectives
- Manage sample moisture
- ► Select/reduce particle size
- Collect subsamples for analysis
- ► Apply Quality Assurance
- ► Examine options for lab certification

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.

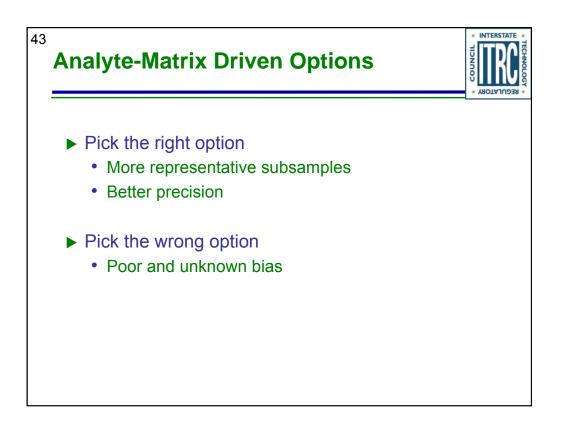


•Many process choices

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.

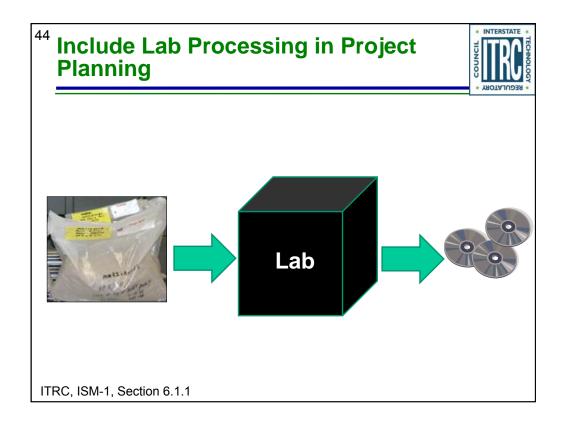
There is no universal process that is optimal for all analytes and matrices. Although the general dry, grind, subsample and analyze is the most common it is not appropriate in all cases. There are other options.



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.



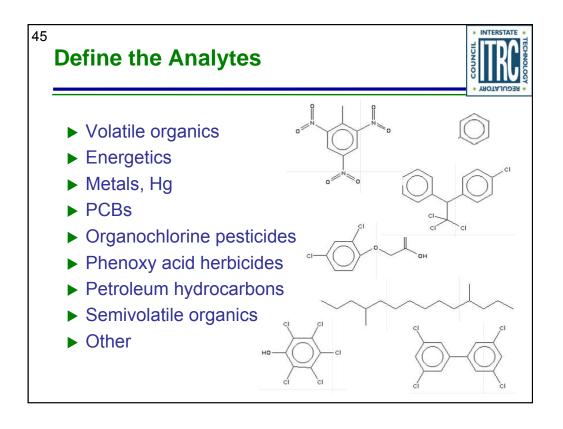
•Project planning, include lab processing

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



•Determine analyte groups

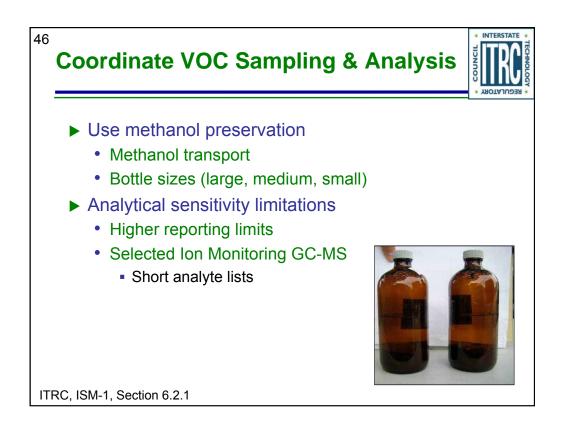
List individual analytes

·Energetics such as explosives and propelents

•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.



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

Narrative

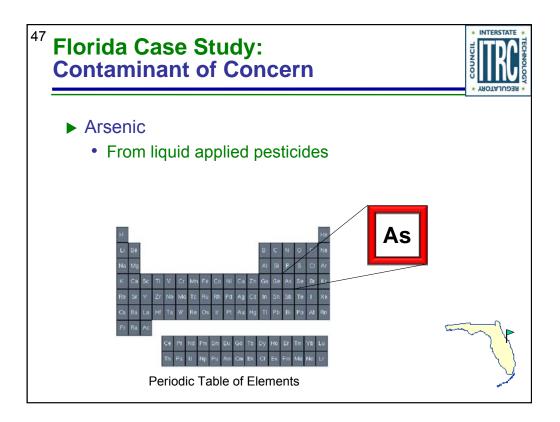
Careful coordination between lab and field crew is usually needed because the lab supplies custom methanol preserved bottles designed for the number of increments in each specific decision unit. Recall that Department of Transportation regulations restrict shipment of containers with more than 30 mL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

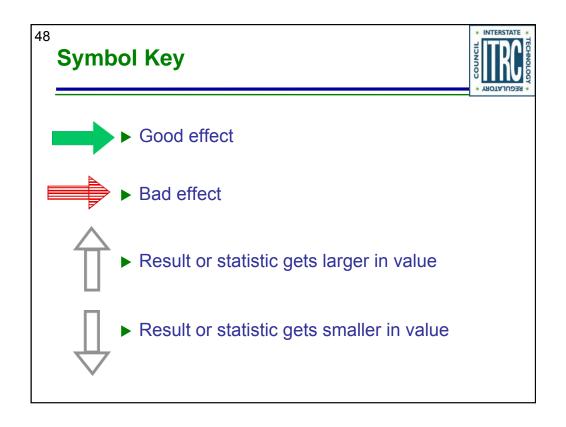
Section 6.2.1



ArsenicLiquid pesticide

Narrative

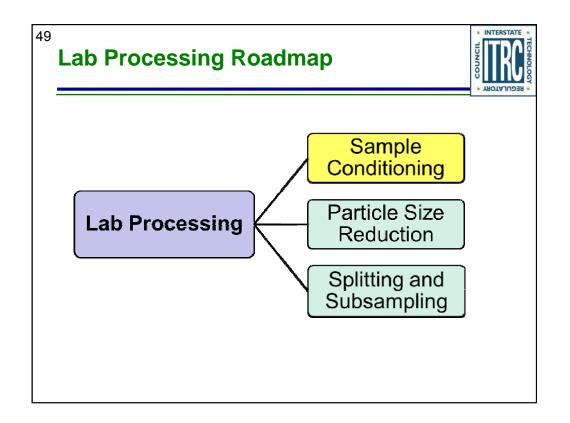
Arsenic was the contaminant of concern for the Florida golf course study. It had been applied in liquid form as a pesticide.



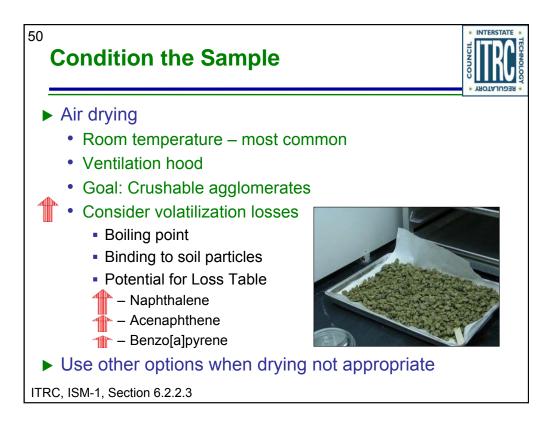
- Coded arrows
- •Solid green good
- •Striped red bad
- •Upward pointing larger value
- Downward pointing smaller value

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.



No associated notes.



Air dry
Ventilation
Low boiling – volatilization losses
Binding to particles
Loss risk table

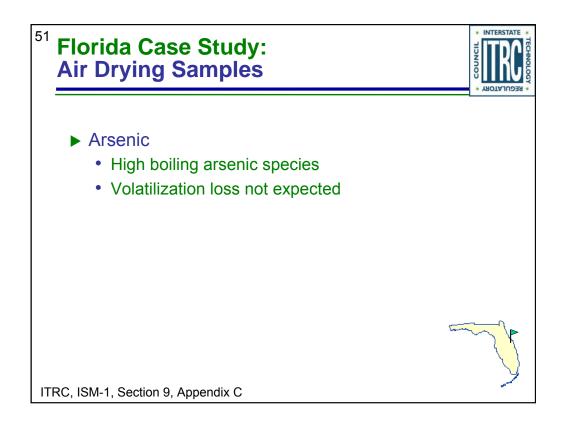
•Other conditioning options available when air drying not appropriate

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 loosing various semivolatile organics and energetics during air drying. For example, the risk of loosing 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



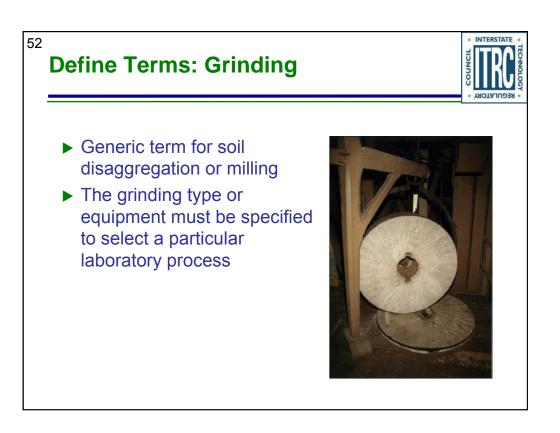
Case study example, look for thisHighlight the process decisions for lab process moduleData shown in next moduleAir drying

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

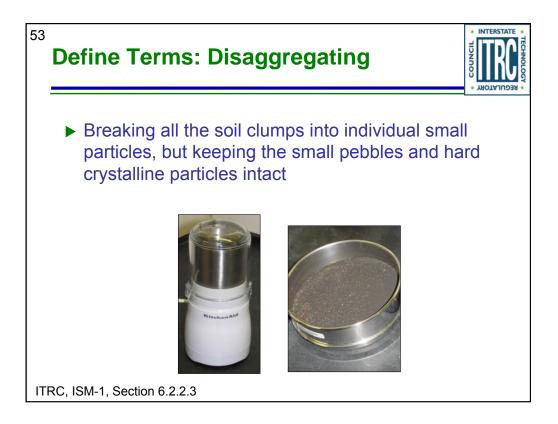
Chapter 9 Appendix C



* 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.



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

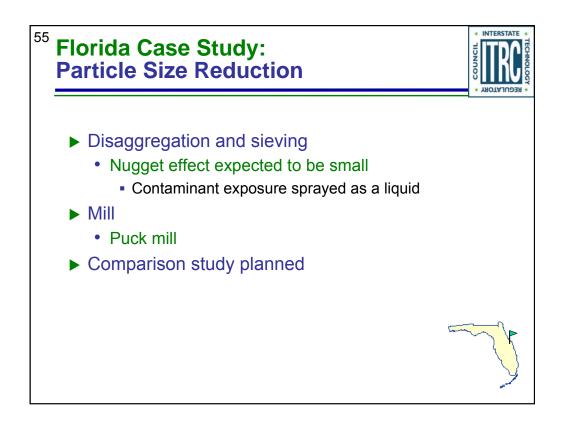


Milling defined
Compared to disaggregation
Lower std dev
Higher measured metals concentration

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.

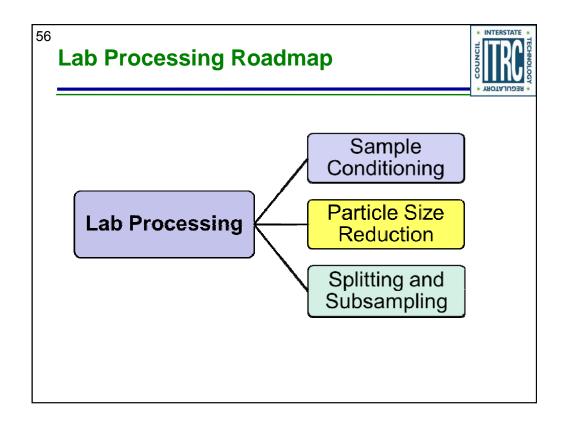
Supplemental information



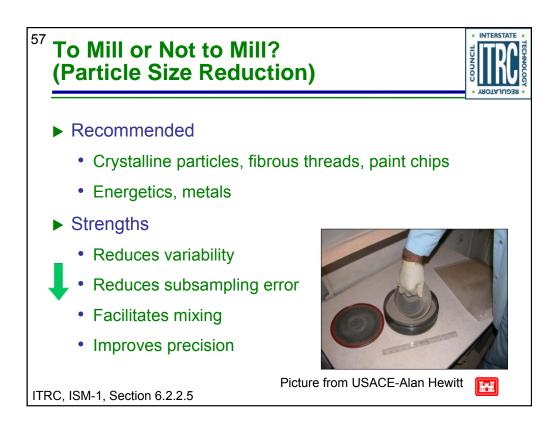
Disaggregate and #10 sieveSample split for milled and unmilled processing

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.



No associated notes.



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



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

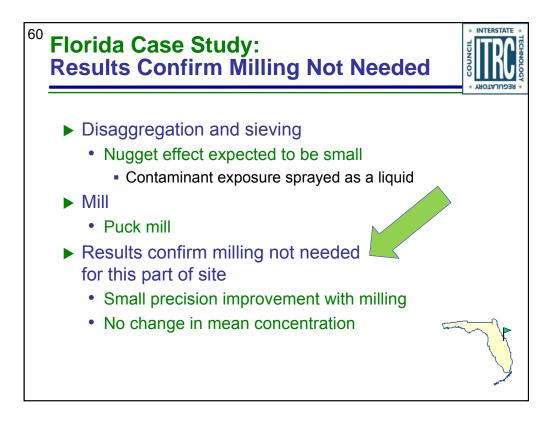


Puck mill
Ball mill
Mortar and pestle
Compare analyte to mill materials

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.

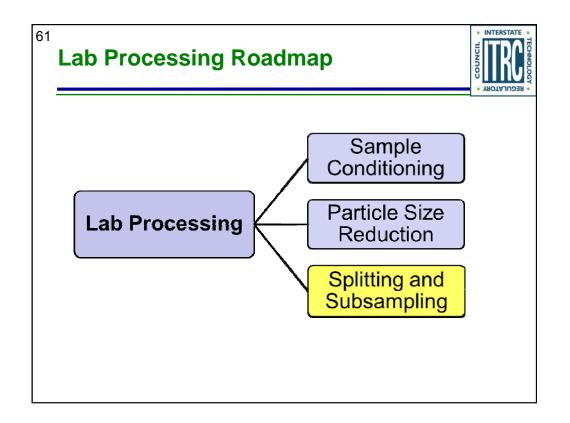
Supplemental information



Only a small difference between milled and unmilled results.Milling not neededDue 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.



No associated notes.



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



- 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.

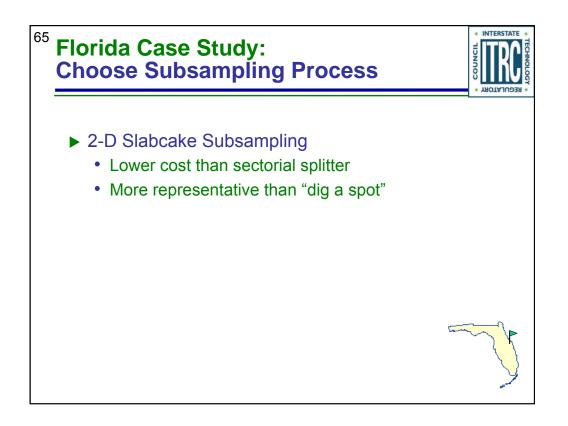
Supplemental information



Non-cohesive soilBlunt scoop with sidesNot round scoop

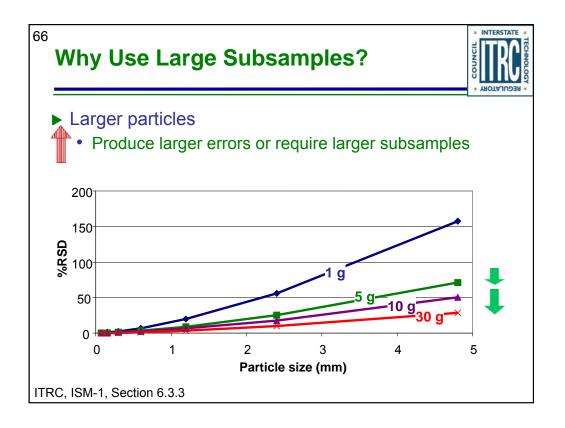
Narrative

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.



Narrative

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



•Large particles >> more variable results

•Reduce variability – larger subsamples

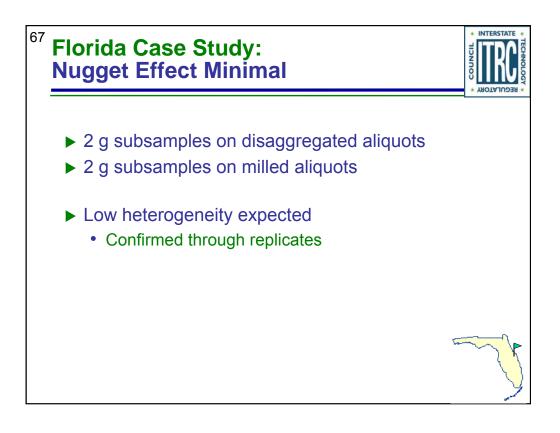
•See Day 1 training about the heterogeneity of soil for the causes

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.

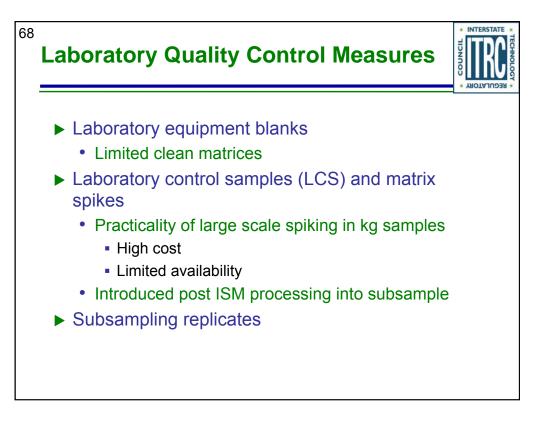
Supplemental information

See Section 6.3.3 and ASTM Standard D6323 Sec A1.1



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.



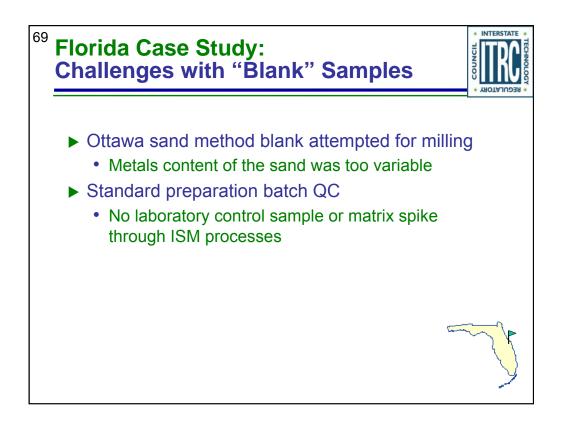
•QC •Blanks – no single clean matrix for all analytes •LCS – matrix + cost •MS - cost •Replicates - triplicates

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.

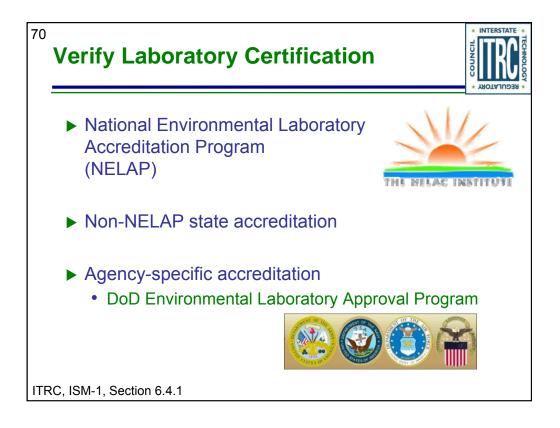
Supplemental information

See Section 6.4



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.



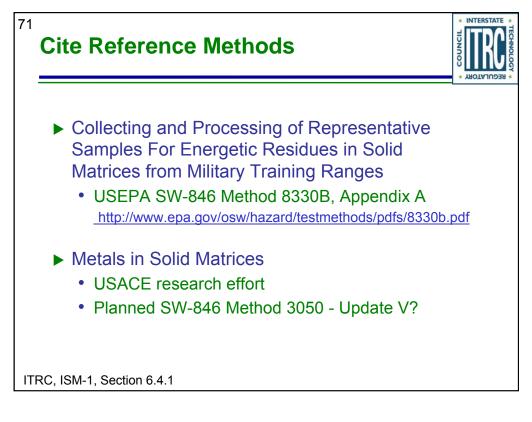
•Certification options from these •NELAP •State •Agency (e.g. DoD)

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



•Reference methods •8330B •3050

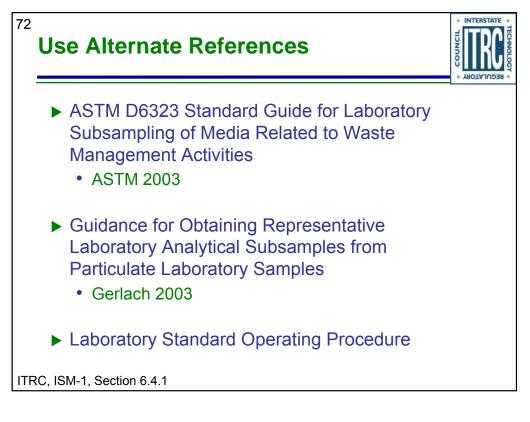
•Certification usually based on analysis method, secondarily extraction of digestion and subsampling ignored, but now covered for ISM

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



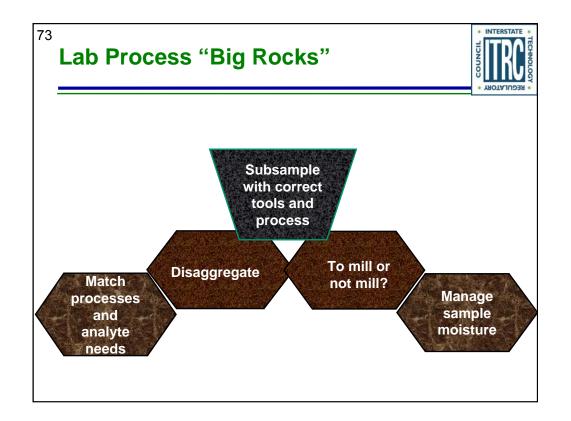
•General references •ASTM •EPA •Lab SOP

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



Speaker bullets

•Big Rocks

- •Match analyte and processes
- Manage moisture
- Disaggregate
- •Mill or not

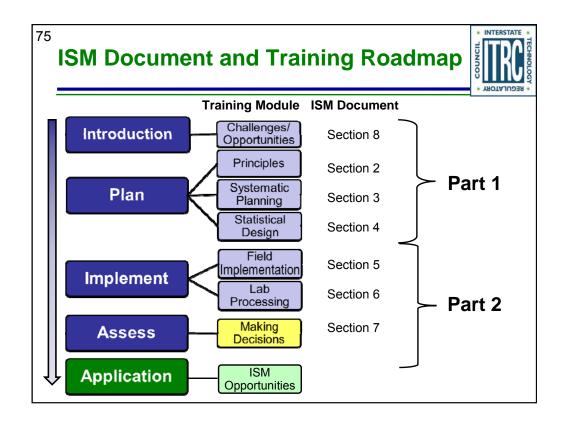
•Subsample

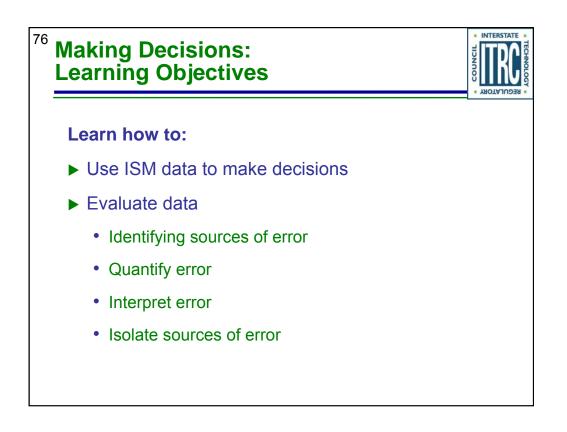
Narrative

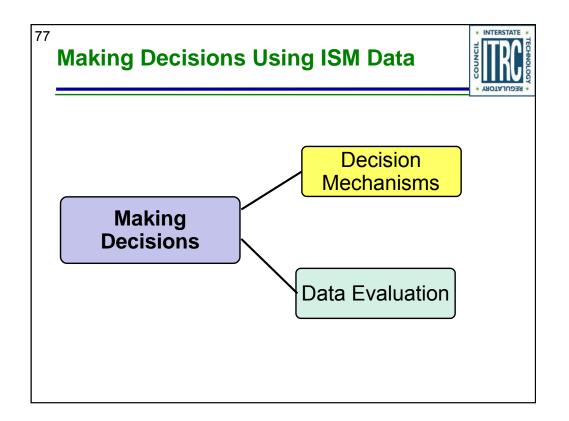
In closing, there are five foundational "big rocks" that must be addressed for lab processing of ISM samples. First, match the processes and the analyte needs. Second, manage sample moisture according to these analyte needs. Third, disaggregate samples by breaking up the soil clods. Fourth, answer "To 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)









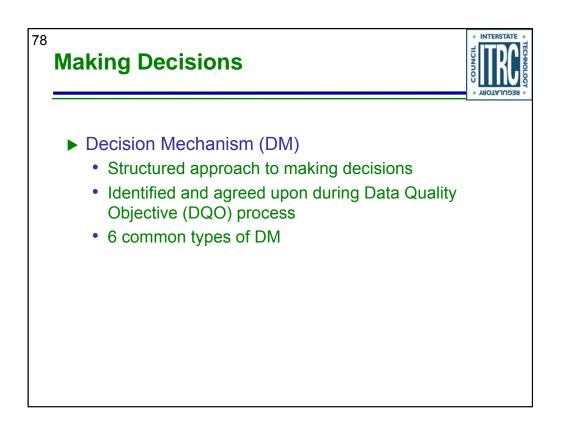
Speaker bullets

•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

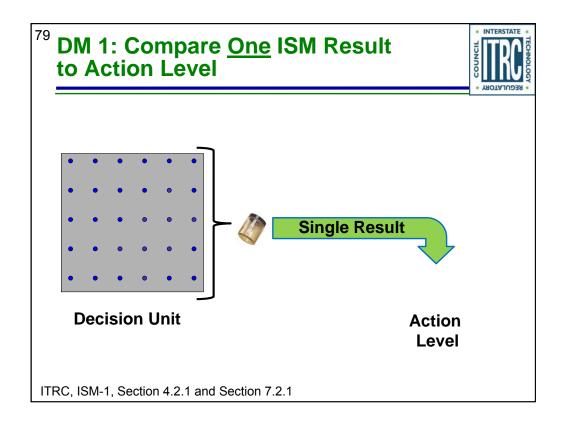


Bullet 1: ITRC Definition: an algorithm or protocol that results in the decision about a potential contaminant of concern or for a decision for a volume of media.

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;

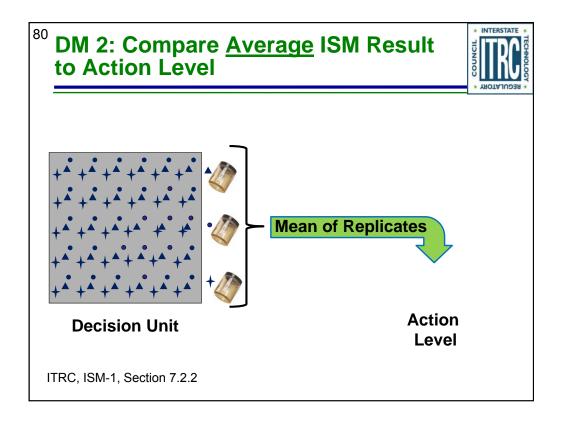
Bullet 3: indicate that these are examples; you should come up with site-specific criteria (not all DMs will be acceptable in all states, scope before hand)



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 -

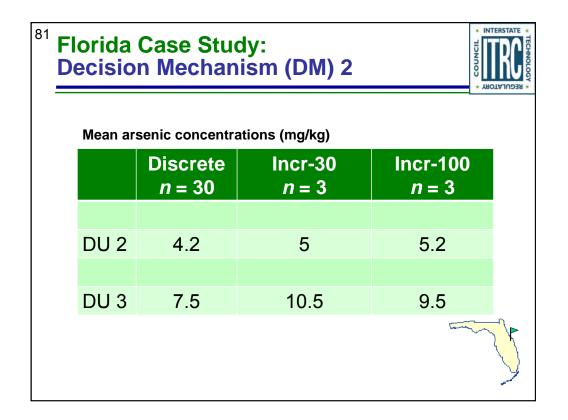
This approach might be acceptable when the predicted mean concentration is much greater than, or much less than, the action level. In this situation, the ISM sample provides confirmation of what is already strongly suspected—that the DU clearly passes or fails. However, when the ISM sample result is 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.



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 <u>mean concentration</u> of the replicates is calculated and compared to the action or screening level identified during systematic planning.

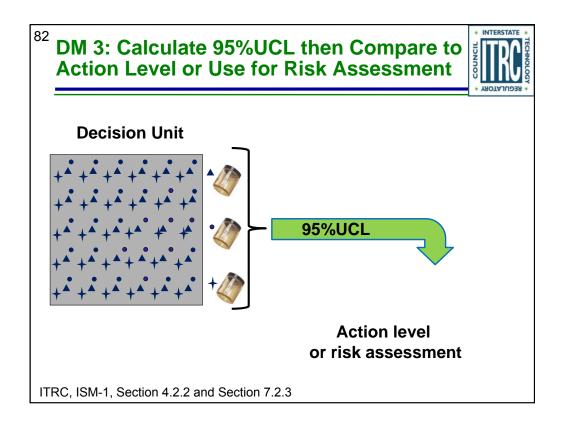
The mean concentration from replicate samples is likely to be closer to the true mean of the DU than the result from a single ISM sample (see Ch 4), and could, therefore, be considered to provide a more reliable estimate of the mean. There is no assurance, however, that the actual mean concentration 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*****



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



• 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

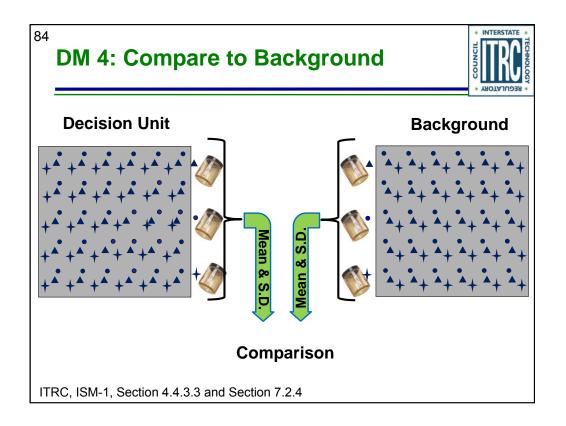
Narrative - Project objectives may specify that the estimate of the mean concentration provided by ISM sampling must be health protective, meaning that there is low chance of underestimating the actual mean concentration within the DU, such as for use in a baseline risk assessment.

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

Discrete $n = 10$ (mg/kg) Incr-30 $n = 3$ (mg/kg) Incr-100 $n = 3$ (mg/kg) Mean21.81.7
Mean 2 1.8 1.7
Std Dev 1.4 0.08 0.03
95UCL 3.0 2.0 1.8

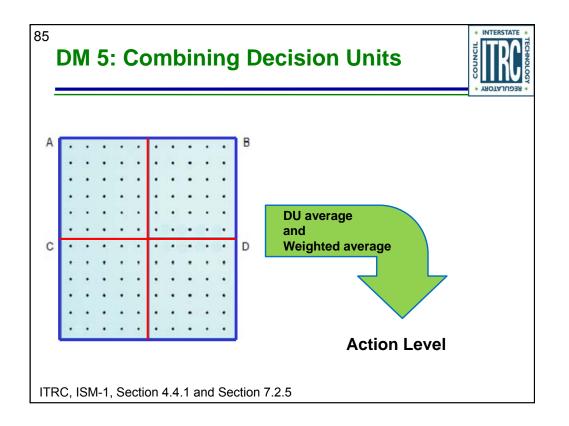
Speaker Bullets - Means are similar; variability (std dev) much lower for incremental samples; variability affects 95% UCL; in this case – method used determines the decision to be made



Narrative:

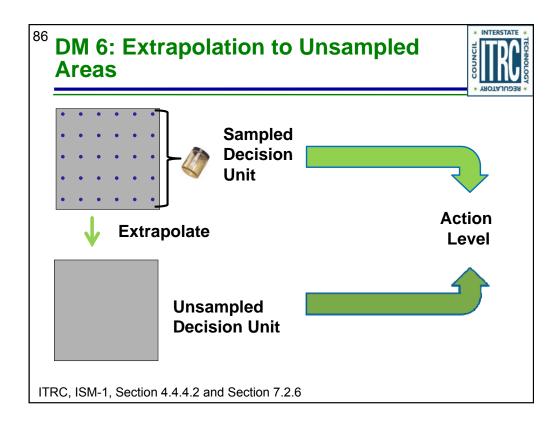
ISM results are not suitable for point-by-point comparison with UTLs generated from discrete sample background data because ISM and discrete datasets have fundamentally different characteristics. If background and site data are both generated using ISM, comparisons of central tendencies (e.g., medians) 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



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



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

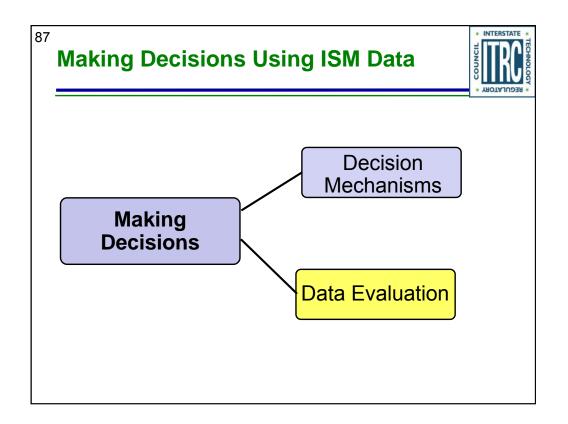
Frequently proposed for large tracts of land or large volumes of soil to reduce costs.

•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.

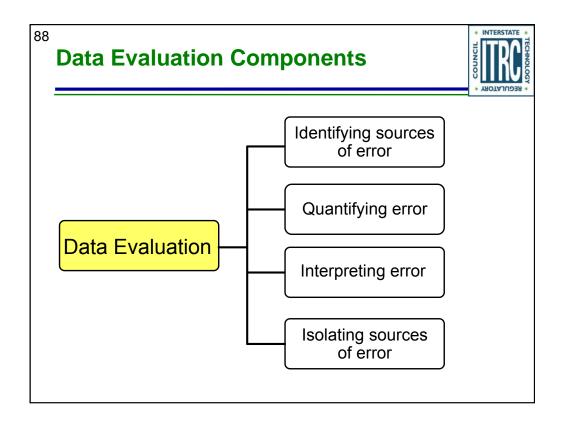
• The issue is the same whether the DU is sampled by ISM or with discrete sampling – is there sufficient basis to assume that the mean concentration estimated there is the same (or higher) than the mean concentration in unsampled DUs.

Must assume that both the mean and the distribution of contaminants are essentially equivalent across DUs

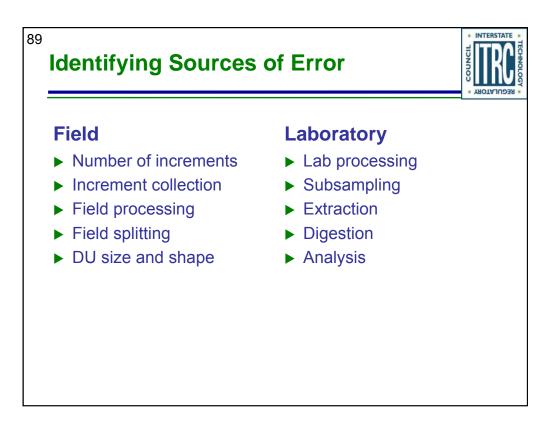


Speaker bullets - decision mechanisms decided upfront / data evaluation

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



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

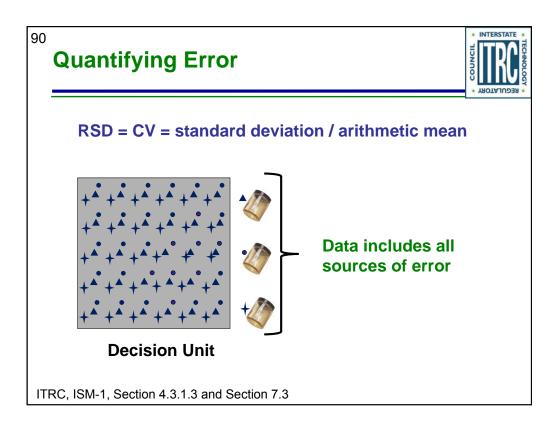


Error (Variability) - not "mistake"

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

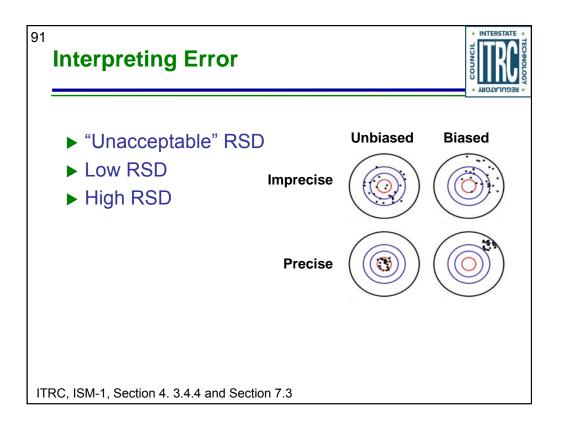
It is useful when evaluating data to consider the steps in the process where errors (variability) may have been introduced

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



Narrative - Evaluation of overall error can by 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.



• 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.

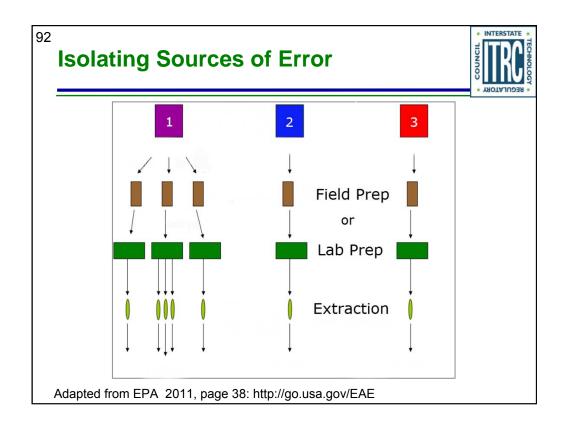
•High RSD may indicate errors introduced through lab or field activities or may be attributable to heterogeneity in site concentrations. Generally leads to the need to isolate potential sources of error – discussed on the earlier slide "Identifying Sources of Error" (see Section 4.3.4.4 for more info)

•Lab

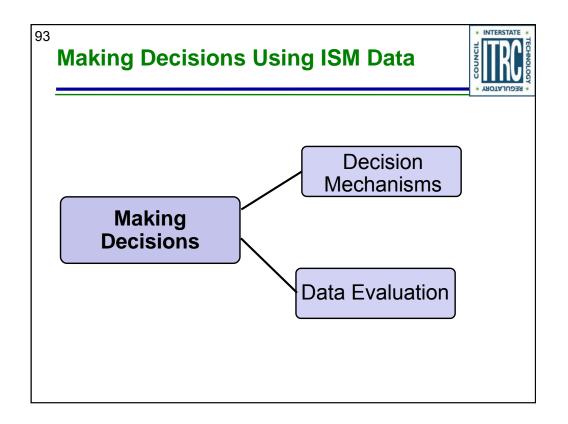
•Field

RSD measures reproducibility, not accuracy of the results (see Section 4 for addition 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.

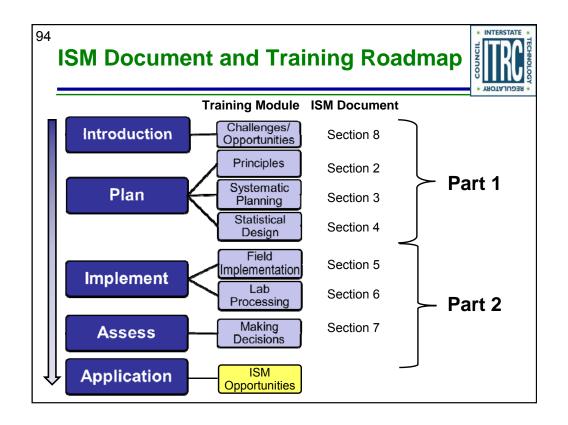


Ideally, the project team will then designate one of these replicates for separation into laboratory replicates.) Replicate RSD data is intended to quantify the total error of the measurement system; and if one sample is split into laboratory replicates then the total error can be attributed to either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



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.



Take home messages

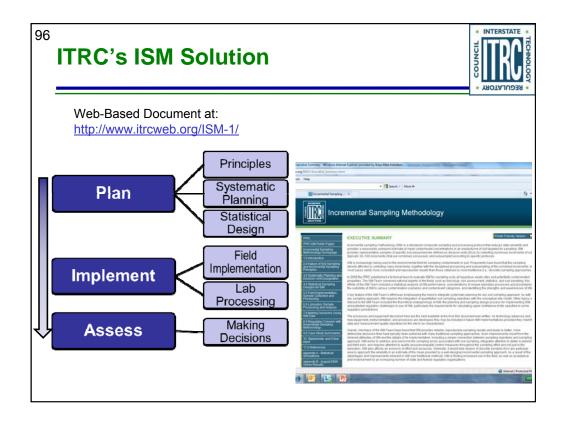


IF YOU REMEMBER ANYTHING FROM THIS TWO DAY TRAINING...

•All sampling is about getting a representative sample. In the case of soil sampling at contaminated sites, the challenge of getting a representative sample includes dealing with some pretty extreme heterogeneity. Remember – a 'representative sample' has all the elements in the sample that you are interested in (normally contaminant level) in the same proportion as is in the entire population. You can't put the entire population (the 'sampling unit') into a jar – so somehow you have to get a representative sample that accomplishes your goal.

•So we hope you have thought a bit about how you are achieving 'representativeness' with the type of soil sampling you are doing now. **Something to consider - are you getting a representative sample at your sites?**

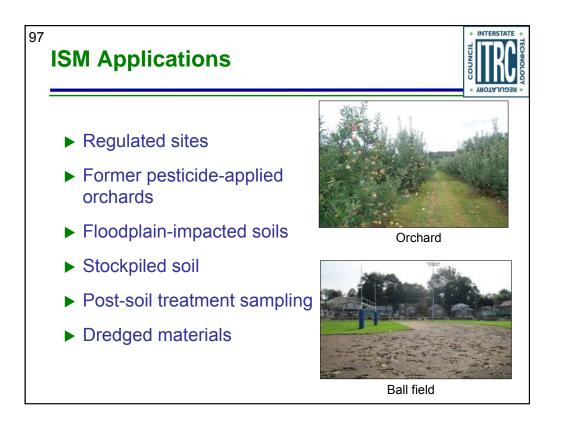
•Picture Reference: http://www.swrcb.ca.gov/rwqcb2/brownfields.shtml



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.



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.

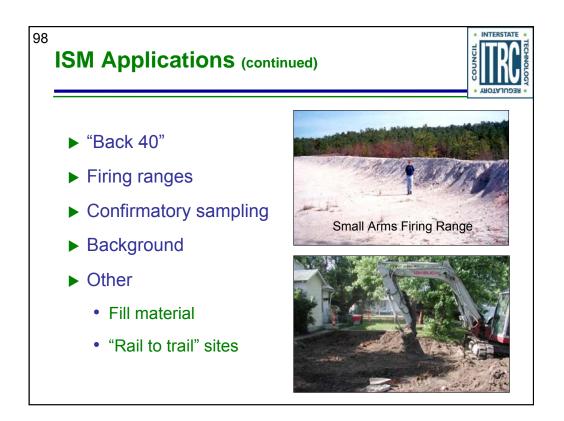
However, we recognize that there can be reluctance and resistance to implementing ISM on regulated sites, so perhaps you can think of other opportunities – other circumstances and types of sampling – where there may be an easier 'point of entry' for demonstrating ISM in your state/region/jurisdiction. 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

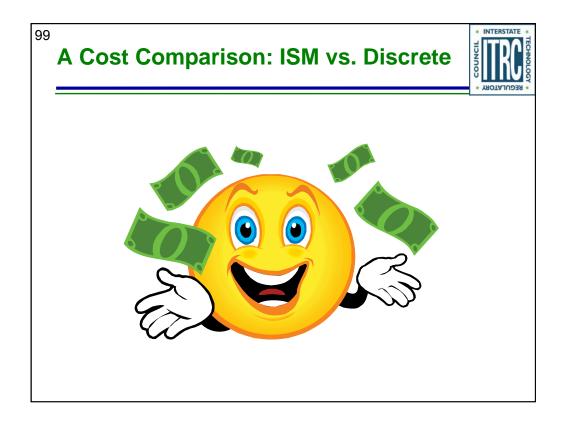


SLIDE on ISM Applications (continued)

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.



MONEY MATTERS (Cost Comparison/Chapter 8 in ISM doc)

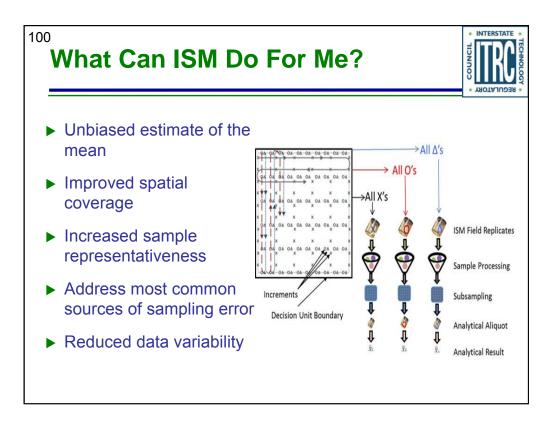
Cost Comparison Studies – not enough time/not definitive in regards to which is really cheaper – discrete or ISM. However,

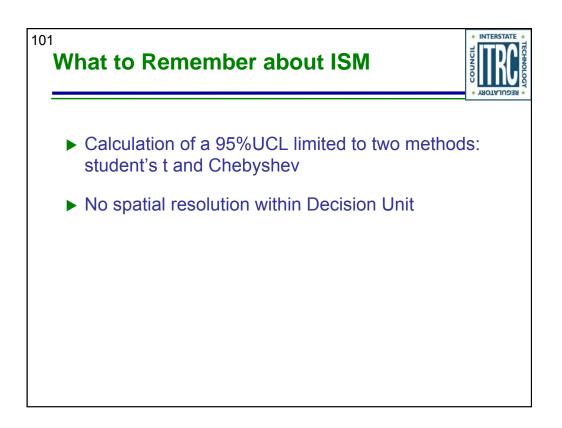
•The biggest concern from the perspective of an RP is the **potential future costs of making the wrong decision based on inadequate data**, not the cost of the field work and or laboratory analyses.

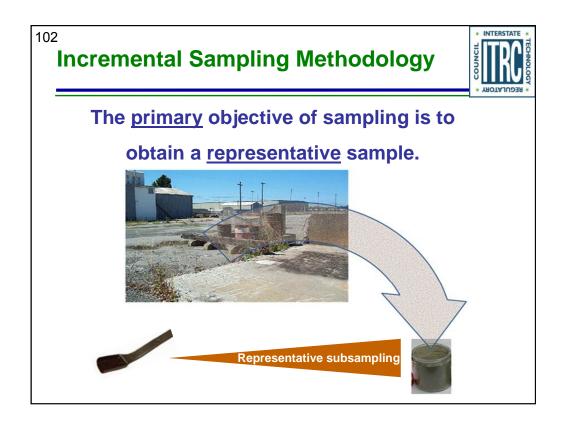
•DU-ISM investigation approaches **help to reduce uncertainty and associated environmental liability** because of the better decisions that can be made than with traditional discrete sampling.

•ISM results are repeatable and should alleviate concern about missing something and also reduce number of remobilizations.

•This is the really the "bottom line" with respect to costs.





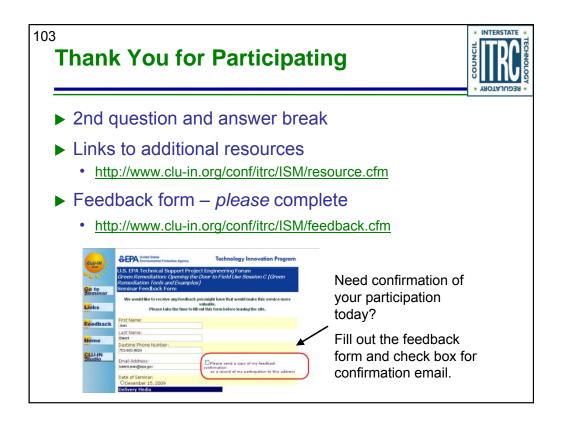


ISM OBJECTIVE (Final Message)

So here again is our favorite slide – an illustration of the challenge of getting a representative (and meaningful!) sample from an area that typically has heterogeneous contamination. We've talked for two training sessions on just what goes into meeting that challenge, and how ISM can really fit the bill.

So as we go on to the final question and answer period, let's just revisit one last time that the primary objective of sampling is to obtain a representative sample – and hopefully this simple illustration will reinforce – and remind us – of just how ISM accomplishes that goal.

Thank you for your attention and now we will take any questions that you may have...



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

 \checkmark Guiding technology developers in the collection of performance data to satisfy the requirements of multiple states

 \checkmark 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:

 \checkmark 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
- $\checkmark \text{Use ITRC}$ products and attend training courses
- ✓ Submit proposals for new technical teams and projects