Indicator Mineral Chemistry
Quality Control Discussion

Mary Doherty
Geochemist, Director USA
ALS Chemex
Quality Control Discussion

- Value of Quality
- Define Sources of Error
- Measurement of Error
- Systems to minimize and control error
Value of Quality
Quality Control Discussion

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Define Sources of Error

Field Variability

Field Collection
Sampling Error

Sample Preparation

Mineral ID

Mineral Analysis
Stream Sediment Sample

Weight: 10 Kg

Size Fraction: -2 mm
Gy’s Safety Curve

Assume:
- Homogeneous sample
- Initial sample size
Sample Heterogeneity

Sample A: 3,0,1,1,1,0,0,0,1
Answer: <1

Sample B: 9,4,3,2,4,3,3,4,5
Answer: 5

(Pitard, 1993)
Define Sources of Error

- Field Variability
- Field Collection
  - Sampling Error
- Sample Processing
- Mineral ID
- Analytical
Sample Processing

- Sieve
- Gravity Density Separation
- Liquid Density Separation
- Magnetic Separation
- Mineral Picking
Define Sources of Error Processing

- Sample processing is complicated by mineral chemistry variations, magnetic susceptibility and density.
- Loss sample
- Sample switches
- Grain losses
- Sample contamination/carry-over
- Splitting error
Split Error

“Taking a representative split from a large sample is not as simple as many people believe.”

(Rocklabs)
Split Error

- Split all sample
- Even split
- No density separation
- No loss of fines or size segregation
- Error Comparison
  - Cone and Quarter 13.6%
  - Scoop Sample 10.3%
  - Riffle Split 2%
  - Rotary 0.25%
  
  (Allen and Kahn, 1970)
- Riffle may be as good if operator careful, less likely for larger samples requiring multiple splits.
- Rotary not too fast to segregate fines, more cones preferable.
Define Sources of Error

Field Variability

Field Collection
Sampling Error

Sample Processing

Mineral ID

Analytical

EMP
LA-ICPMS
LA MC ICPMS
- Spot sizes range from 10 to 75 microns
- Output energy range
- Integration time
- Carrier gas flow rate
- Fractionation variation with depth
- Fractionation variation with position on the mount surface

(www.geo.arizona.edu/alc/Analytical%20Methods.htm)
Ablation Error
Ablation Error Pyrite

- Melting and splatter
- Trulli

(Woodhead et al, 2009) Pit ~100 µm diameter
ICP-MS Error

- Batch Baseline Shifts
- Instrumental Drift
ICP-MS Analytical Errors

➢ Standard Data
Time /Batch Variation over 2 years.

(Woodhead et al, 2009)
Laser Ablation/ICPMS Counts over time

- Decrease in Counts with time
- Delay to purge the previous sample
- Interferences (Hg in argon)

(Bernhard Bühn; Márcio M. Pimentel et al, 2009)
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Measure and Monitor Quality Control

Field duplicates

- Two samples in field
- Are they useful for indicator mineral sampling?

Probability of field recovery
5%

Probability of lab recovery
92.99%

- Suggest 5%
Blanks:

- Ceramic grade coarse sand
- Detects process sample carry-over or sample switches
- First sample of batch to prevent carry-over from previous work.
Measure and Monitor Quality Control

Indicator Mineral Preparation and Identification:

- **Multiple site replicates, Site “standards”**
  Set of samples taken from a known valuable field site
- **Spike samples**
  Known number of distinct grains
  - **Unique color diamonds**
Measure and Monitor Quality Control

Indicator Mineral Preparation and Identification:

- **Spike samples**
  - Known number of distinct grains
- **Laser Etching**

(photos from Whiteford)
Measure and Monitor Quality Control

ICP-MS Checks

Multiple Grain Re-analyses in batch and over time
Ablation Standards

NIST Glass
USGS Standards

- Matrix Matched standards
- Sulphide vs. Glass
- Calibration

(USGS Standard Photo)
### Ablation Standards

#### Arizona LaserChron Center

<table>
<thead>
<tr>
<th>Sample</th>
<th>Age</th>
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<tbody>
<tr>
<td>ECSTALL</td>
<td>91 Ma</td>
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<tr>
<td>OU49127</td>
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<tr>
<td>TEMORA</td>
<td>417 Ma</td>
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<tr>
<td>R33</td>
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<td>91500</td>
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<td>DULUTH</td>
<td>1099 Ma</td>
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<tr>
<td>ORACLE</td>
<td>1434 Ma</td>
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</tbody>
</table>

Mean & uncertainty of 10 analyses (2σ SEM)
- ■ 50 micron spot (faradays)
- □ 15 micron spot (channeltrons)

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(www.geo.arizona.edu/alc/Analytical%20Methods.htm)
Project Precision and Accuracy

Assess Accuracy and Precision against project requirements

(Woodhead et al, 2009)
Measure and Monitor Quality Control

- Grain Analysis, LA-ICPMS
  - Known grains
  - Randomize
  - Duplicate grains
  - Batch repeats
  - Duplicate samples to second lab
  - Quantify Precision and Accuracy against project requirements
Quality Control Recommendations

- Insert 10% quality control samples in both processing and grain analytical batches.
  - Field Duplicates – Site Variability: 1 in 20-30
  - Blanks – carry-over contamination: 1 in 20
  - Analytical Control Samples – Accuracy: 1 in 20
  - Lab Replicates – Precision: 1 in 20
  - Standards Matrix Match
- Confirm Data with Follow-up Sampling (5%)
- Check with a second lab (10%)
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Value of Quality

Hit the mark, with a well designed system which will:
recognize error,
measure the error,
minimize error.