# GEOC5089 ME5089 Exploration Geochemistry SAMPLING CONT

**INSTRUCTOR**—Dr. Virginia T. McLemore

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**Occupational Safety and Health Administration** 

Mine Safety and Health Administration



Promoting productive workplaces through safety and health research /



# Always be Safe

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#### Why is workplace safety important?

- - The company is protecting itself, its employees, and its customers. It is important to follow guidelines and procedures to remain compliant with local and national occupational safety authorities.
- A safe workplace is a happy workplace comfortable and conducive environment for employees to effectively do their jobs.
- - The Human Resources department is in charge of making sure safe workplace standards are being met.
- - Other companies may not have an HR representative, and in that case, management and supervisors would ensure the workplace is kept safe.
- - It is the responsibility of everyone to create and maintain a safe and functional work environment.

- - Stay hydrated
- Drink enough water to remain alert and avoid dehydration.
- Wear all necessary safety gear
- Practice good posture when sitting or lifting
  Seated posture puts lots of strain on your body
  Exaggerated cures are bad
  Stretch frequently



- If your work involves sitting at a desk, discomfort doesn't have to be part of the job.
- You may be able to avoid some of the health problems associated with seated work, such as neck and back pain and sore wrists and shoulders, by using proper office ergonomics.
- Chair height, equipment spacing and desk posture all make a difference.



#### • - Take regular breaks

- Look away from your computer screen regularly to avoid eye strain.
- - Be aware of your surroundings
  - Look for spills or items on the floor that could be tripped over.
  - Note the appropriate safety equipment and gear for each task you are doing.
  - Choose mechanical aids such as a dolly to help move items.
  - Keep emergency exits clear and uncluttered so they are accessible in the event of an emergency.
  - Label hazardous materials with appropriate signage.
  - Know where the first aid kits are and which staff members are trained to administer first aid if an injury occurs.
  - Only use secure, steady ladders, and never use boxes or anything else as an improvised ladder.

- - Use tools, equipment, and machinery properly
  - Only operate machines you are trained or certified to use.
  - Ensure that machines are cleaned and maintained regularly.
  - Report any unsafe conditions
    - Fix any unsafe conditions or workplace hazards as soon as you notice them.
    - If it is dangerous for you to remove the risk, notify a supervisor right away.

Questions? Comments?

# • TH&NK YOU!



### **Francis Pitard**

".... The excellent preparation and high quality analytical work undertaken in the laboratory was wasted on a sample collected so poorly as to be beyond resurrection...."

# **Considerations:**

- What am I looking for?
- What size / shape / orientation is anticipated?
- What is my environment?
  - Climate
  - Topography
  - R-E-D
- What is the scale of my survey?
- What information do I already know e.g. geophysics?

# **Typical Scales**

- Regional
  - Large scale stream sediments (e.g. 1 per 10km<sup>2</sup>).
  - Large scale soils (e.g. 1 per 1km<sup>2</sup>).
  - Large scale Talus (e.g. 1 per 1km<sup>2</sup>)
- Follow up
  - Detailed streams (e.g. >> 1 per 1Km<sup>2</sup>).
  - Soils (e.g. 500m space lines by 200m samples).
  - Talus (e.g. 200m by 500m contour samples)
- Detailed
  - Soils (e.g. 200m lines by 50m samples).
- Pre Drill
  - Very detailed soils (e.g. 100m by 25m).
  - Rock chips, trenches.



#### **Regional Stream Sediments**

- Principles
  - Broad based sampling
  - Identification of anomalous basin area(s)
  - Long dispersion trails
  - Anomalies often low order and subtle
  - Multi-element Evaluation and ranking

#### **Regional Streams**

- Practice
  - Select long stream section
  - Sample +/-200m section,
  - multiple samples composited
  - >200m upstream of confluence
  - Careful to retain fines
  - Measure pH
- Remember geology
  - Review oversize fraction







 Original survey

Concentration



#### Follow-up sample plan





NURE data reanalyzed by the USGS



Soil survey in Gallinas Mountains, NM



Ce content in soils, Gallinas Mountains, NM

# **Rock Samples**

- Point sample type
- Highly biased
  - Subjective sample selection
- Requires consistent sampling practice
- Fresh un-weathered material

### **DATA VERTIFICATION**

"All analytical measurements are wrong: it's just a question of how large the errors are, and whether they are acceptable" (Thompson, 1989).

# **ERROR TERMINOLOGY**

- ACCURACY-nearness to truth
- BIAS-average departure from truth
- PRECISION-reproducibility of values
- RANDOM ERROR-symmetric with respect to mean
- STATISTICAL ANALYSIS-methods for dealing with error

# DEFINTIONS

- **Precision**—the degree of agreement among repeated measurements of the same characteristic. Precision is monitored by multiple analyses of many sample duplicates and internal standards.
- **Accuracy**—measures how close your results are to a *true* or expected value and can be determined by comparing your analysis of a standard or reference sample to its actual value. Analyzing certified standards as unknown samples and comparing with known certified values monitors accuracy.
- **Completeness**—the comparison between the amount of valid, or usable, data you originally planned to collect, versus how much you collected.
- **Comparability**—the extent to which data can be compared between sample locations or periods of time within a project, or between projects.

#### The difference between precision and accuracy



### **QUALITY CONTROL/QUALITY ASSURANCE**

- QC is referred to a program designed to detect and measure the error associated with a measurement process. QC is the program that ensures that the data are acceptable.
- QA is the program designed to verify the acceptability of the data using the data obtained from the QC program. QA provides the assurance that the data meets certain quality requirements with a specified level of confidence.

# **QUALITY CONTROL/QUALITY ASSURANCE**

- What is the purpose of your project?
- What do you need the analyses for and how accurate should they be?
- Where are the results going to be released or published?
- What is the mineralogy?
- What are appropriate certified standards (may need to develop lab standards)?
- What are the detection limits (both upper and lower)?
  - Analytical errors vary from element to element, for different ranges of concentration, and different methods
- Duplicate or more analyses of standards and unknowns verses duplicate runs of same sample

### **QUALITY CONTROL/QUALITY ASSURANCE**

- Analyze a separate set of standards rather than standards used for calibration
- Send samples and standards to other laboratories
- Establish written lab procedures
- Are blanks and field blanks used and analyzed?
- What are the custody procedures (collection date, preservation method, matrix, analytical procedures)?
- Does the chemical analyses make geological sense? Is it consistent with the mineralogy and type of mineral deposit?
- Sometimes there is more paper work than making sure the data is accurate
- What do you do if there are problems with QA/QC?

# **TYPES OF ERRORS**

- Systematic verses bias (constant, unintentional)
- Random errors (unpredicted but nonsystematic errors, imprecise practices)
- Gross or illegitimate errors (procedural mistakes)
- Deliberate errors



Reproducibility and bias in sampling (Adapted from Pitard 1993)

# **MEASUREMENT ERRORS**

- Wrong sample
- Wrong reading
- Transposition or transcription errors
- Wrong calibration
- Peak overlap
- Wrong method
- Contamination

- Losses
- Inattention to details
- Sampling problems
- Instrument instability
- Reagent control
- Variability of blank
- Operator skill
- Sample variability

## Example of QA/QC chemical analyses

- Duplicate analyses (1-2 duplicates for every 20 samples)
  - Laboratory will analyze duplicate samples
- Certified standards
- In-house standards (1-2 standards for every 20 samples)
- Sum of major oxides should equal 99-101%, unless other elements are in high concentrations
- Blanks (measure of contamination)
- Resubmit samples or use same standard in different batches
- Submit samples with known values
- Submit to different laboratories
- Collect duplicate samples in the field (but need to be certain they are same)
- Examples (NMBGMR OF-587, A7; OF-523, A8; OF-603)

#### Geochemistry must be consistent with the mineralogy and petrology

### What if there is a problem?

- Review all paperwork and procedures
  - Tour laboratory—contamination, cleaning, comp of grinders, crushers
  - Are standards appropriate for project?
  - Are types of analyses appropriate for project?
  - Analyses within 1 order of magnitude of detection limits can have large errors
  - Nugget effect
- Reanalyze some samples or the entire batch
- Submit to a different laboratory
- Contact and work with the original laboratory

Balance	True Value / g	Determined value / g	<b>percentage relative error =</b> <u>true value - determined value</u> true value
1.	7.90	7.90	$\frac{7.90 - 7.90}{7.90} \times 100 = 0.00 \%$
2.	7.90	5.78	$\frac{7.90 - 5.78}{7.90} \times 100 = 26.8 \%$
з.	7.90	7.62	$\frac{7.90 - 7.62}{7.90} \times 100 = 3.54 \%$
4.	7.90	7.58	$\frac{7.90 - 7.58}{7.90} \times 100 = 4.05 \%$

https://www.ausetute.com.au/precision.html

TABLE 8-8. Summary statistics for multiple analyses of internal standard STD-DEW0002 2005-2008 (sample from Orogrande rock pile, Otero County, New Mexico). Oxides in percent, trace elements in parts per million. WSU= Washington State University (7 analyses) and NMSU=New Mexico State University (9 analyses). (OF-523)

Oxide/trace element	Theoretical value	Standard deviation	Mean	Maximum	Minimum	Standard deviation	error (difference/ accepted)*100
Laboratory	NMSU						
SiO2	55.20	0.19	55.43	56.24	54.90	0.49	-0.42
TiO2	0.39	0.01	0.38	0.40	0.37	0.01	0.98
Al2O3	10.30	0.13	10.28	10.50	10.10	0.12	0.22
FeOT	6.93	0.24	6.88	7.08	6.43	0.26	0.67
MnO	0.07	0.00	0.07	0.08	0.06	0.01	3.92
MgO	1.14	0.02	1.16	1.24	1.12	0.05	-1.98
CaO	12.24	0.23	12.13	12.50	11.93	0.20	0.89
Na2O	2.64	0.04	2.49	2.69	1.24	0.47	5.55
K2O	2.34	0.07	2.23	2.40	1.35	0.34	4.67
P2O5	0.16	0.00	0.15	0.17	0.12	0.02	5.70
LOI	7.94	0.09	7.74	8.05	7.04	0.40	2.51
Sum of oxides	99.35		98.94				

TABLE 8-9. Summary statistics for multiple analyses of internal standard CAP-MLJ0001 2005-2008 (rock pile sample from Capulin rock pile, Questa mine). WSU= Washington State University (35 analyses) and NMSU=New Mexico State University (2 analyses of major oxides, 1 analyses of trace elements). FeOT is total iron calculated as FeO. (OF-523)

Oxide or trace element	Mean	Maximum	Minimum	Standard deviation	Error (max- min/mean)*100	Mean	Maximum	Minimum
Laboratory	WSU					NMSU		
SiO2	74.83	76.08	73.76	0.5885586	3.1003596	76.5	76.65	76.35
TiO2	0.21	0.212	0.2	0.0031134	5.7501481	0.22	0.22	0.22
Al2O3	11.81	11.97	11.69	0.0837692	2.3700758	11.885	11.91	11.86
FeOT	2.55	2.61	2.5	0.0347695	4.3127826	2.61	2.61	2.61
MnO	0.04	0.042	0.04	0.0005586	4.9275581	0.035	0.04	0.03
MgO	0.43	0.453	0.42	0.0110022	7.5882324	0.435	0.44	0.43
CaO	0.13	0.2	0.09	0.0315038	86.566838	0.125	0.13	0.12
Na2O	0.95	0.964	0.92	0.012499	4.6504432	1.015	1.04	0.99
K2O	4.81	4.88	4.74	0.04243	2.9132123	4.835	4.84	4.83
P2O5	0.05	0.06	0.049	0.0034528	20.719424	0.01	0.01	0.01
LOI	3	2.98	2.66	0.0796281	11.883639	2.665	2.74	2.59
Sum of oxides	98.81					100.335	100.63	100.04
1.11	27	10	20	2 00/22204	10 51//00	31		

TABLE 7-3. Duplicate analyses of selected samples. Major oxides, S, C, LOI, Sum are in percent, trace elements are in parts per million (ppm). Sum is the total major oxides. FeOT and Fe2O3T are total iron calculated as FeO or Fe2O3. LOI is loss on ignition. (OF-587)

SAMPLE	SL 60	SL 60b	SL24	SL24a
Al <sub>2</sub> O <sub>3</sub>	5.46	6.2	4.15	4.1
BaO	0.06	0.06	0.03	0.02
CaO	0.15	0.17	0.11	0.11
Fe <sub>2</sub> O <sub>3</sub> T	17.62	14.14	30.5	31.1
K <sub>2</sub> O	0.79	0.69	0.73	0.75
MgO	0.21	0.24	0.1	0.1
MnO	0.37	0.28	0.18	0.19
Na <sub>2</sub> O	0.23	0.21	0.3	0.29
P <sub>2</sub> O <sub>5</sub>	0.14	0.18	0.1	0.1
SiO <sub>2</sub>	63.77	64.43	60.88	59.71
TiO <sub>2</sub>	5.75	8.01	0.55	0.45
LOI	4.32	4.18	2.43	2.81
a	00 70	00.01	100 0	00.03

## Additional sampling procedures

# **Chemical analyses**

Each batch of samples submitted for chemical analyses should have

- 1-2 duplicates (many times the lab will select samples and run the duplicate)
  - Some projects require triplicates
- A standard sample (internal or commercial)
- Optional—an additional sample that you are certain of the analyses
- Water samples in particular require blanks, samples of the water used to clean equipment, bottles, etc.
  - Water analyses have more QA/QC requirements

## Drill core

- Split the core in half (or quarter)
- Always leave part of the core in the core box—DO NOT TAKE ALL OF THE CORE
- Leave a note with your name and reason for sampling the core

# Shipping

### Procedures

- Vary with laboratory
  - USGS 45 in a batch
- Prevent spread of invasive species—wash off plants and soil
- Make sure each sample bag is firmly sealed and packed into the bag
- Fill all voids in the box with packing material to prevent spillage or leakage and cross contamination
- Do not ship wet sediments or soils
- Print a copy of the submittal form and put it into the box with the samples
  - Confirm that the submittal form marches the physical samples
- Email the form to the lab
- Fully encircle each dimension of your three-dimensional box with strips of strapping tape
- Do not send real heavy boxes





#### Mark will discuss databases Sept 19

# Evan will discuss IoGas and data presentation Oct 17

# Zuni Mountains study progress

Туре	Number		
sediment	57		
ALS	12		
pan con	2		
rocks	62		
total	133		

- Sediments to be sieved
- Sediments to be dried
- Sediments to be shipped
- Rock samples to be shipped
- One set of ALS samples shipped
- Missing sample data?? Harriett?







- Areas 1 and 2
  - Finished sampling—private land
- Areas 3-5
  - Examine maps and decide where else to sample
  - Start looking at drainages of the Proterozoic rocks
- Next sampling trip Sept 22-24
- Mostly collecting sediments
  - Some mineralized rock samples from prospects