Better Site Characterization Through Incremental Sampling Methodology – Status Report on ITRC Guidance

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Chasing Uncertainty Sources

- Instrumental analysis
- Sample preparation
- Laboratory sub-sampling
- Field sample collection
Does the decision unit fit in the sample jar?

Representative subsampling
Why is this important?

$\bar{X}$ with known and less uncertainty

Representative subsampling
Better Data Drives Better Decisions
Incremental Sampling

- Systematic Random Design

Random starting locations in first grid
• **Incremental Sampling Methodology Team**
  
  ~ [www.itrcweb.org/teampublic_MIS.asp](http://www.itrcweb.org/teampublic_MIS.asp)
  
  ~ Formed Jan. 2009

Disclaimer: Most of the material in this presentation has been derived from the Spring 2011 draft guidance developed by the ITRC ISM team. ITRC does not endorse the use of specific vendors or technologies. This presentation is not official ITRC sanctioned training material. It has been reviewed by ITRC for compliance with the ITRC usage policy.
ISM Guidance

Introduction
ISM Principles
Systematic Planning
Statistical ISM Design
Field Implementation

Laboratory Process
Making Decisions
Regulatory Concerns
Case Studies
Stakeholder Input
• Introduction
• Laboratory Processing
• Laboratory Analysis
• Quality Assurance
Real Life ISM is Complicated
Include Lab Processing in Project Planning
• Goal: Improve subsampling representativeness

• Goal: Improve precision & minimize bias
• Always
  ~ Improved precision

• Hopefully
  ~ Improved accuracy (of single measurements)
    ° Retain contaminants of concern
    ° Avoid contamination

• Sweat the details or risk misleading data
Symbol key

- Good effect
- Bad effect
- Good or Bad effect depending the question
- Result or statistic gets larger in value
- Result or statistic gets smaller in value
Identify the Sample

- Materials to remove
  - Vegetation
  - Oversized material
  - Decantable water
- Manual removal
- Sieve (after drying)
  - < 2 mm (#10)
- Lead source example
  - Paint fines
  - Intact slugs
Analyte Groups

- Volatile organics
- Energetics
- Metals, Hg
- PCBs
- Organochlorine Pesticides
- Phenoxy acid herbicides
- Petroleum hydrocarbons
- Semivolatile organics
Semivolatile Organic Compounds and Inorganics

- Bulk Sample Splitting
- Sample Conditioning
- Particle Size Reduction
- Splitting and Subsampling

Analyte Specific Considerations
Bulk Sample Splitting

- Limited Applicability

- Sample Splitting for multiple analyte groups
  - Alternate or fractional shoveling
  - Consider “nugget” effect
  - Increases fundamental error (variance)
Semivolatile Organic Compounds and Inorganics

Bulk Sample Splitting

Sample Conditioning

Particle Size Reduction

Splitting and Subsampling

Analyte Specific Considerations
Sample Conditioning

- Air drying
  - Room temperature - most common
  - Ventilation hood
  - Consider volatilization losses
    - Boiling point
    - Binding to soil particles (lower conc. > higher binding > lower losses)
    - Loss risk table
      - naphthalene
      - 2-methylnaphthalene
      - acenaphthene
      - dibenzofuran
    - Loss risk test
  - Goal: Crushable agglomerates
• As-received
  ~ Least air exposure
    ◦ Fewest analyte losses
  ~ Limits soil processing options
    ◦ Fractional shoveling
    ◦ Manual forced sieving
Semivolatile Organic Compounds and Inorganics

1. Bulk Sample Splitting
2. Sample Conditioning
3. Particle Size Reduction
4. Splitting and Subsampling

Analyte Specific Considerations
Defining Terms

- Grinding:
  ~ Generic term for soil disaggregation or milling. The grinding type or equipment must be specified to select a particular laboratory process.
• Disaggregating:
  ~ Breaking the soil clumps into individual small particles, but keeping the small pebbles and hard crystalline particles intact.
Defining Terms

- **Milling:**
  ~ Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. < 250 um or < 75 um).
To mill or not to mill

• Yes - recommended
  ~ Crystalline particles, fibrous threads
  ~ Energetics, metals
  ~ Strengths
    ° Reduces fundamental error
    ° Reduces sub-sampling error
    ° Facilitates mixing
    ° Improves precision
Milling Improves Precision

Lead Precision

% RSD

Unground  5-min Puck Mill  8-hr Ball Mill  12-hr Ball Mill  16-hr Ball Mill  20-hr Ball Mill
To mill or not to mill

- No – not recommended
  - Volatile, thermally labile, increased “availability”

- Examples
  - Low boiling PCBs, OCPs, TPHs, SVOCs, metals

- Limitations
  - Analyte losses
  - Metals contamination
  - Potential bias to metals risk assessment (pebbles)
Mill Erosion Elevates Cr Results

Chromium Results

Concentration (mg/kg)

Unground  5-min Puck Mill  8-hr Ball Mill  12-hr Ball Mill  16-hr Ball Mill  20-hr Ball Mill

0  50  100  150  200  250
Milling Releases Metals from “Pebbles”

Lead Results

<table>
<thead>
<tr>
<th>Concentration (mg/kg)</th>
<th>Unground</th>
<th>5-min Puck Mill</th>
<th>8-hr Ball Mill</th>
<th>12-hr Ball Mill</th>
<th>16-hr Ball Mill</th>
<th>20-hr Ball Mill</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>20000</td>
<td>25000</td>
<td>15000</td>
<td>10000</td>
<td>5000</td>
<td>0</td>
</tr>
</tbody>
</table>
How best to mill

- Puck mill or ring and puck mill
  ~ “stable” energetics
- Ball mill
- Mortar and pestle
- Consider
  ~ Analytes
  ~ concentration of interest
  ~ grinder materials
  ~ Particle size needed
How fine is the grind?

- What is the target particle size?
- How to determine completeness
  ~ Timer
  ~ Visual inspection
  ~ Pinch of “flour”
  ~ Sieve #200 (~75 um)
Mills have Limitations

• 5 Minutes puck mill grinding
  ~ 94% of Material < 100 mesh
  ~ 6% > 100 mesh
  ° 8.6 g of deformed metal fragments
  ° 47.6 g of other material
Semivolatile Organic Compounds and Inorganics

- Bulk Sample Splitting
- Sample Conditioning
- Particle Size Reduction
- Splitting and Subsampling

Analyte Specific Considerations
Sample Condition Affects Subsampling Options

- Wet sticky sample
  - Alternate shoveling
  - Fractional shoveling
  - 2 Dimensional Japanese Slabcake

- Dry flowable powder sample
  - All splitting and subsampling techniques
Wet Splitting Options

- Alternate shoveling
- Fractional shoveling
Dry Splitting Options

- Riffle splitter

Dry Splitting Options

- Rotary sectorial splitter
- Paper cone sectorial splitter

Dry Splitting Options

• 1-Dimensional Japanese Slab Cake
Sub-sampling Options

- 2-Dimensional Japanese Slabcake
Sub-sampling Tools

- Square straight sided scoops for dry non-cohesive soil
Using large subsamples

- Larger particles
  - Produce larger errors or require larger subsamples

![Graph showing Fundamental Error vs Particle size (mm) with different sample weights (1 g, 5 g, 10 g, 30 g)].

ASTM D6323 Sec. A1.1
Sample Preparation Modifications

- Dry, fine particulate samples
  - Health and Safety – dust control
- Larger sub-samples
  - (driven by fundamental error concerns)
    - Metals 10 g vs 1 g
    - Hg 5 g vs 0.6 g
- Water added samples
  - Additional drying agent and time
• 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
Matrix Options for Laboratory Quality Control Measures

- Reagent Water
- Ottawa sand
- Teflon Boiling Chips
- Soda Lime Glass
- Reference Sample
- Split field sample
Laboratory Certification

- National Environmental Laboratory Accreditation Program
- Non-NELAP State Accreditation
- Agency-specific Accreditation

~ DoD Environmental Laboratory Approval Program
• Incremental Sampling MIS-Based Laboratory Requirements for the Analysis of Explosives
  ~ (USEPA SW-846 Method 8330B)

• Metals in Solid Matrices
  ~ (USACE 2008)
  ~ Planned SW-846 Method 3050 Update V?
Reference Methods

- 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
Lab Process “Big Rocks”

- Subsample with correct tools and process
- Disaggregate
- Mill or not
- Manage sample moisture
- Match processes and analyte needs
• Full ITRC (non-DoD) review – Early Q2, 2011
• DoD & EPA review – Late Q2, 2011
• Final to ITRC communications – Q4, 2011
• Internet based training – Q2, 2012
Purpose of ISM

Representative samples

Better data

Better decisions
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