Laboratory Processing Learning Objectives

Learn how to:

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

Report Documentation Page

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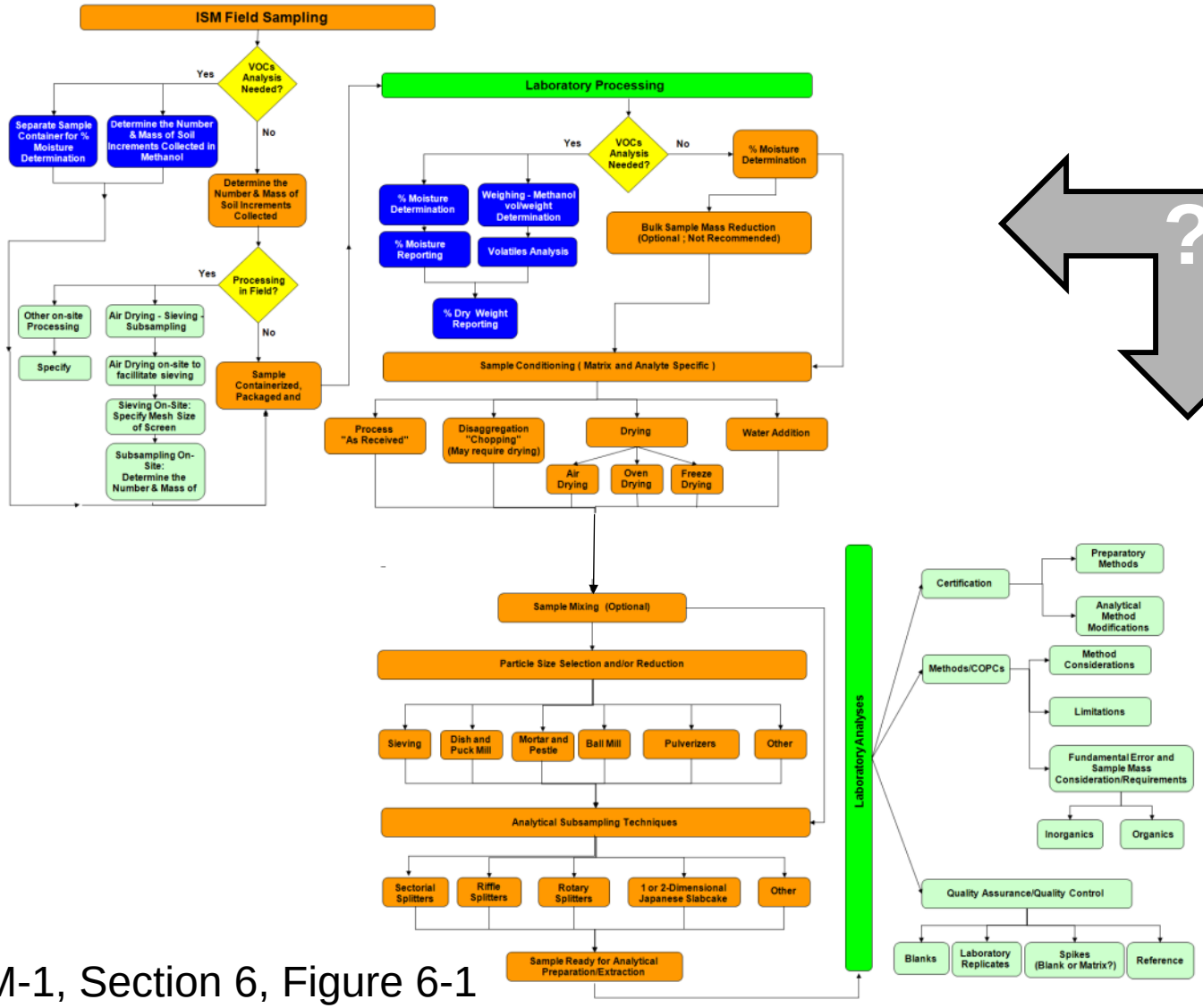
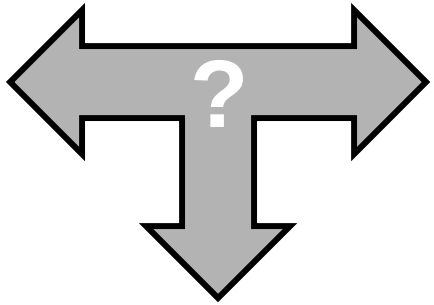
Real Life ISM has Choices



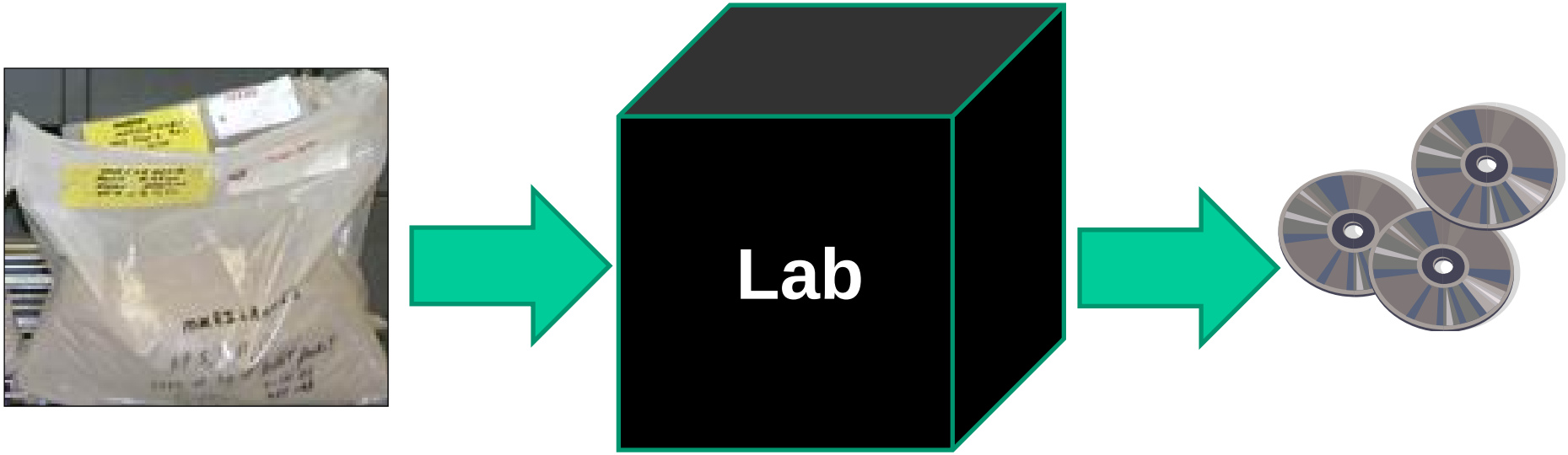


ITRC, ISM-1, Section 6, Figure 6-1

Analyte-Matrix Driven Options

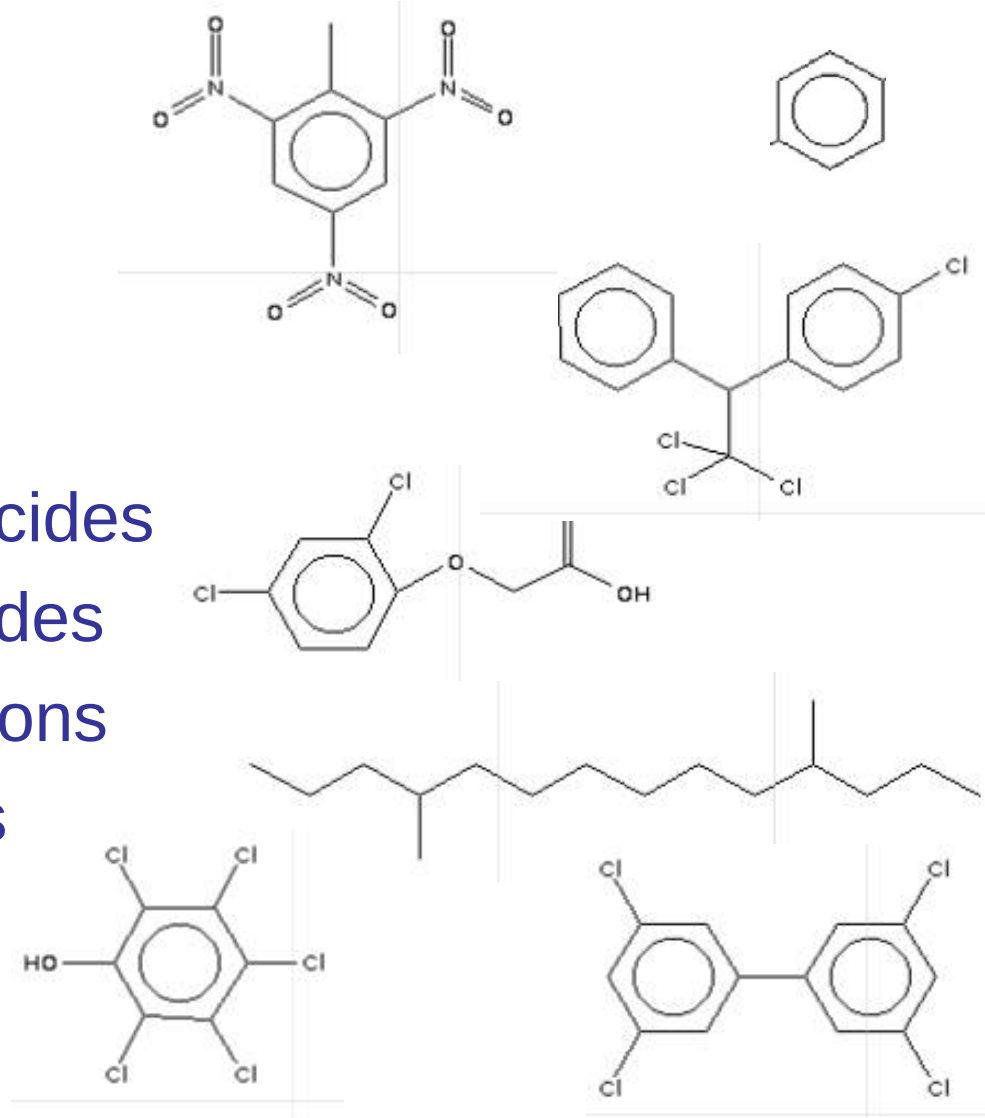
- Pick the right option
  - More representative subsamples
  - Better precision
- Pick the wrong option
  - Poor and unknown bias

Include Lab Processing in Project Planning



Define the Analytes

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

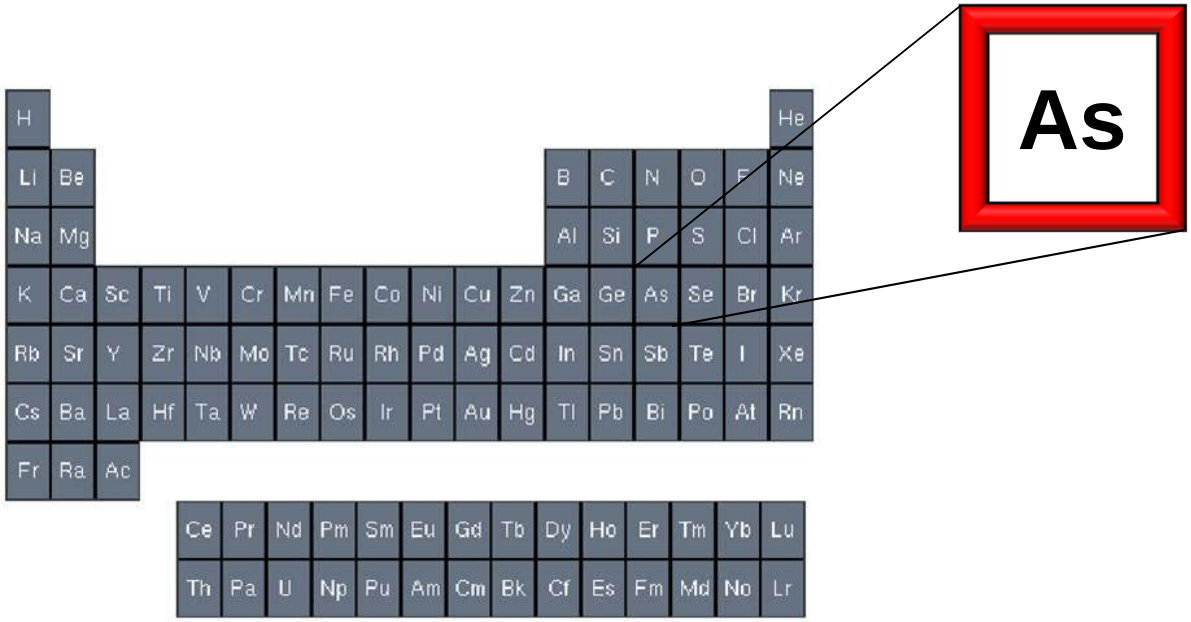


Coordinate VOC Sampling & Analysis

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

Florida Case Study: Contaminant of Concern

- Arsenic
  - From liquid applied pesticides

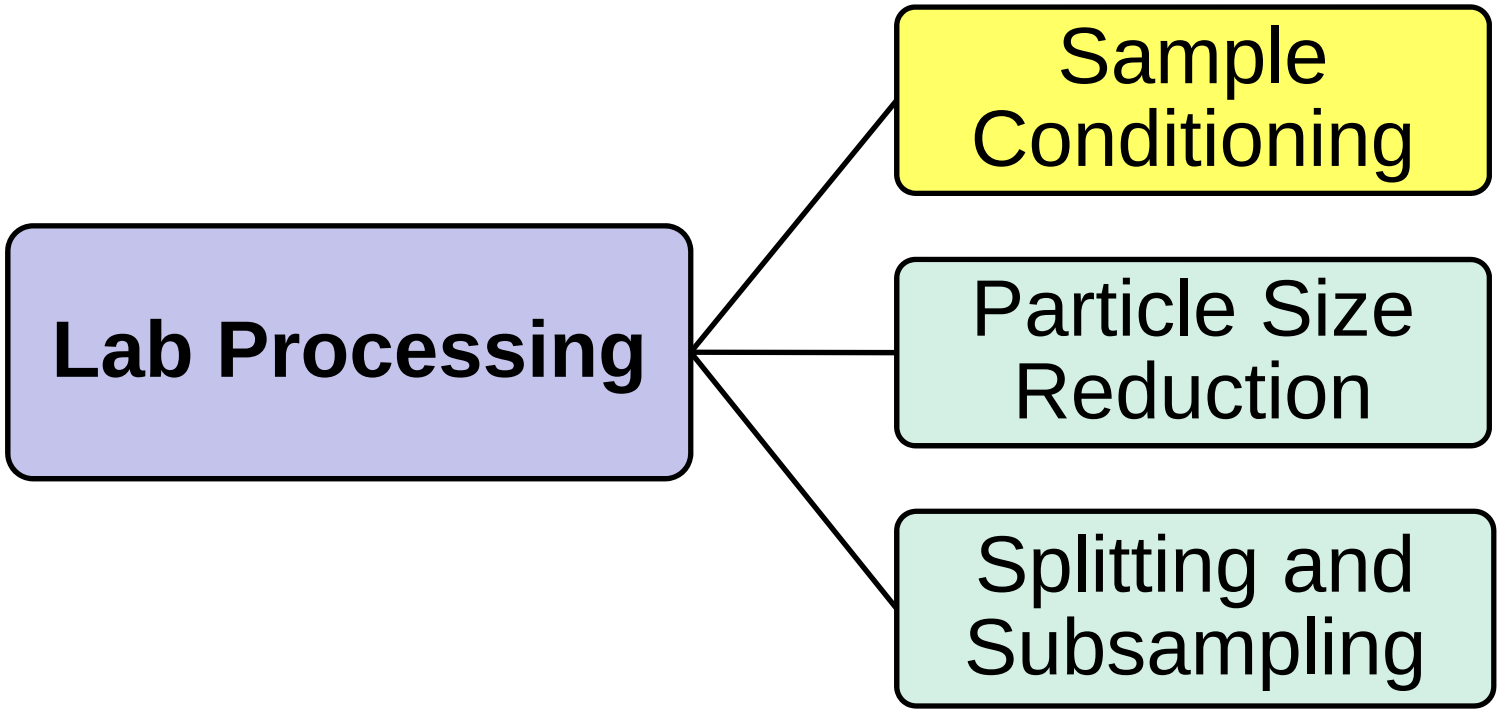


Periodic Table of Elements

Symbol Key

- Good effect
- Bad effect
- Result or statistic gets larger in value
- Result or statistic gets smaller in value

Lab Processing Roadmap



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

Florida Case Study:
Air Drying Samples

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

Define Terms: Grinding

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

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

Define Terms: Milling

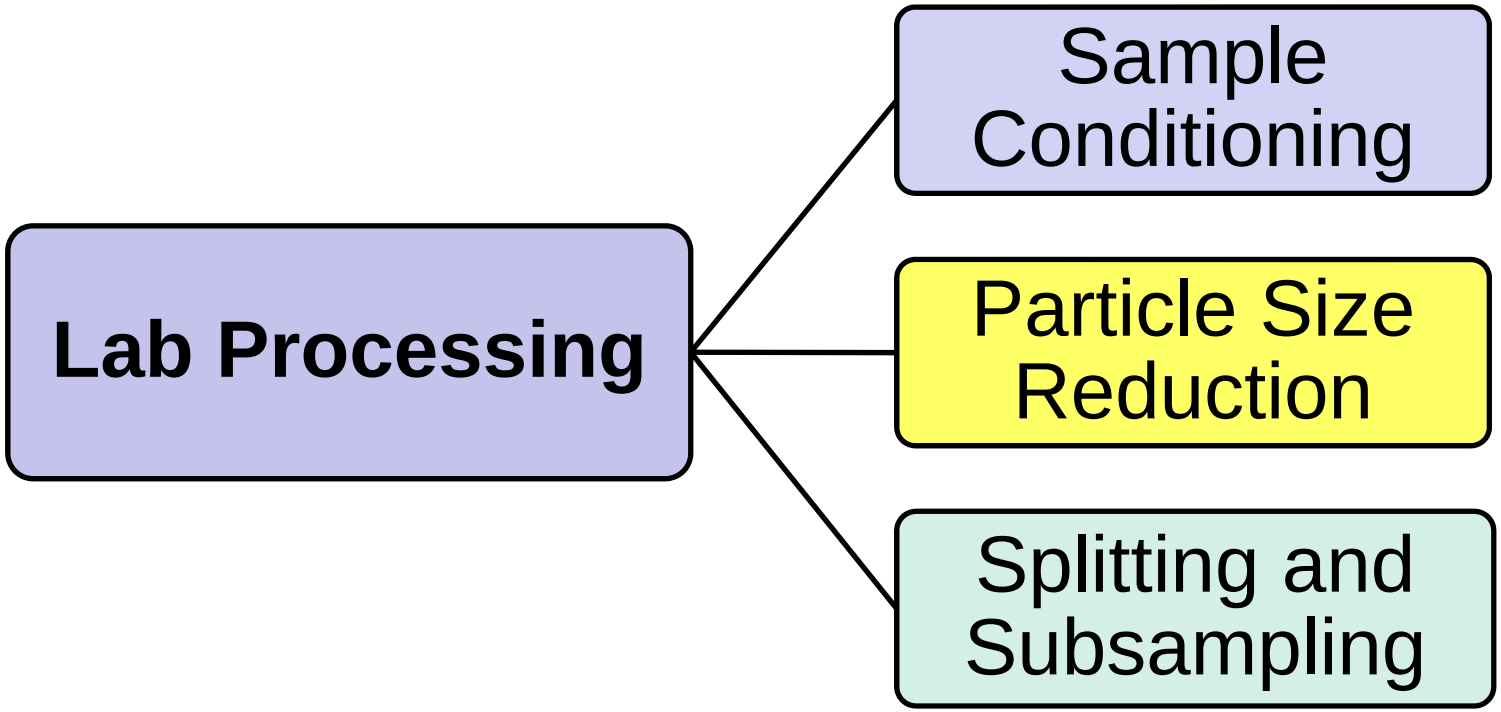
- Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. < 75 µm)

Picture from USACE-Alan Hewitt

Florida Case Study: Particle Size Reduction

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

Lab Processing Roadmap



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

- Recommended
  - Crystalline particles, fibrous threads, paint chips
  - Energetics, metals
- Strengths
  - Reduces variability
  - Reduces subsampling error
  - Facilitates mixing
  - Improves precision

Picture from USACE-Alan Hewitt

ITRC, ISM-1, Section 6.2.2.5

To Mill or Not to Mill

- Not recommended
  - Volatile, thermally labile, increased "availability"
  - Examples
    - Monochloro PCBs, reactive SVOCs, decane, elemental mercury
  - Limitations
    - Analyte losses
    - Metals contamination
    - Potential high bias to metals risk assessment (pebbles)

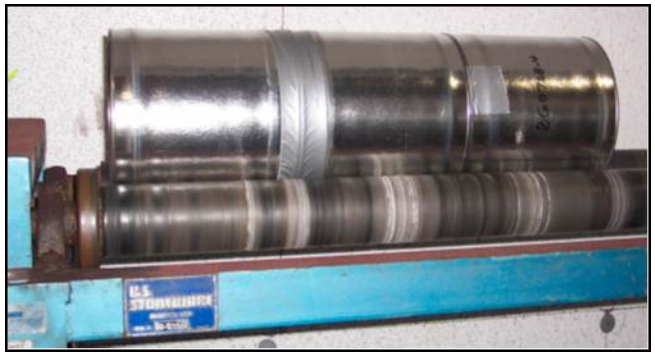
If uncertain, do milled & unmilled

ITRC, ISM-1, Section 6.2.2.5

How Best to Mill

- Puck mill or ring and puck mill
  - "Stable" energetics
- Ball mill
- Mortar and pestle
- Consider
  - Analytes
  - Concentration of interest
  - Mill materials
  - Particle size needed

Picture from USACE-Alan Hewitt

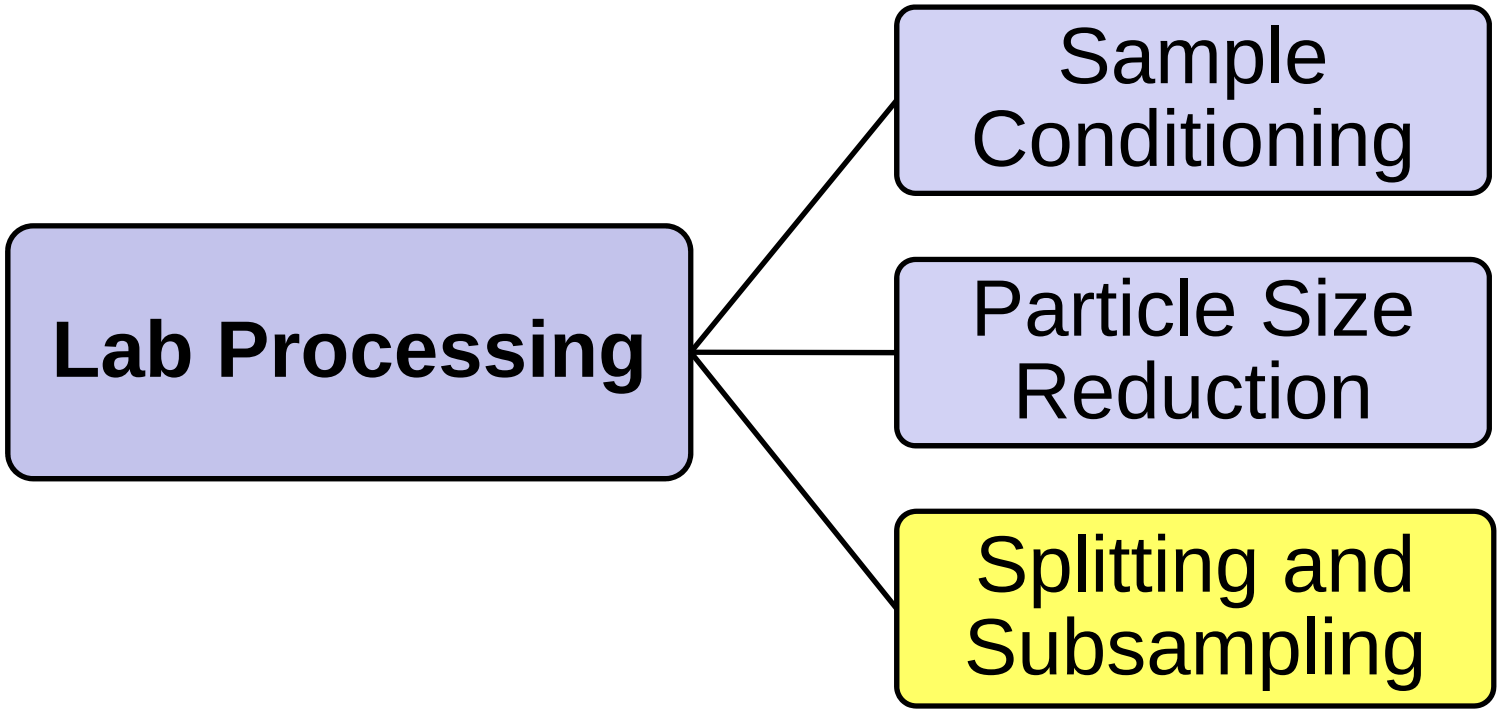


Example mills, other types are possible as well

Florida Case Study: Results Confirm Milling Not Needed

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

Lab Processing Roadmap



Dry Splitting Options

- Rotary sectorial splitter

Subsampling Options

- 2-Dimensional Japanese Slabcake

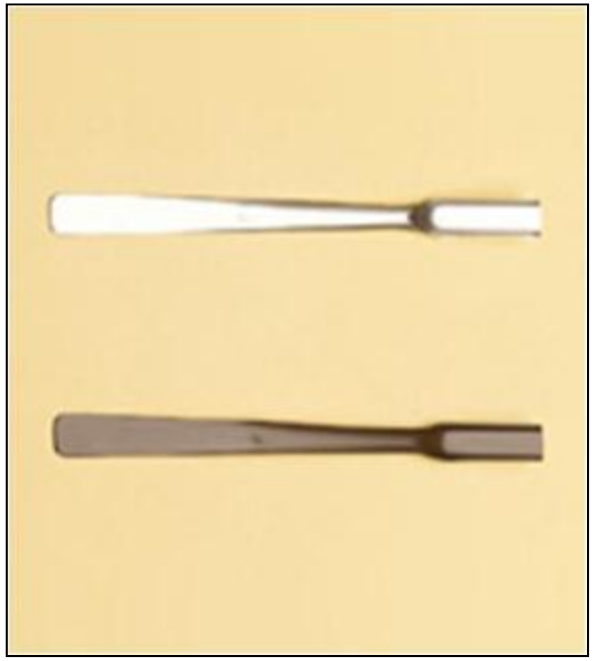
Dry

Wet

ITRC, ISM-1, Section 6.2.2.7

Subsampling Tools

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

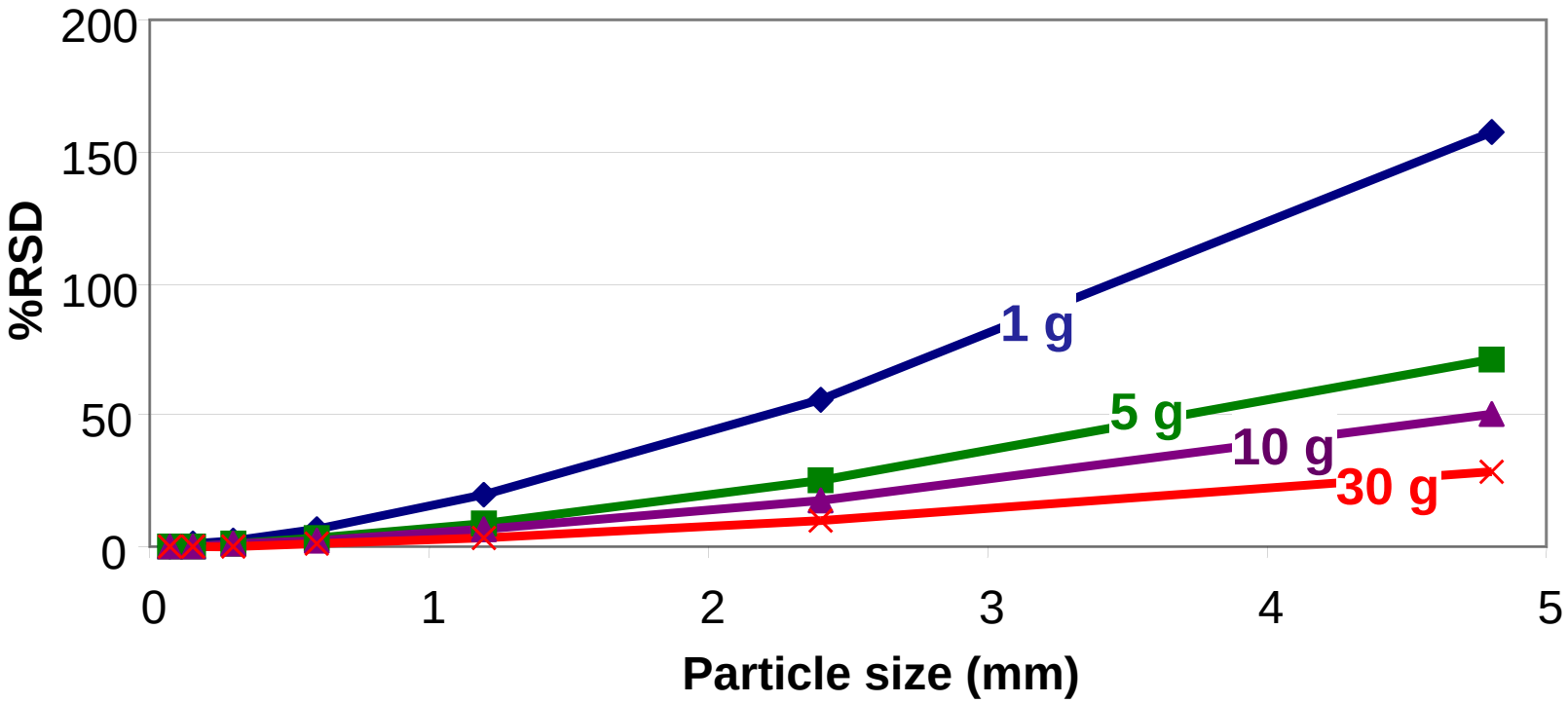


Florida Case Study: Choose Subsampling Process

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

Why Use Large Subsamples?

- Larger particles
  - Produce larger errors or require larger subsamples



ITRC, ISM-1, Section 6.3.3

Florida 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

- 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

Florida Case Study: Challenges with "Blank" Samples

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

Verify Laboratory Certification

- National Environmental Laboratory Accreditation Program (NELAP)

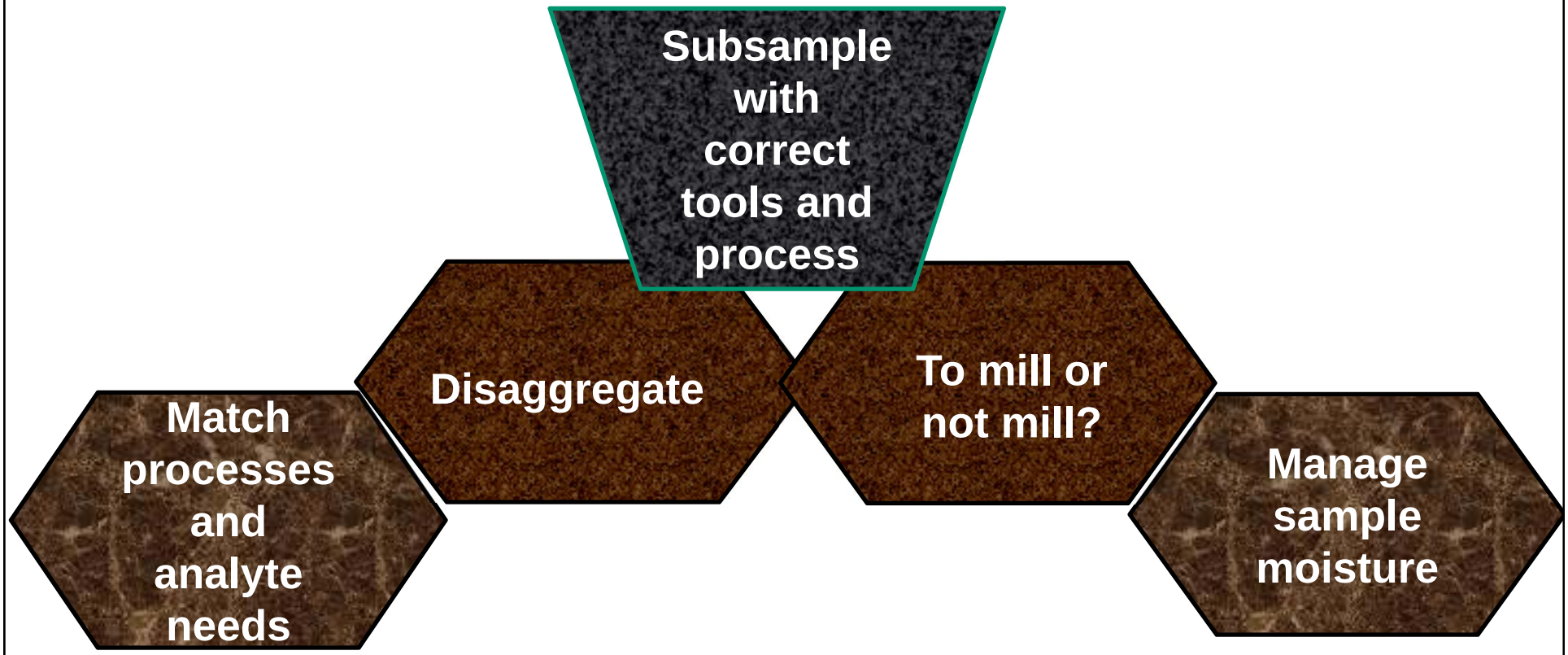
- Non-NELAP state accreditation

- Agency-specific accreditation
  - DoD Environmental Laboratory Approval Program

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?

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

Lab Process "Big Rocks"