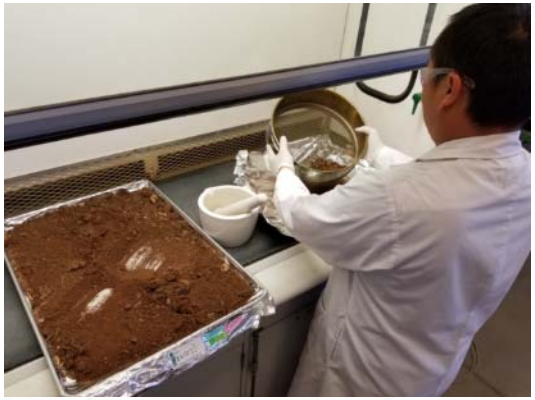
Lab Processing and Analysis of Multi Increment® Samples

Webinar begins November 15, 10AM HST



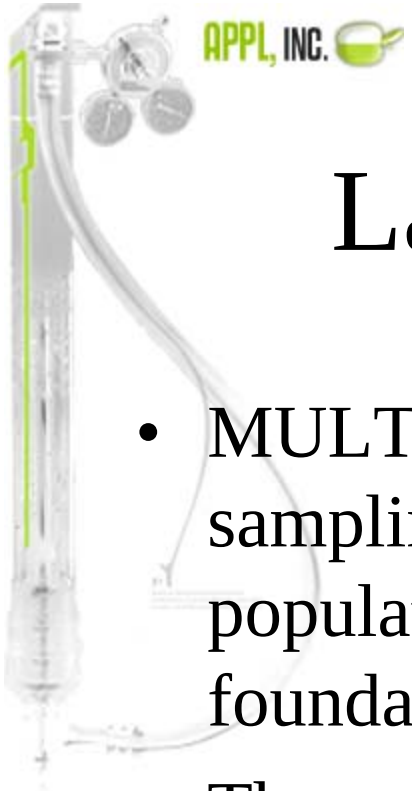


APPL, Inc.

- Agriculture and Priority Pollutants Laboratories Inc.
- In business for 35 years
- ISO IEC 17025:2005 accredited
- Department of Defense accredited
- NELAC accredited
- Certified in 10 states
- On the SW846 Methods contract from 1993-2010
- Served as a reference laboratory for various methods
- Started using *MIS* in 2006
- On ITRC committee for Incremental Sampling
- Chuck Ramsey “Accredited”

Where *MIS* has been used by APPL

- Hawaii
- 24 additional states
- EPA Region 3
- Afghanistan
- Denmark
- Japan
- Pacific Islands including Guam and Saipan
- Puerto Rico

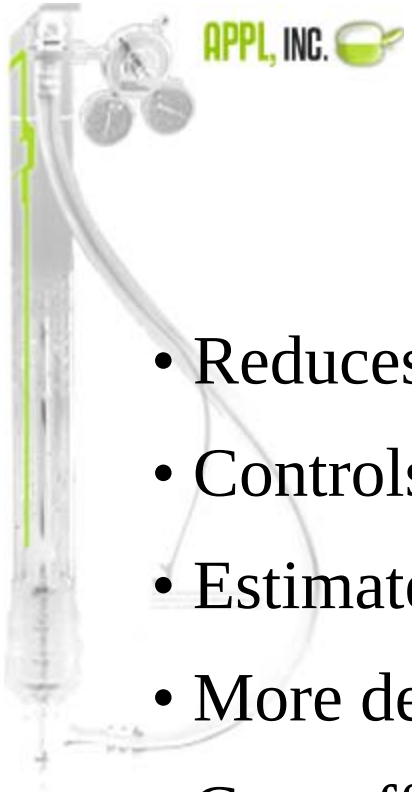


Laboratory's Role in *MIS*

- MULTI INCREMENT® – a comprehensive sampling methodology used to represent a specific population (decision unit) and provide a foundation for defensible decision making.
- The process used is the result of proper planning based on Data Quality Objectives
- *MIS does not stop in the field!!!*

Sampling Theory

- Applies to both field and laboratory
- Based on the fundamentals of Gy's sampling theory
- Staff trained prior to performance
  - Basic Concepts
  - Sources of Measurement Error
  - Fundamentals of Gy's theory



Why Use *MIS?*

- Reduces error
- Controls sample variability (error) due to heterogeneity
- Estimates of mean less uncertain and closer to true mean
- More defensible because it is based on science
- Cost-effective
- Discrete sampling methods are unreliable
  - In general underestimate contamination

Discrete Subsampling Issues

- Sometimes stirred first
  - Causes finer particles to settle to the bottom
  - Subsample taken from top of the container
- Insufficient increments to compensate for heterogeneity
- Insufficient mass to compensate for fundamental error
  - EPA Methods for metals require **only** 1 gram
  - Other EPA Methods also have insufficient mass
  - Likely to underestimate contaminant concentrations

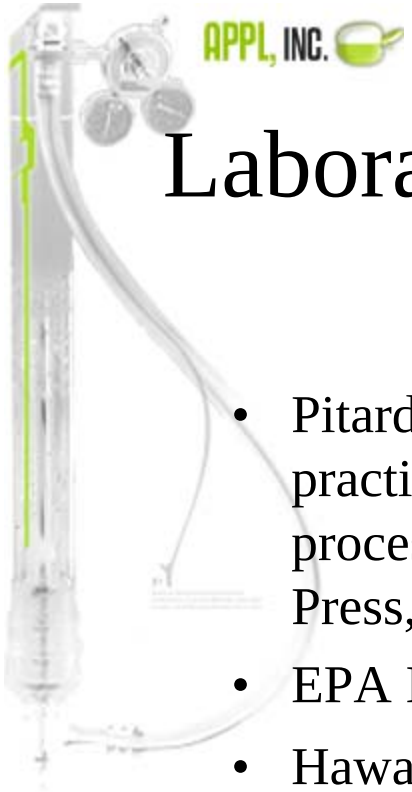
Typical Discrete Sample

MIS is Representative of the Entire Sample

I am looking for……..

Data that represents my entire sample!

Sampling Tools



Laboratory Processing and Subsampling References for *MIS*

- Pitard, Francis F. Pierre Gy's sampling theory and sampling practice: heterogeneity, sampling correctness, and statistical process control / author, Francis F. Pitard. - 2nd ed. 1993 by CRC Press, Inc.
- EPA Method 8330B, Appendix A USEPA SW846 2006
- Hawaii State Department of Health (HDOH), Hazard Evaluation and Emergency Response Office (HEER Office), Technical Guidance Manual – see Section 4. (On-line document, first posted in 2008 and updated last in 2017). www.hawaiidoh.org
- Interstate Technology & Regulatory Council. Incremental Sampling Methodology. ISM-1. http://itrcweb.org/ism-1/Executive Summary.html. February 2012

Implementing *MIS in the Lab*

- Start with Data Quality Objectives (DQOs)
  - What is the question we need to answer?
  - You need to convey your DQOs to the lab

Decision Unit (DU)

- Must be completely defined
- For the field
  - Arial extent
  - Depth
  - Particle size
  - Organic/inorganic material
- The decision unit is the same in the field and in the laboratory

Analyte Integrity

- Must be maintained from collection through analysis (reporting)
  - Compounds of interest
  - Concentration of concern (action level)
  - Detection Limits
  - Sample handling
  - Preservation Techniques

Resources

- Coordinate with your regulator!
  - Understand what they want...This is critical
  - You only have one attempt
    - If you make a mistake…..you resample!!!
- Coordinate with your lab
  - Lab MUST process and subsample properly or your field sample design effort is wasted

Choosing a Laboratory

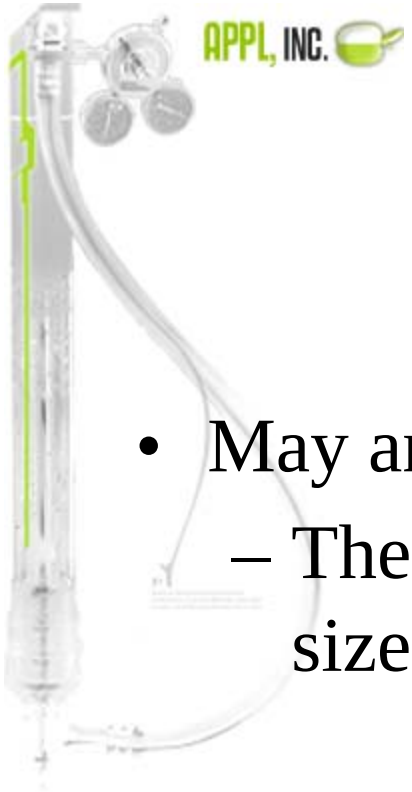
- What Questions Should I be Asking?
  - What kinds of sample processing do you do?
  - Can you provide SOPs for procedures?
  - What kind of QC do you perform?
  - What kind of equipment do you use?
  - What kind of references do you have?
- Schedule an on-site visit!
- Trust but verify!

How Can the Lab Help Me?

- Answer questions, ask questions
- Explain what will the processing do to my compounds of interest
- Explain the equipment used and how it may affect the samples
- Preservation techniques
- Proper container selection
- Provide pictures for documentation

What Particle Size Do I Need?

- Soil
  - Generally defined as particles <2mm
- Bioaccessiblility
  - For Lead and Arsenic prior to 2016 <250um
  - Updated by the EPA in 2016 to <150um for ingestion
- Particle size separation with different size sieves
- Mechanical grind with puck mill for particle size reduction
  - Results in particle size of <75um



Particle size

- May analyze more than one particle size fraction
  - The DU may contain more than one particle size (various receptors)

- May need to answer multiple questions
  - Bioaccessibility
  - Totals

Particle Size Separation

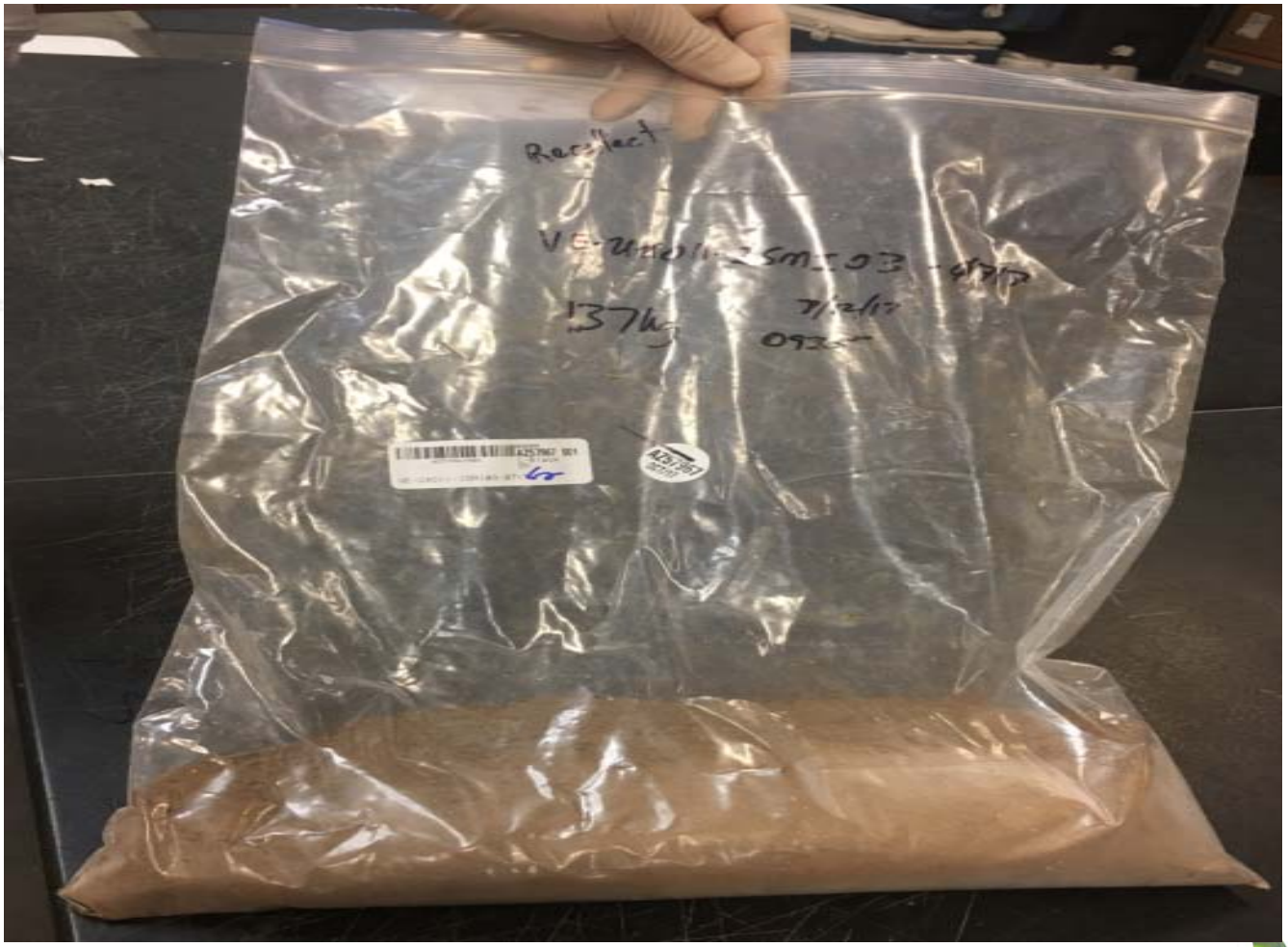
Particle Size		U.S. Std. Sieve	
Inches	Microns	Std. Sieve	Opening in inches
0.1570	4000	5	0.1575
0.0132	3350	6	0.1319
0.1110	2820	7	0.1102
0.0937	2380	8	0.0929
0.0787	2000	10	0.0787
0.0661	1680	12	0.0669
0.0555	1410	14	0.0551
0.0469	1190	16	0.0465
0.0394	1000	18	0.0394
0.0331	841	20	0.0335
0.0278	707	25	0.028
0.0234	595	30	0.0236
0.0197	500	35	0.0197
0.0165	420	40	0.0167
0.0139	354	45	0.014
0.0117	297	50	0.0118
0.0098	250	60	0.0098
0.0083	210	70	0.0083
0.0083	177	80	0.0071
0.0059	149	100	0.0059
0.0049	125	120	0.0049
0.0041	105	140	0.0042
0.0035	88	170	0.00351
0.0029	75	200	0.003
0.0052	63	230	0.0025
0.0021	53	270	0.0021
0.0017	44	352	0.0018
0.0015	38	400	0.0015
0.0010	25	500	0.001
0.0008	20	635	0.0008

To Process Soil at 2mm

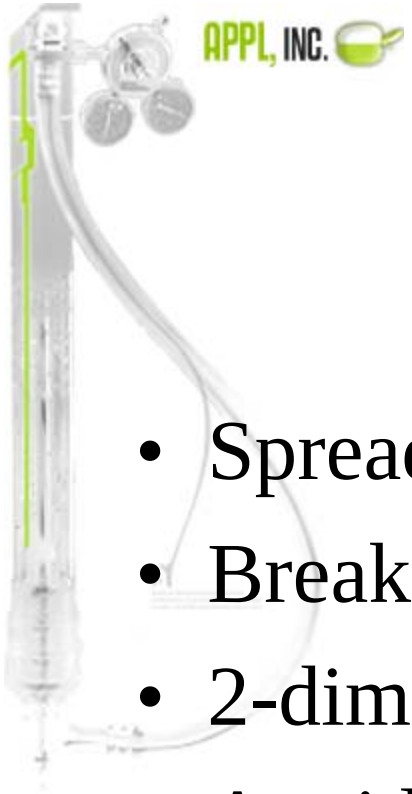
MIS Options in the Lab

- Literally hundreds of ways to process samples (and that's a good thing)
  - Dry or not dry
  - Sieve or not sieve
  - Grind or not grind
- Multiple stages of sieving, grinding subsampling
- Multiple ways to subsample

Approximately 1 to 2 Kilos

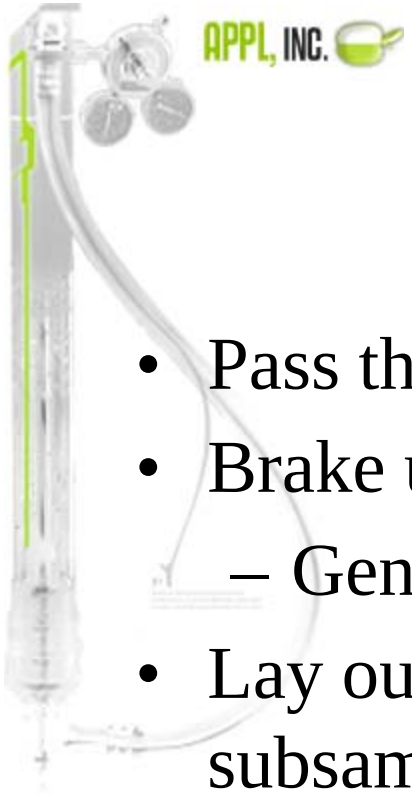


Drying the Bulk MI Sample



Wet *MI* Subsampling

- Spread entire sample evenly
- Break up clumps with gloved hand
- 2-dimensional slab cake min 30 increments
- Avoid selection of particles (gravel/sticks) that are obviously over 2mm
- Collect additional MI sample to determine moisture content of sample (up to 50 grams)



Sieving Without Drying

- Pass through a #10 sieve slowly
- Brake up dirt clods with gloved hand
  - Gently push sample through sieve
- Lay out sample as flat and evenly as possible and subsample immediately
- Subsample using 2-dimensional Japanese slab cake
- Collect additional MI sample to determine moisture content of sample (up to 50 grams)

Dry and Sieve (no Grinding)

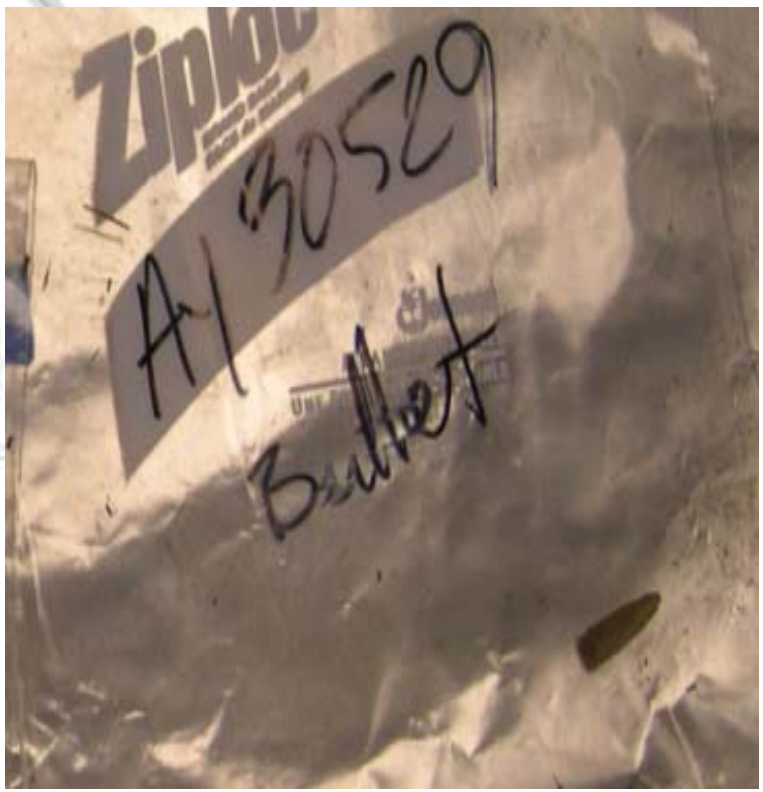
- Dry completely (typically air-dried)
- Sieve through #10 sieve (2mm)
- Or other sieve can be used
  - Depends on particle size needed
- Break up aggregates during sieving
- Mortar and pestle soil
- Only gravel and sticks left on sieve

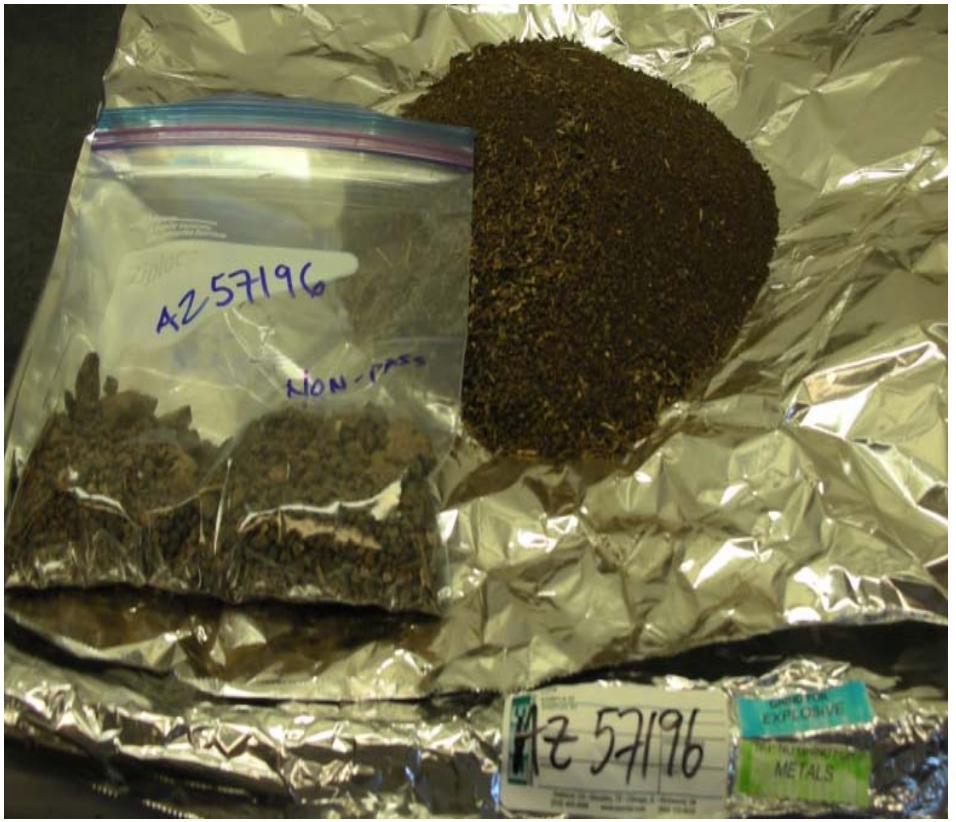
Dry Weight vs. Wet Weight

- In HI data for direct exposure risk based on dry weight
- Air dried samples meet requirement
- Wet and wet sieved samples will need moisture determination

Make sure the Lab Processes the Entire Sample

- This takes time and time is money!
  - And people can be lazy!
  - This is the biggest mistake labs make!
- We take pictures of every sample!
- How do you make sure the entire sample is processed? ASK!

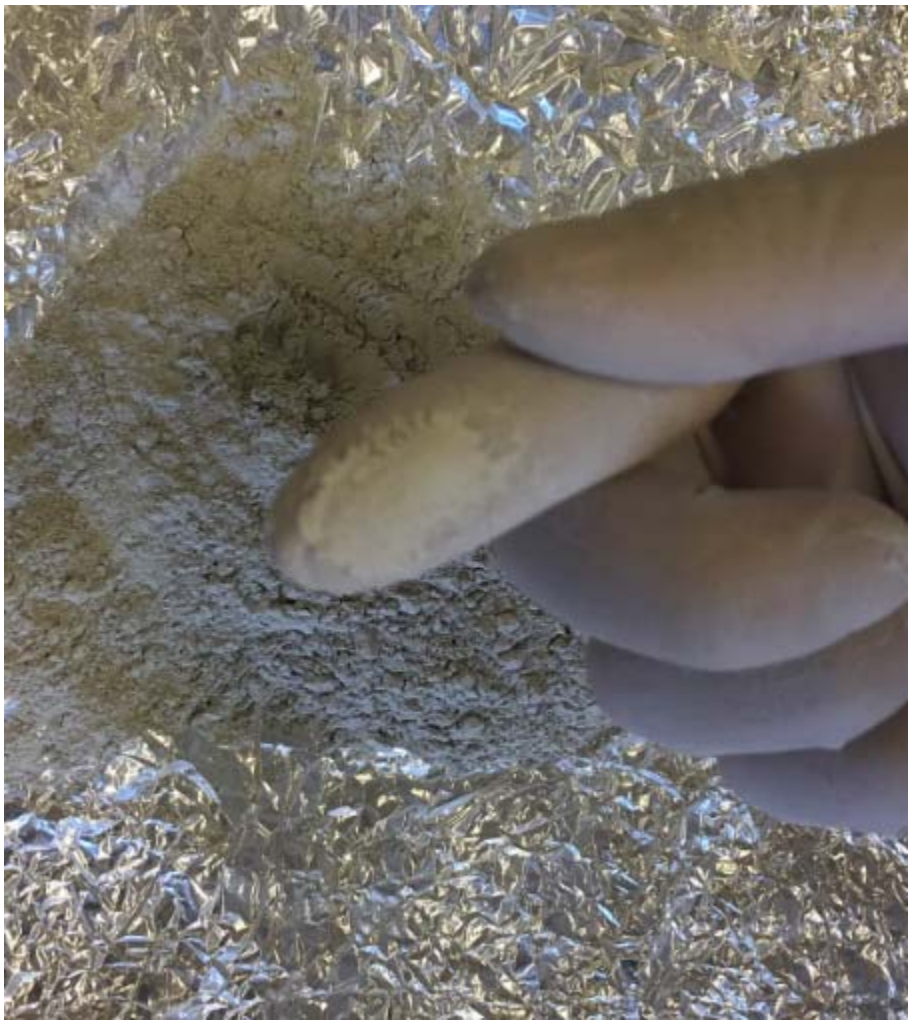

Ziploc
AZ 30529
Bullet


AZ57196
NON-PASS
AZ 57196
EXPLOSIVE
METALS

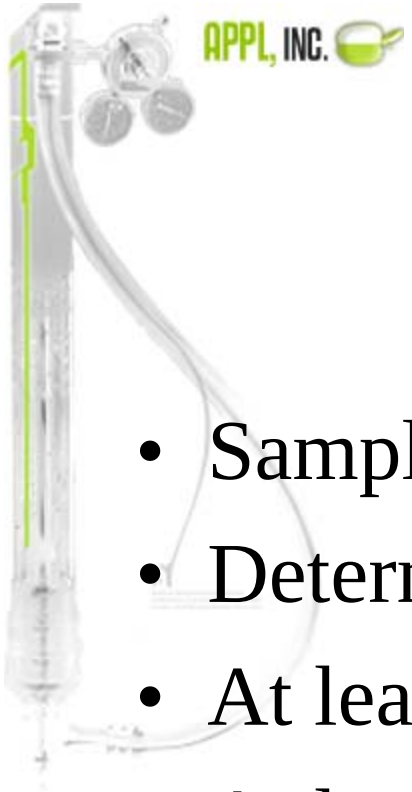
Sieving followed by Grinding

- Mortar and Pestle is not mechanical grinding
  - Not all Grinders are created equal
- Need proper equipment
  - APPL, Inc. uses a Puck Mill

Grinds to 75 microns



Blank Sand



Sample Mass

- Sample mass is important!
- Determine mass based on fundamental error
- At least 5 grams for Hg in HI
- At least 10 grams for metals in HI
- Grinding reduces fundamental error

Grinding Study

- Twenty-four soil samples were analyzed for metals pre- and post-grinding
  - Al, Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, Tl, V, Zn
- Only one metal showed statistically significant post-grinding concentration increases
  - Chromium (15.3 ppm)
- Results are consistent with other studies

QC to Demonstrate Reproducibility

- One triplicate recommended per batch
  - Maximum 10 in a batch
- Randomly chosen
- Compare results
- If %RSD greater than 20% contact client
  - May need to analyze a larger mass
  - May need to reanalyze or grind finer

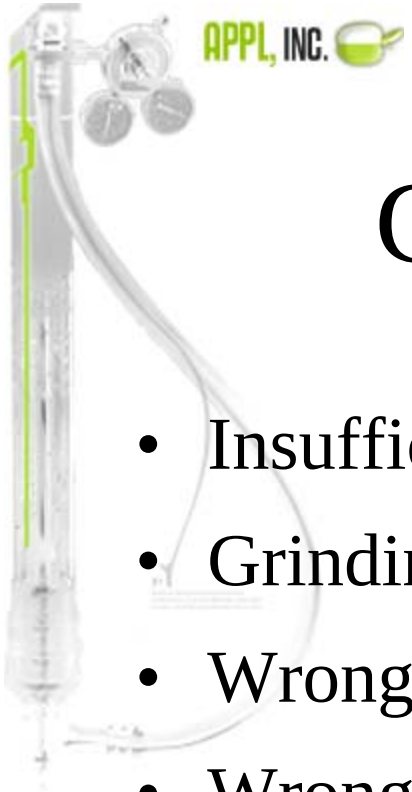
Sectorial Splitter

MIS for Volatiles

- Wide or narrow top containers available
- Max. 30 mL MeOH per container
- Max. 1L per ice chest
- 1:1 ratio of soil (g) to MeOH (mL)

Common *MIS* Mistakes

- Incomplete communication
  - From the client
  - From the lab
- Cutting corners during processing in the lab
  - Not processing entire sample
  - Not doing complete desegregation
  - Inconsistent subsampling
- Not grinding the sample at the appropriate stage
  - Need to *MIS* all unground aliquots before grinding
  - *MIS* before or after drying…or both?



Common *MIS* Mistakes

- Insufficient mass used to decrease sampling error
- Grinding times need to be specified and followed
- Wrong sieve size used/Sieve size not specified
- Wrong type of grinder used
- Poor communication between laboratory sections

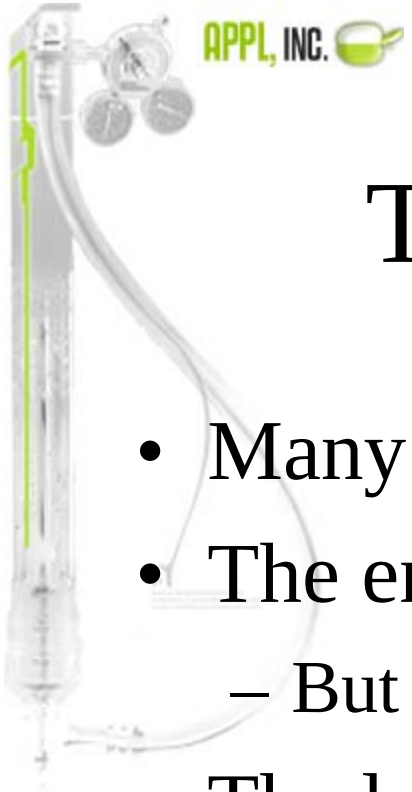
Lab Communication

Pros and Cons for Grinding

- Pro: Decreases fundamental error
- Pro: Need fewer increments
- Pro: Need less mass
- Pro: Improves precision
- Con: Loss of some analytes e.g. volatiles
- Con: Bio availability prior to grinding

Reporting Requirements

- Communicate reporting requirements
- Information in final report
- Size of sample submitted
- Sample preparation information
  - Include lab replicate data and analysis
  - Enough Information in final report
    - To understand entire lab process and data
    - For all analytes
    - To reference specific SOPs



Take Away Information

- Many processing possibilities with *MIS*
- The entire sample must be processed
  - But you must define the sample
- The lab must understand what is needed
- All samples are sub sampled incrementally
- 10% of samples should be run in triplicate

Additional Information

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- Roger C. Brewer, Sr. Environmental Scientist
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  - chuck@envirostat.org
- Josh Barber, RPM at EPA Region 3
  - barber.joshua@epa.gov

Sampling for Defensible Environmental Decisions

- Presented by EnviroStat, Inc.
  - Feb. 26-March 1, 2018
  - http://www.envirostat.org/training.htm

Questions?