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

**Mark Bruce Ph. D.**



August 15, 2011

# Chasing Uncertainty Sources

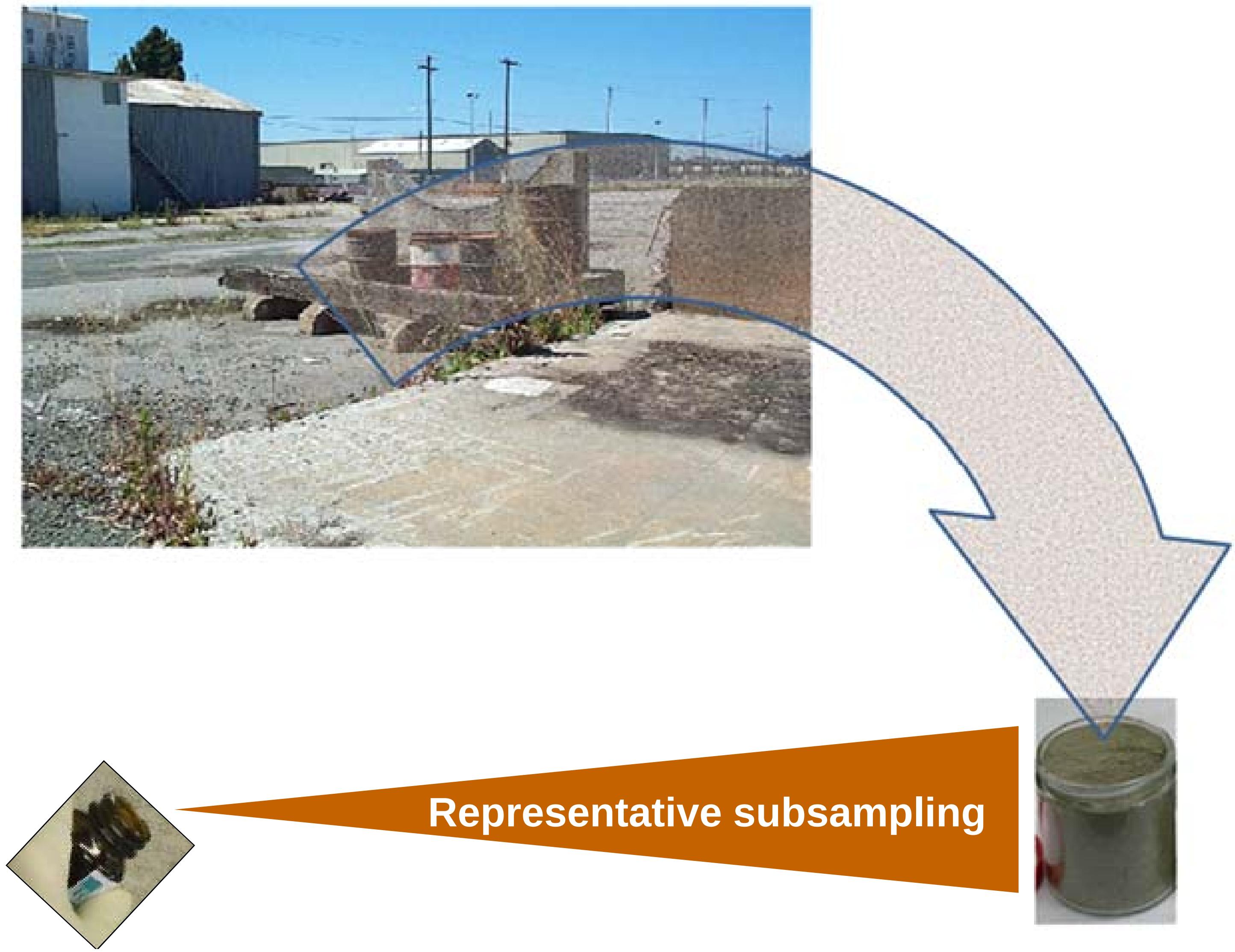
- Instrumental analysis

- Sample preparation

- Laboratory sub-sampling

- Field sample collection

# Does the decision unit fit in the sample jar?



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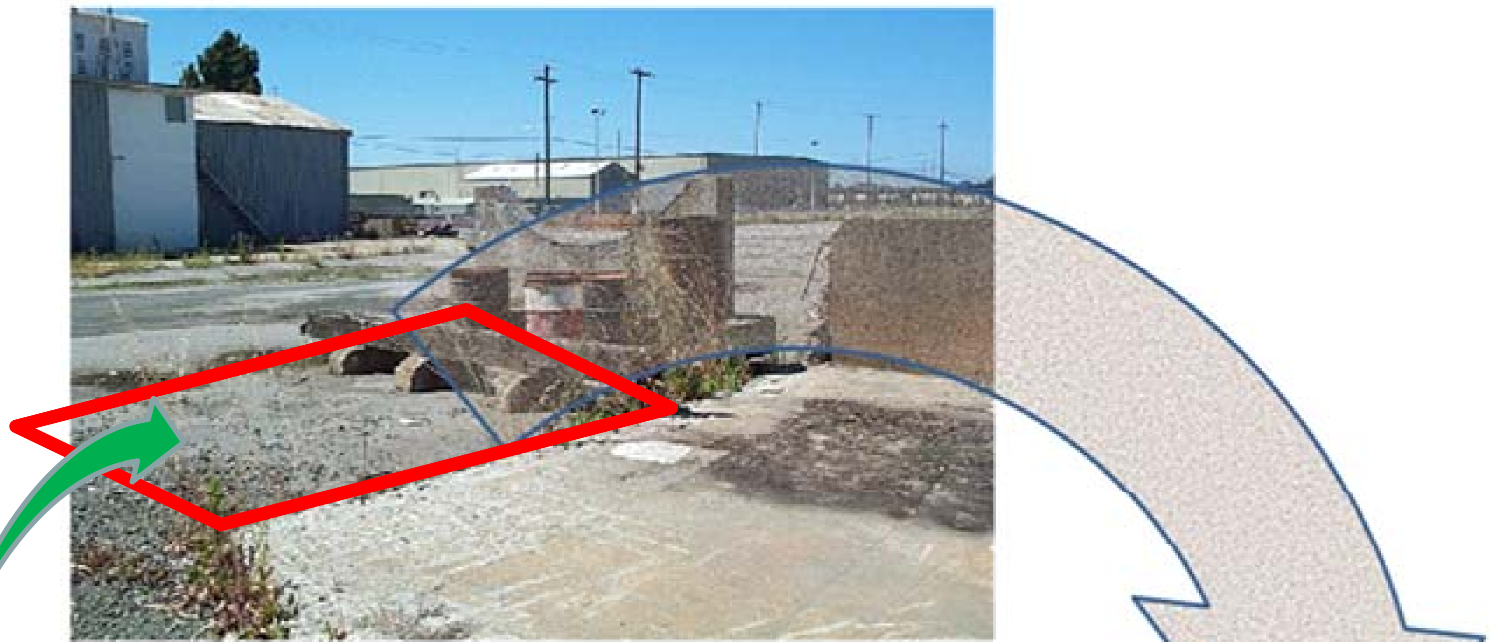
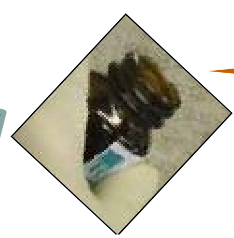
# Why is this important?



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

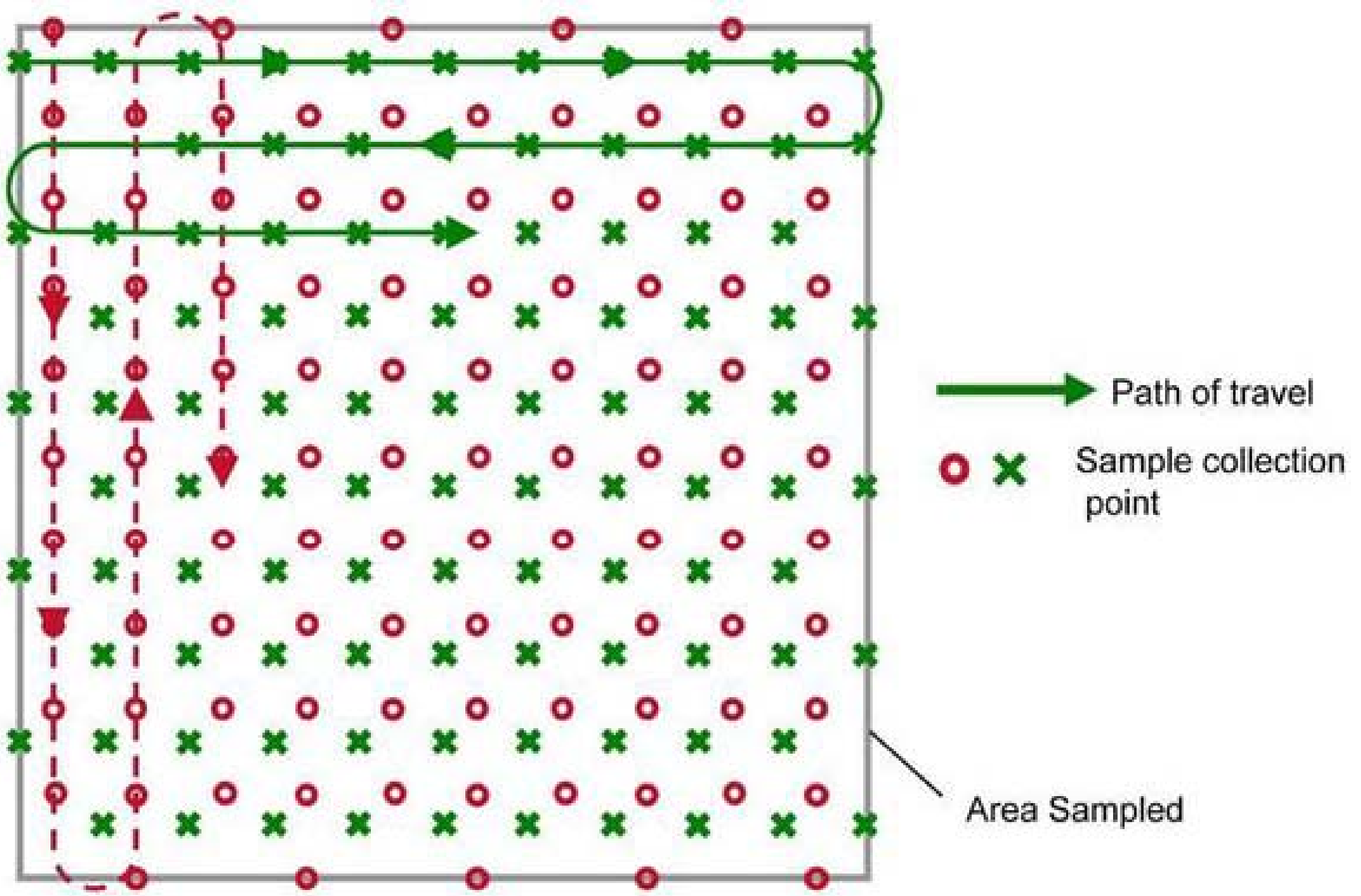




# Better Data Drives

# Incremental Sampling

- Systematic Random Design




Random starting locations in first grid

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# ITRC Developing Guidance

INTERSTATE TECHNOLOGY & REGULATORY COUNCIL
*Advancing Environmental Solutions*

- **Incremental Sampling Methodology Team**
  - ~ 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Disclaimer: Most of the material in this presentation has been derived from the Spring 2011 draft guidance developed by the ITRC ISM team.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.

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# ISM Guidance

INTERSTATE TECHNOLOGY & REGULATORY COUNCIL

*Advancing Environmental Solutions*

**Introduction**

**ISM Principles**

**Systematic Planning**

**Statistical ISM Design**

**Field Implementation**

**Laboratory Process**

**Making Decisions**

**Regulatory Concerns**

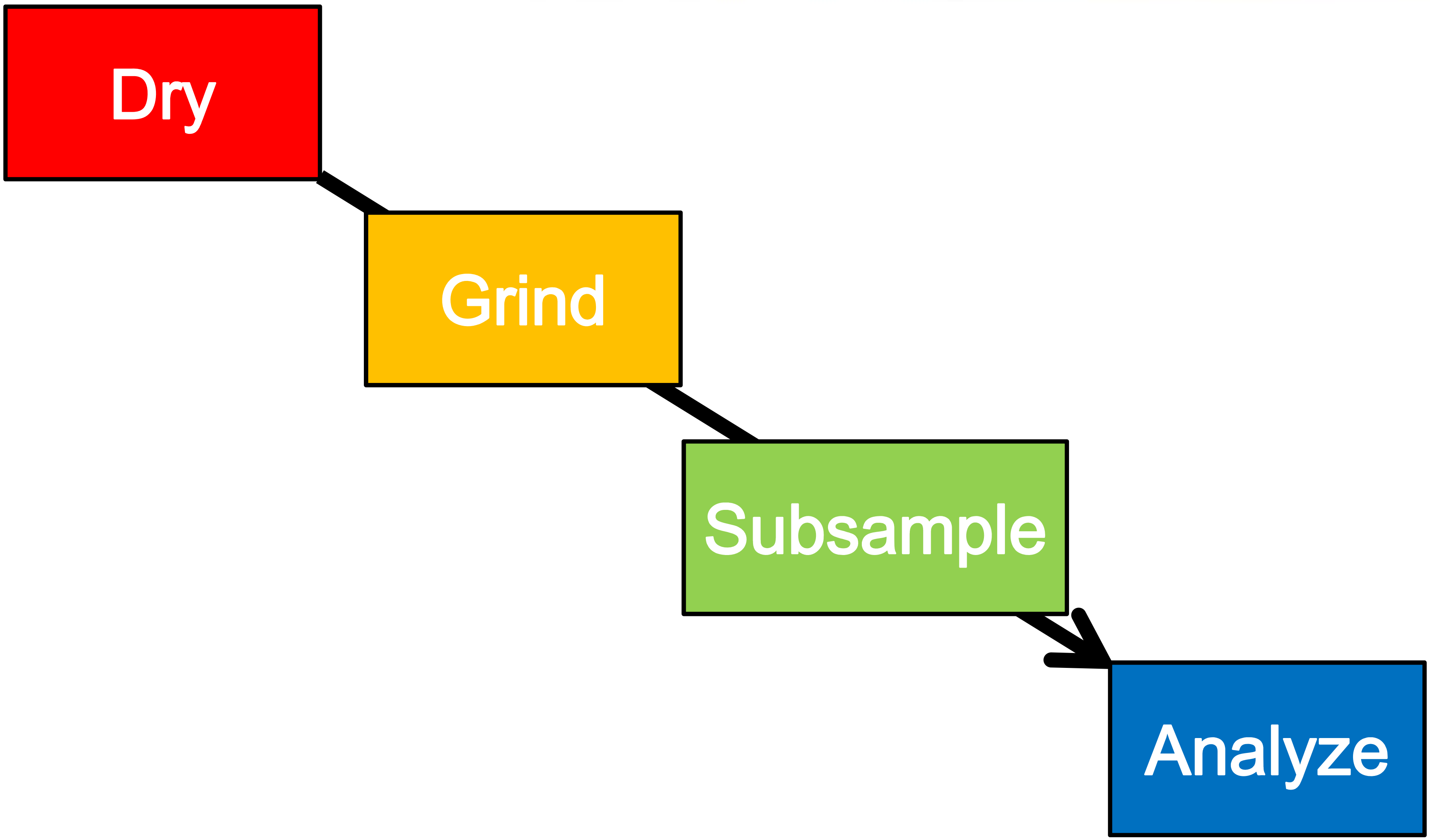
**Case Studies**

**Stakeholder Input**

# Laboratory Processing & Analysis

- Introduction
- Laboratory Processing
- Laboratory Analysis
- Quality Assurance

# No Universal Lab Sample Processing



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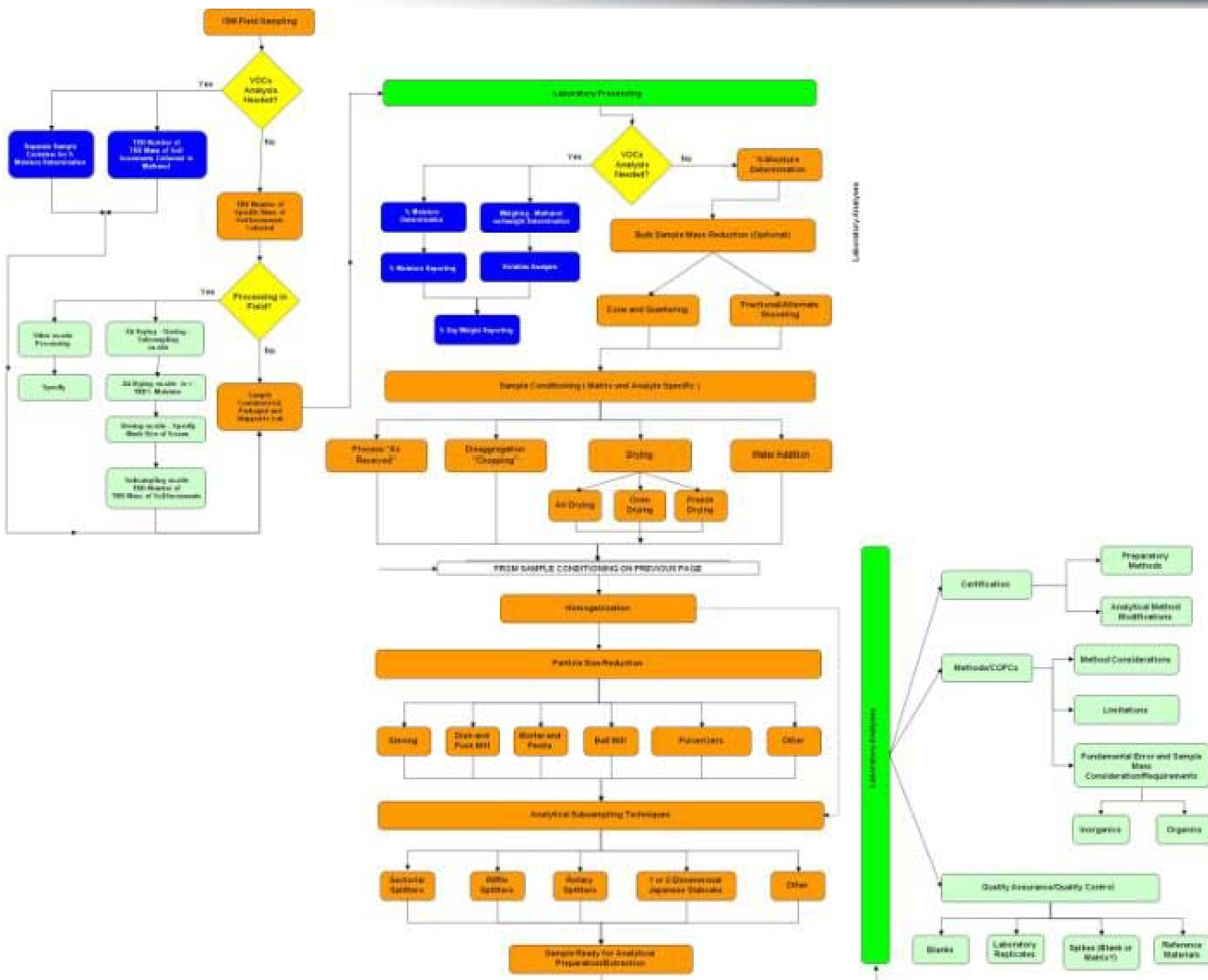
# Real Life ISM is Complicated



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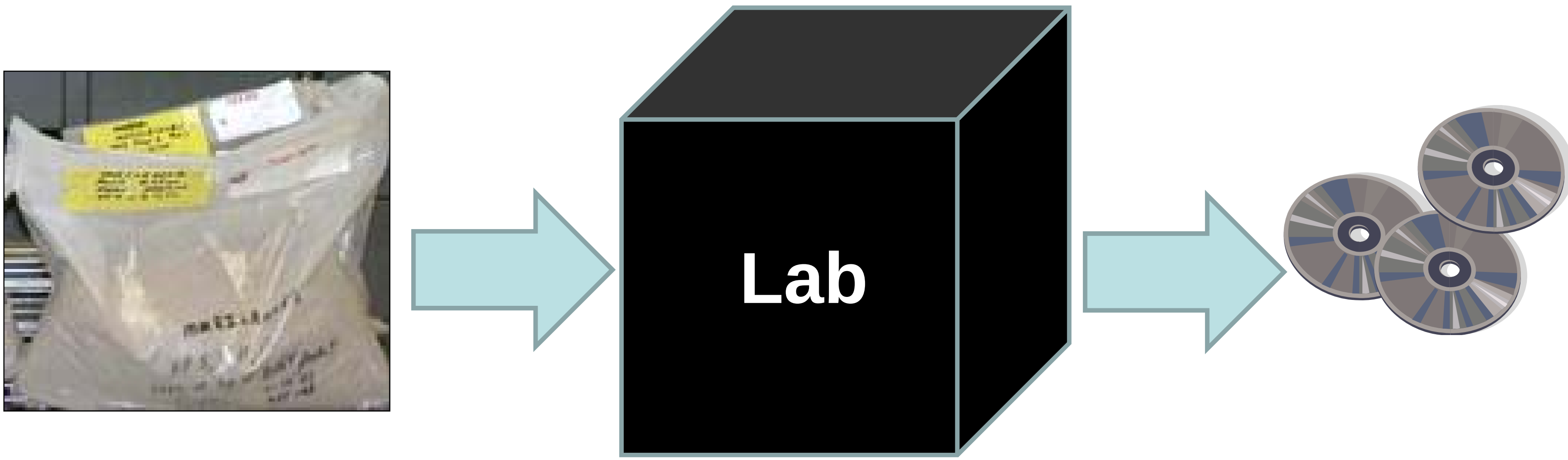
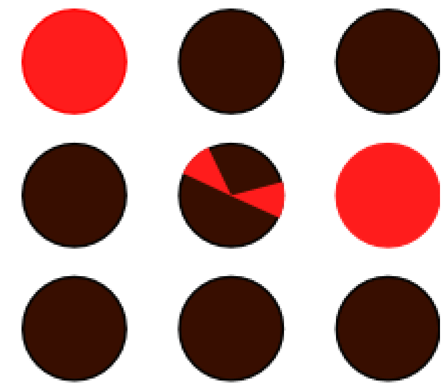
# Include Lab Processing in Project Planning

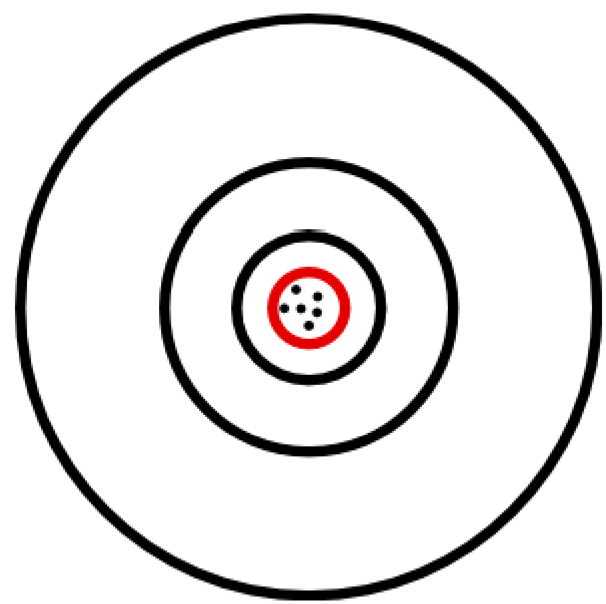


# Sample Processing Goals

- Goal: Improve subsampling representativeness



- Goal: Improve precision & minimize bias



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# Sample Processing Affects Data

- Always
  - ~ Improved precision
- Hopefully
  - ~ Improved accuracy (of single measurements)
    - Retain contaminants of concern
    - Avoid contamination
- Sweat the details or risk misleading data

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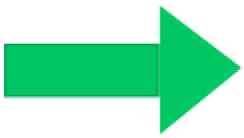
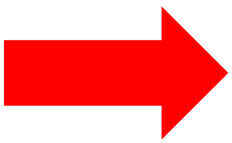
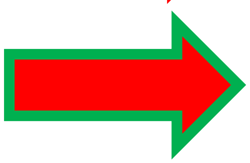
# Symbol key



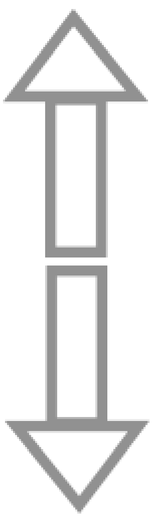
- Good effect



- Bad effect



- Good or Bad effect depending the question



- Result or statistic gets larger in value
- Result or statistic gets smaller in value

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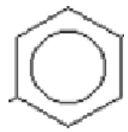
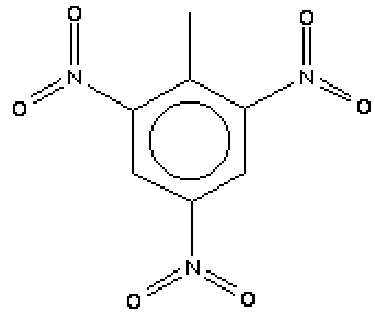
# Identify the Sample

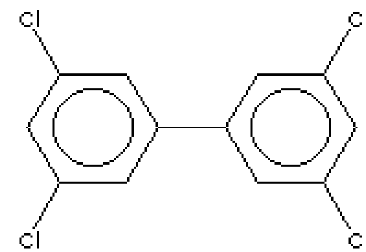
- Materials to remove
  - ~ Vegetation
  - ~ Oversized material
  - ~ Decantable water
- Manual removal
- Sieve (after drying)
  - ~ < 2 mm (#10)
- Lead source example
  - ~ Paint fines ↑
  - ~ Intact slugs ↓

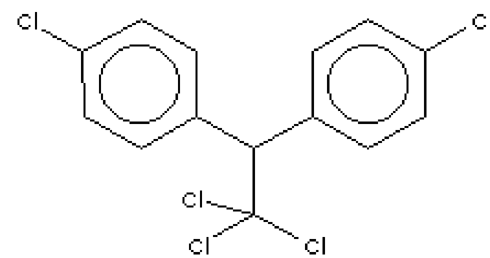
# Analyte Groups

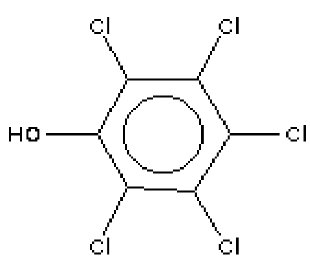
- Volatile organics
- Energetics
- Metals, Hg
- PCBs
- Organochlorine Pesticides
- Phenoxy acid herbicides
- Petroleum hydrocarbons
- Semivolatile organics

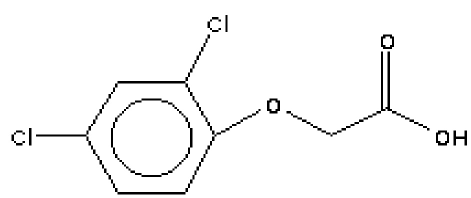


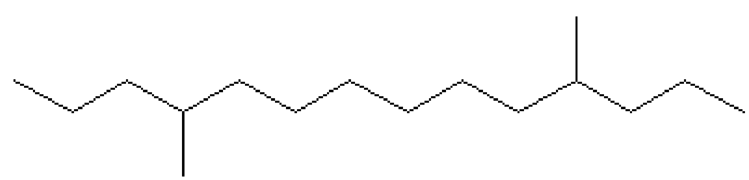




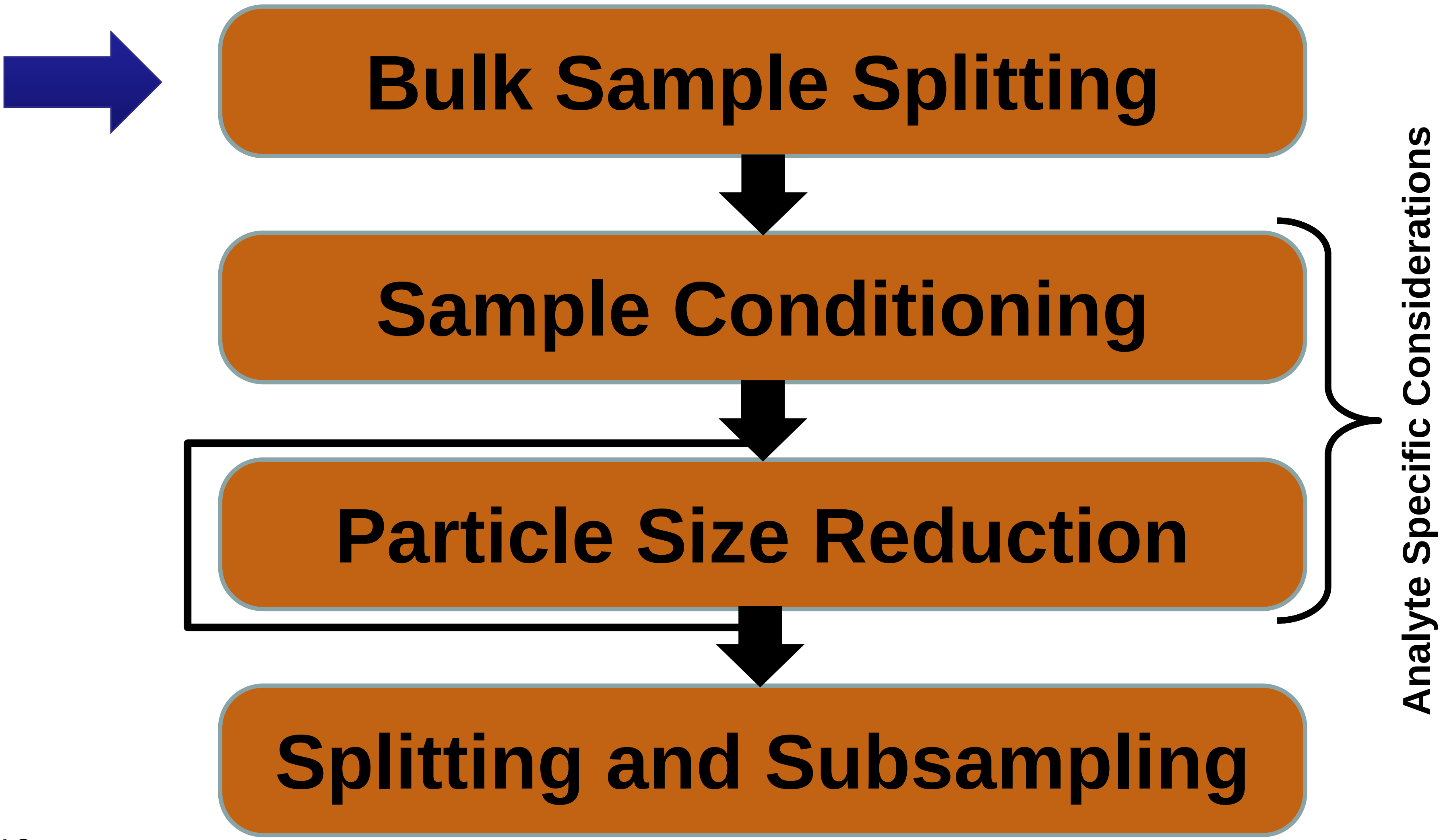








## Semivolatile Organic Compounds and Inorganics



# Bulk Sample Splitting

- Limited Applicability
- Sample Splitting for multiple analyte groups
  - ~ Alternate or fractional shoveling
  - ~ Consider “nugget” effect
  - ~ Increases fundamental error (variance)

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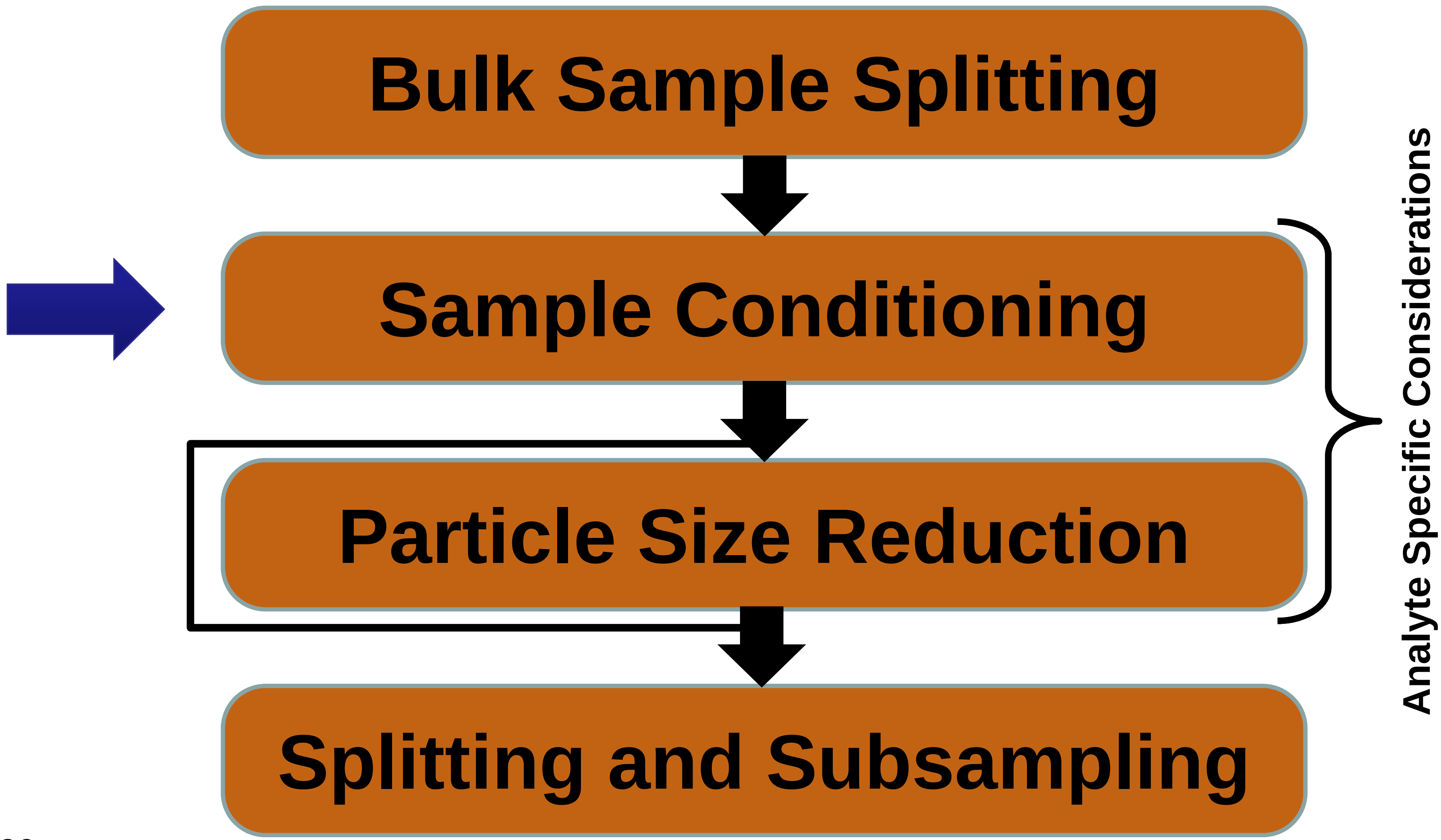
# Semivolatile Organic Compounds and Inorganics



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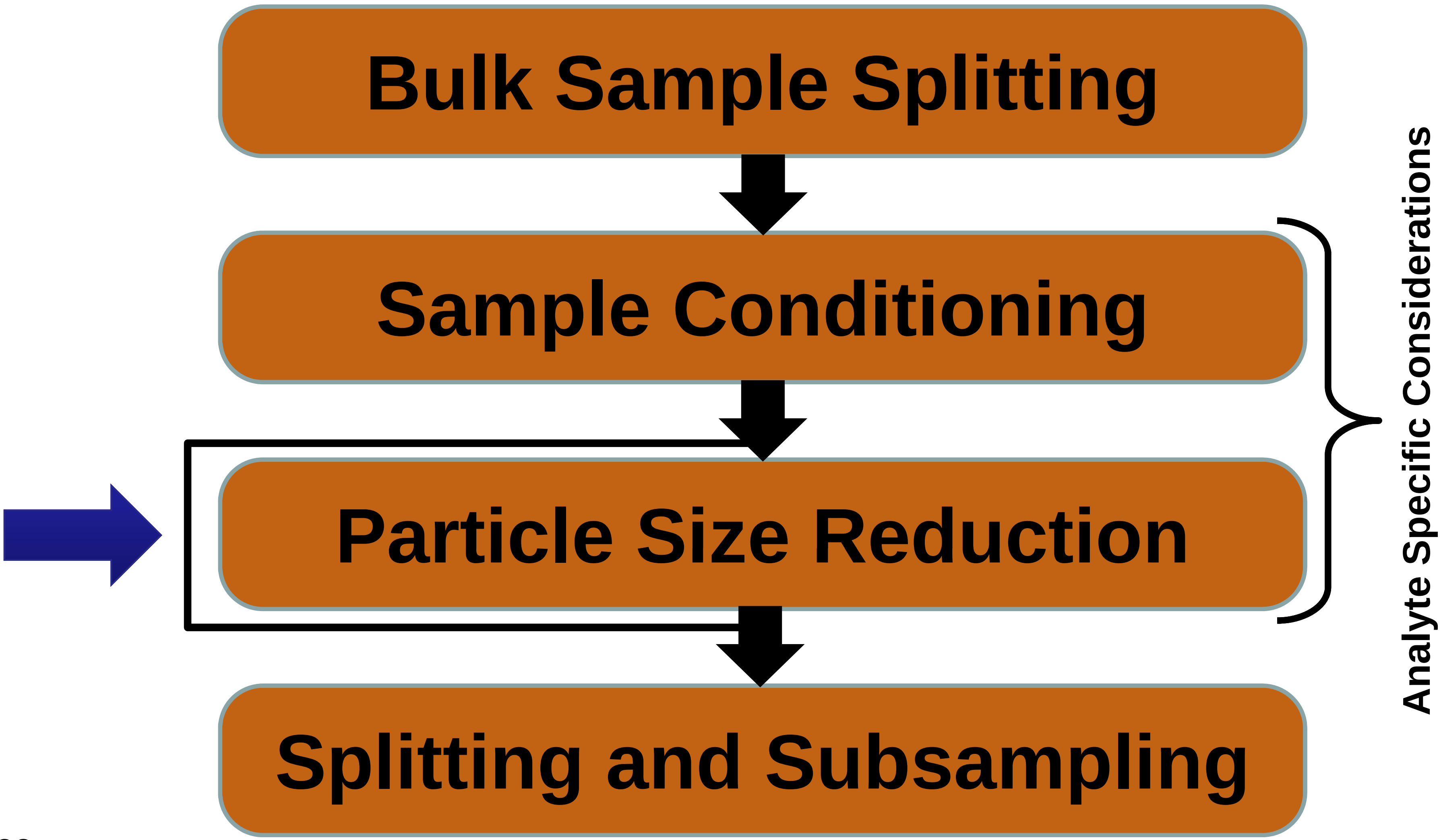
# Sample Conditioning

- 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 (R&D)
  - ~ Goal: Crushable agglomerates

# Sample Conditioning

- As-received
  - ~ Least air exposure
    - Fewest analyte losses
  - ~ Limits soil processing options
    - Fractional shoveling
    - Manual forced sieving

# Semivolatile Organic Compounds and Inorganics

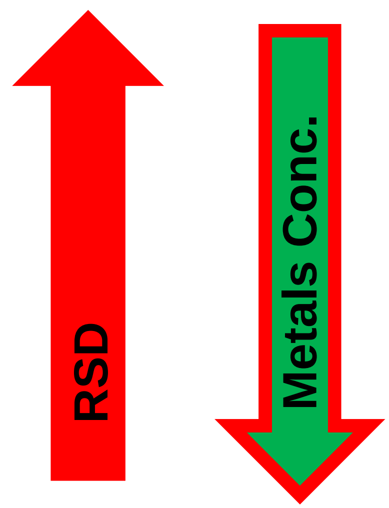


# Defining Terms

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

# Defining Terms

- Disaggregating:
  - ~ Breaking the soil clumps into individual small particles, but keeping the small pebbles and hard crystalline particles intact.


Compared to milling

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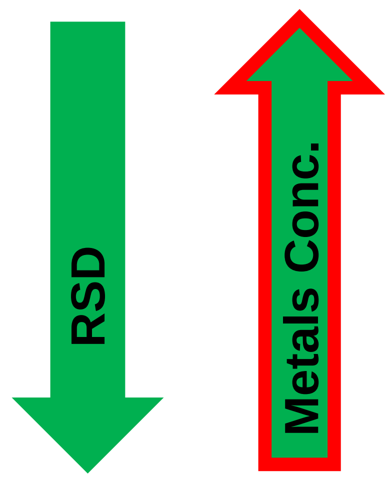
# Defining Terms

- 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).

Picture from USACE-Alan Hewitt

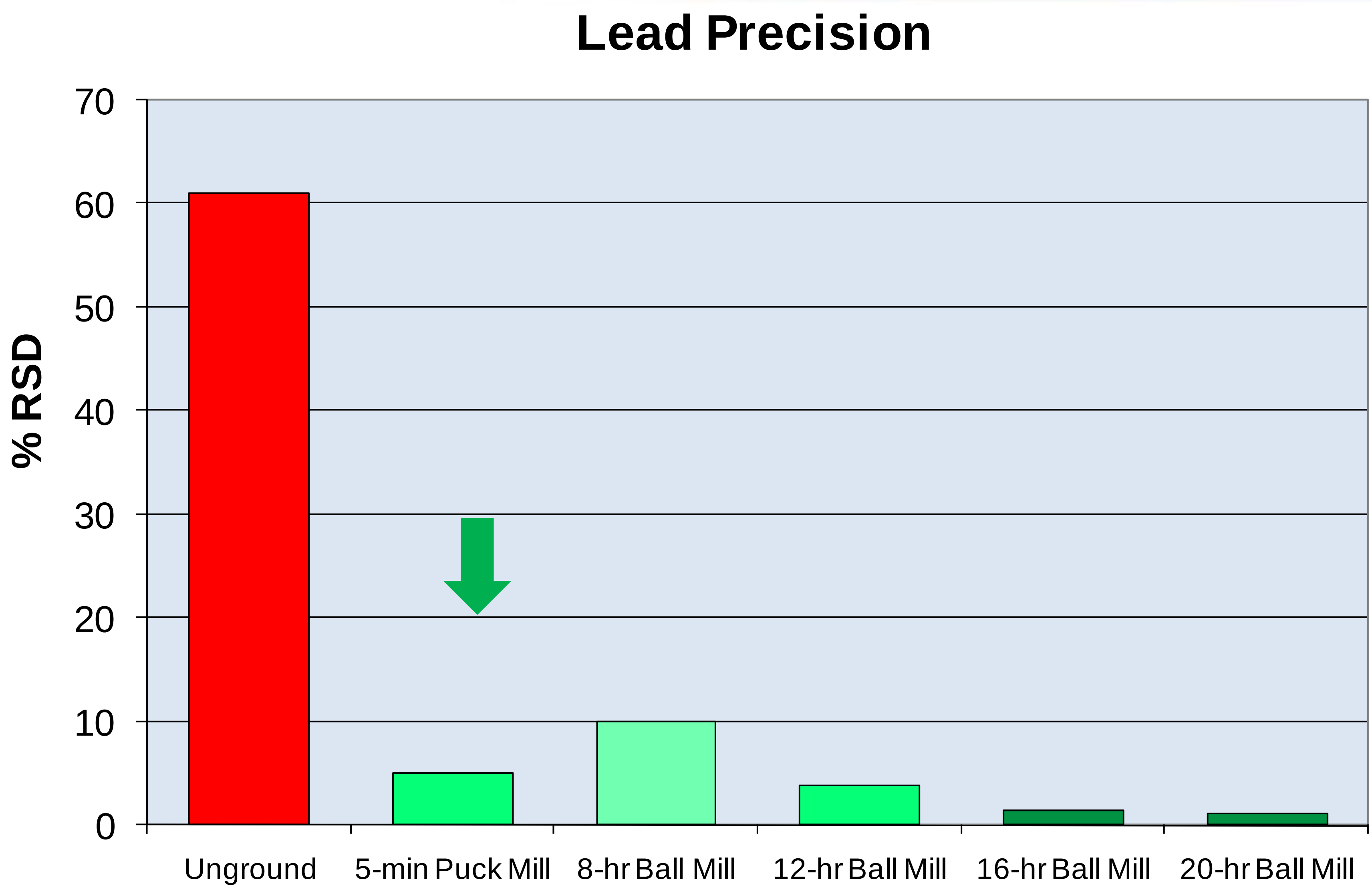



Compared to disaggregating

# 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



# 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)

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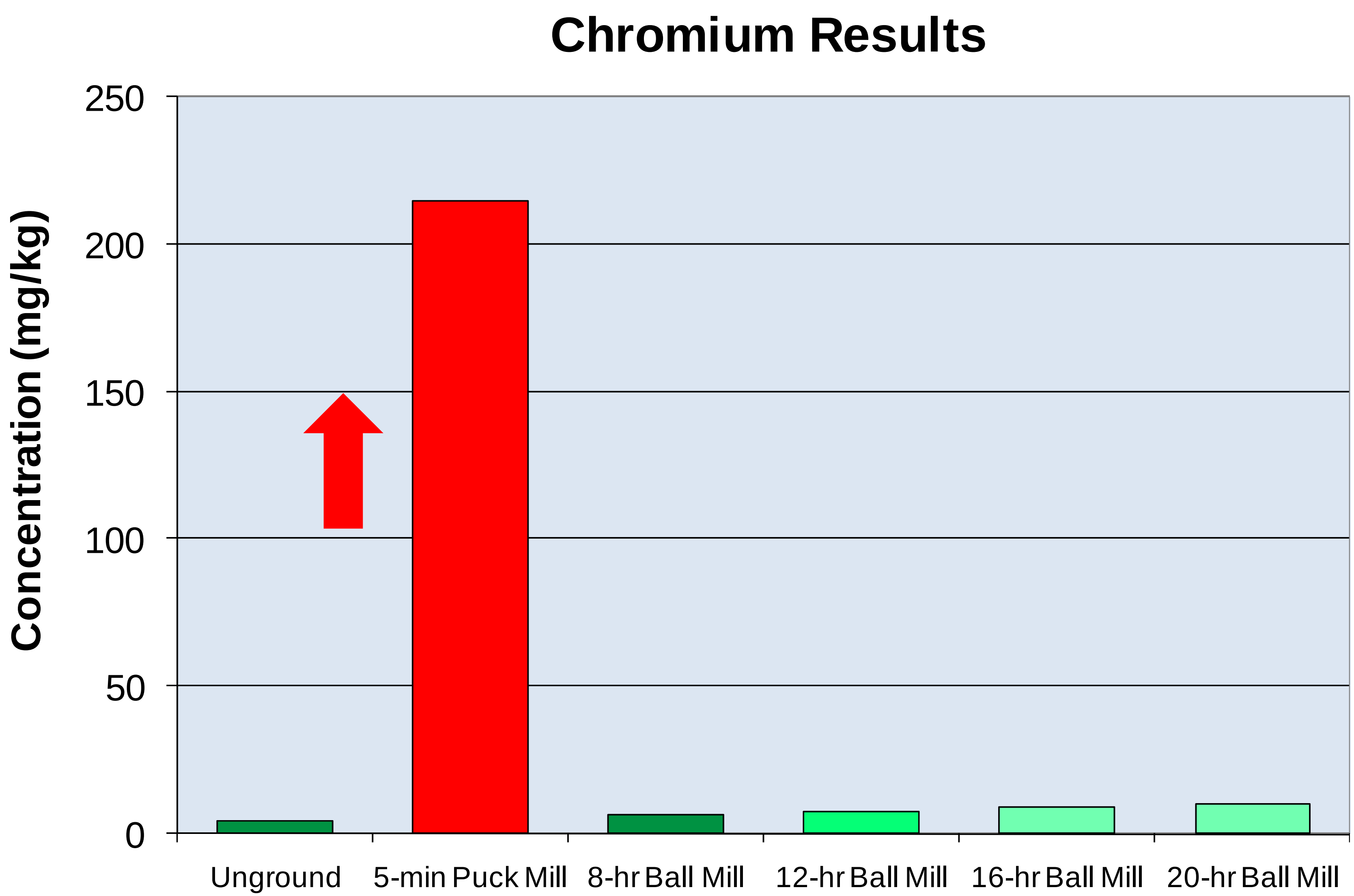
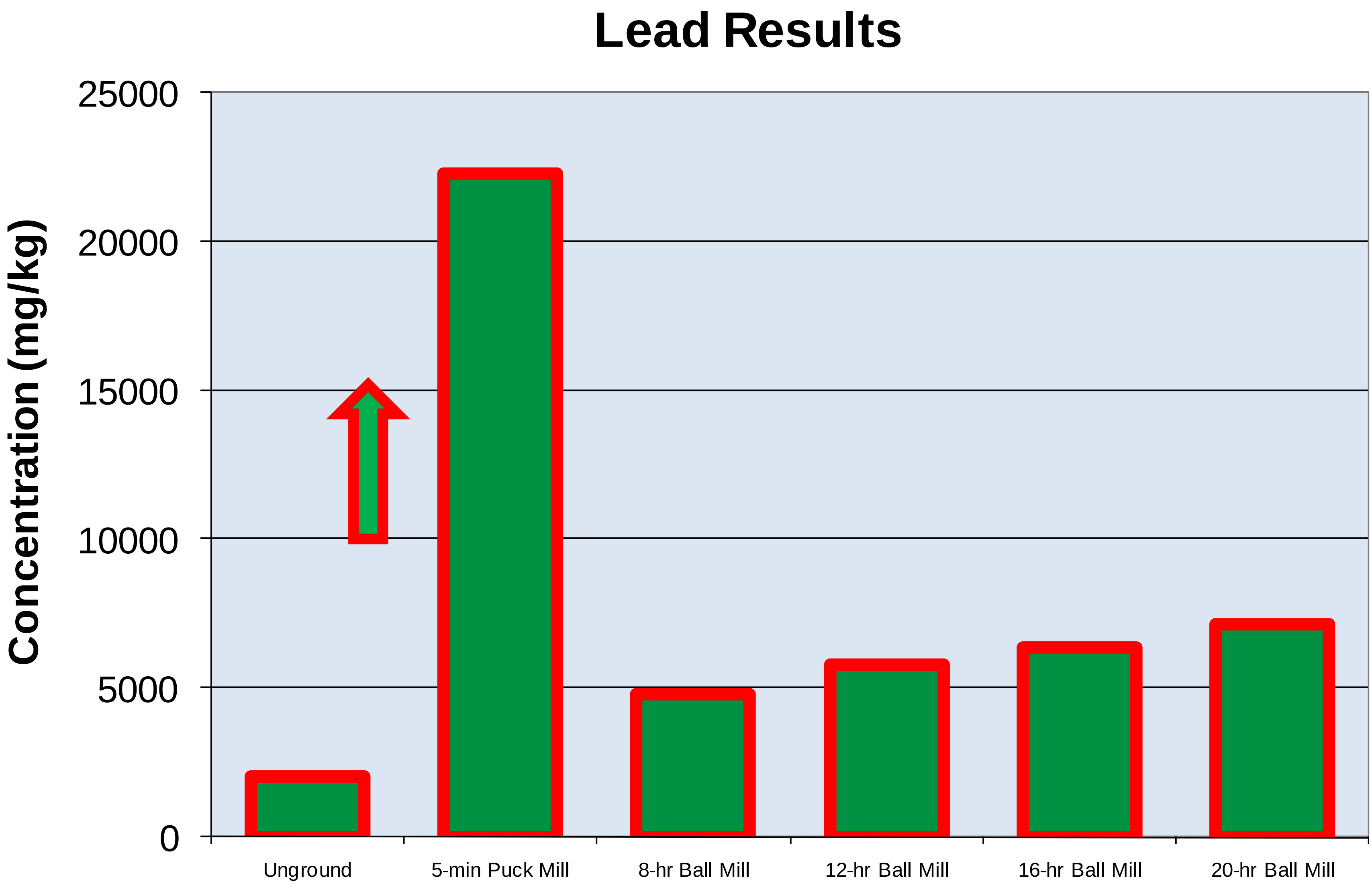
# Mill Erosion Elevates Cr Results



# Milling Releases Metals from “Pebbles”



# 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

Picture from USACE-Alan Hewitt

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# 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

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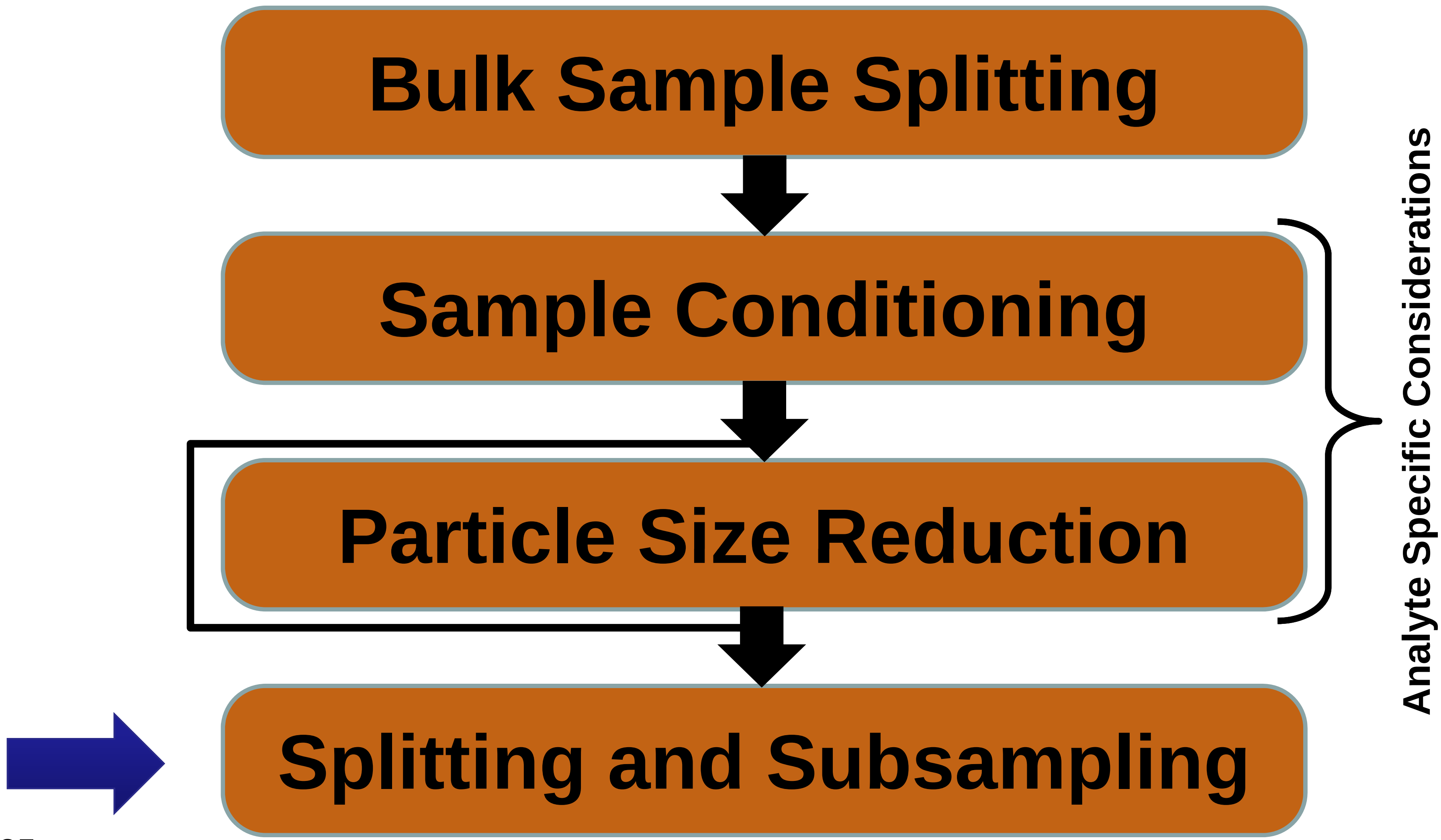
# Semivolatile Organic Compounds and Inorganics



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# Sample Condition Affects Subsampling Options

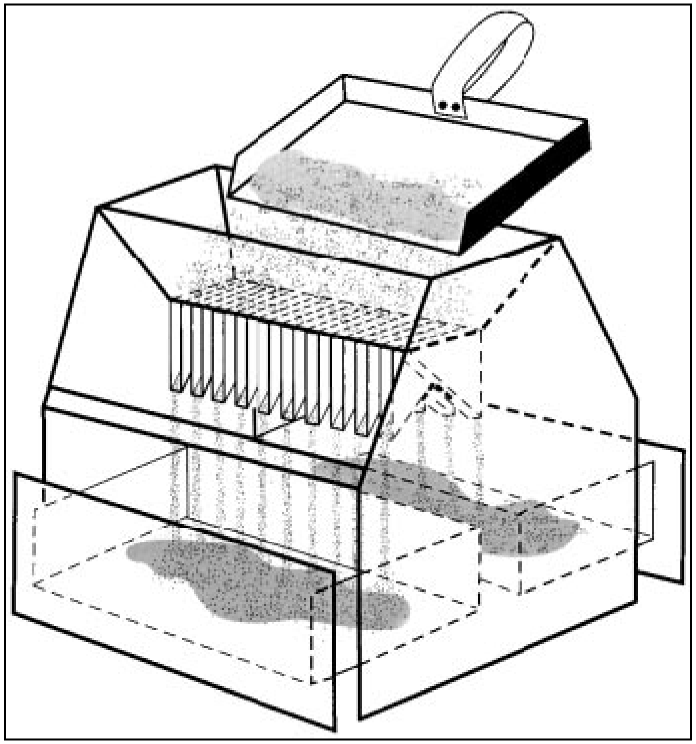
- Wet sticky sample
  - ~ Alternate shoveling
  - ~ Fractional shoveling
  - ~ 2 Dimensional Japanese Slabcake
- Dry flowable powder sample
  - ~ All splitting and subsampling techniques

# Wet Splitting Options

- Alternate shoveling
- Fractional shoveling

# Dry Splitting Options

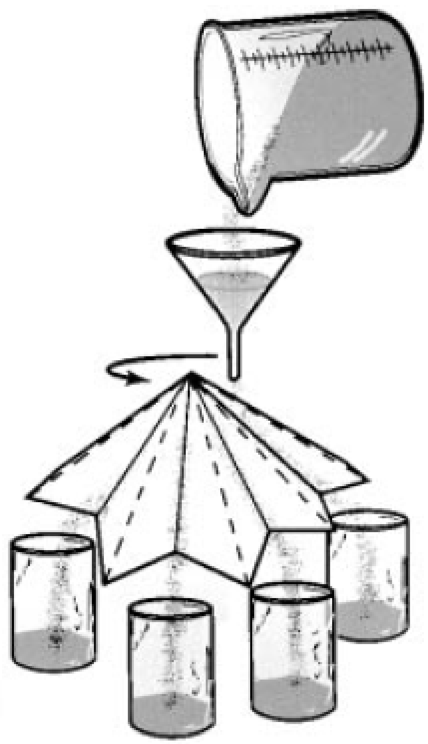
- Riffle splitter



**Gerlach,** *J. Chemometrics 2002; 16: 321-328*

# Dry Splitting Options

- Rotary sectorial splitter
- Paper cone sectorial splitter



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# Dry Splitting Options

- 1-Dimensional Japanese Slab Cake

# Sub-sampling Options

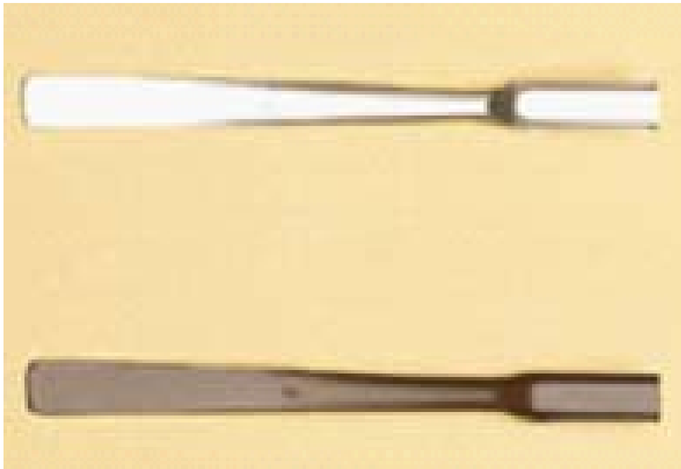
- 2-Dimensional Japanese Slabcake

Dry

Wet

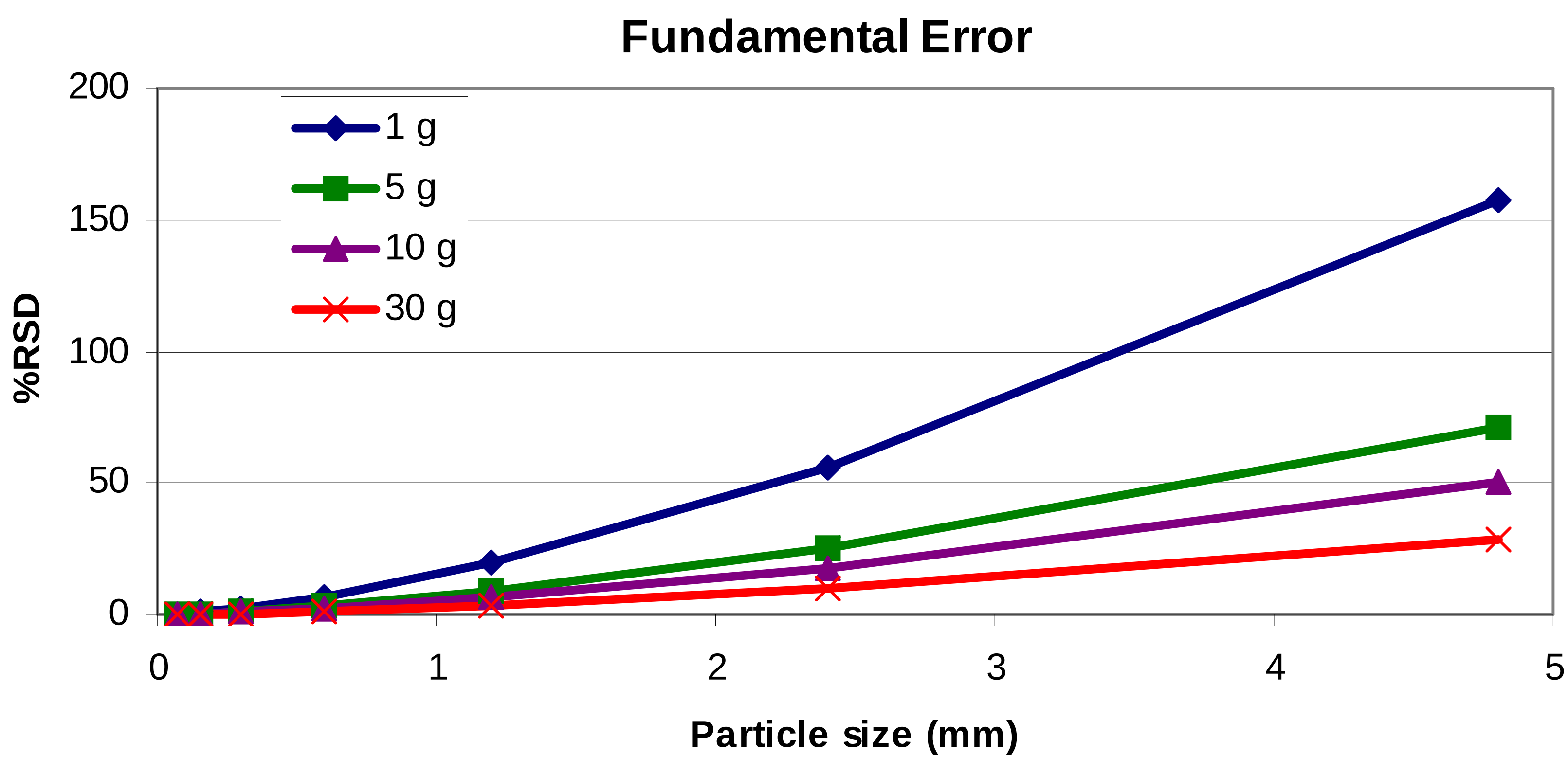
# Sub-sampling Tools

- Square straight sided scoops for dry non-cohesive soil



# Using large subsamples

- Larger particles
  - ~ Produce larger errors or require larger subsamples




ASTM D6323 Sec. A1.1

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# 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

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# 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

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# Matrix Options for Laboratory Quality Control Measures

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



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# Laboratory Certification

- National Environmental Laboratory Accreditation Program
- Non-NELAP State Accreditation
- Agency-specific Accreditation
  - ~ DoD Environmental Laboratory Approval Program

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# Reference Methods

- 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?

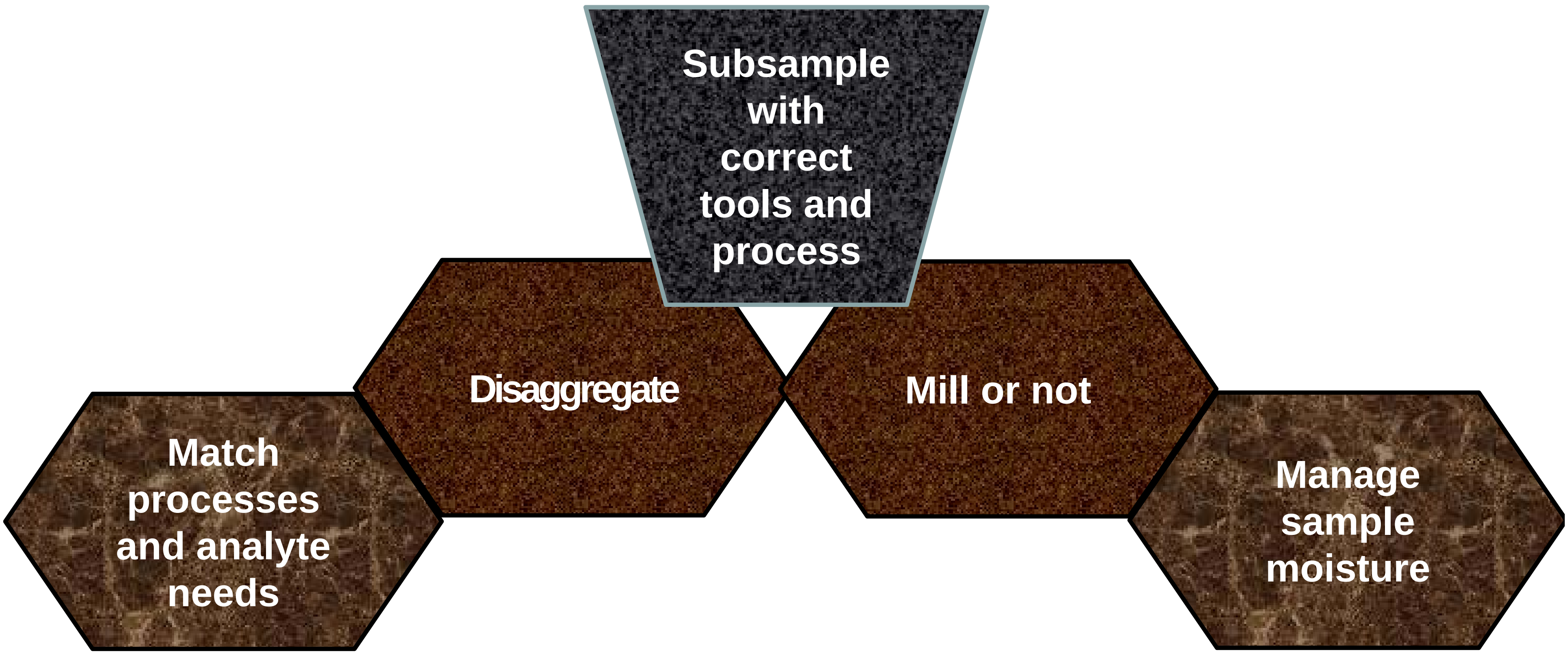
# Reference Methods

- 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"



# Guidance Document Projected Schedule

- Full ITRC (non-DoD) review – Early Q2, 2011
- DoD & EPA review – Late Q2, 2011
- Final to ITRC communications – Q4, 2011
- Tech. Reg. Publically Available – Q2, 2012
- Internet based training – Q2, 2012

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# Purpose of ISM

**Representative samples**

**Better data**

**Better decisions**

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# Acknowledgements

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## Contact Information

Mark L. Bruce Ph.D.
Technical Director

TestAmerica
4101 Shuffel St. NW
North Canton, OH 44720
Tel: 330-966-7267
Email: mark.bruce@testamericainc.com
www.testamericainc.com