ALS Laboratory Group

ANALYTICAL CHEMISTRY & TESTING SERVICES

Indicator Mineral Chemistry Quality Control Discussion

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ALS Chemex

Quality Control Discussion

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

ALS Laboratory Group

ANALYTICAL CHEMISTRY & TESTING SERVICES

Value of Quality

Quality Control Discussion

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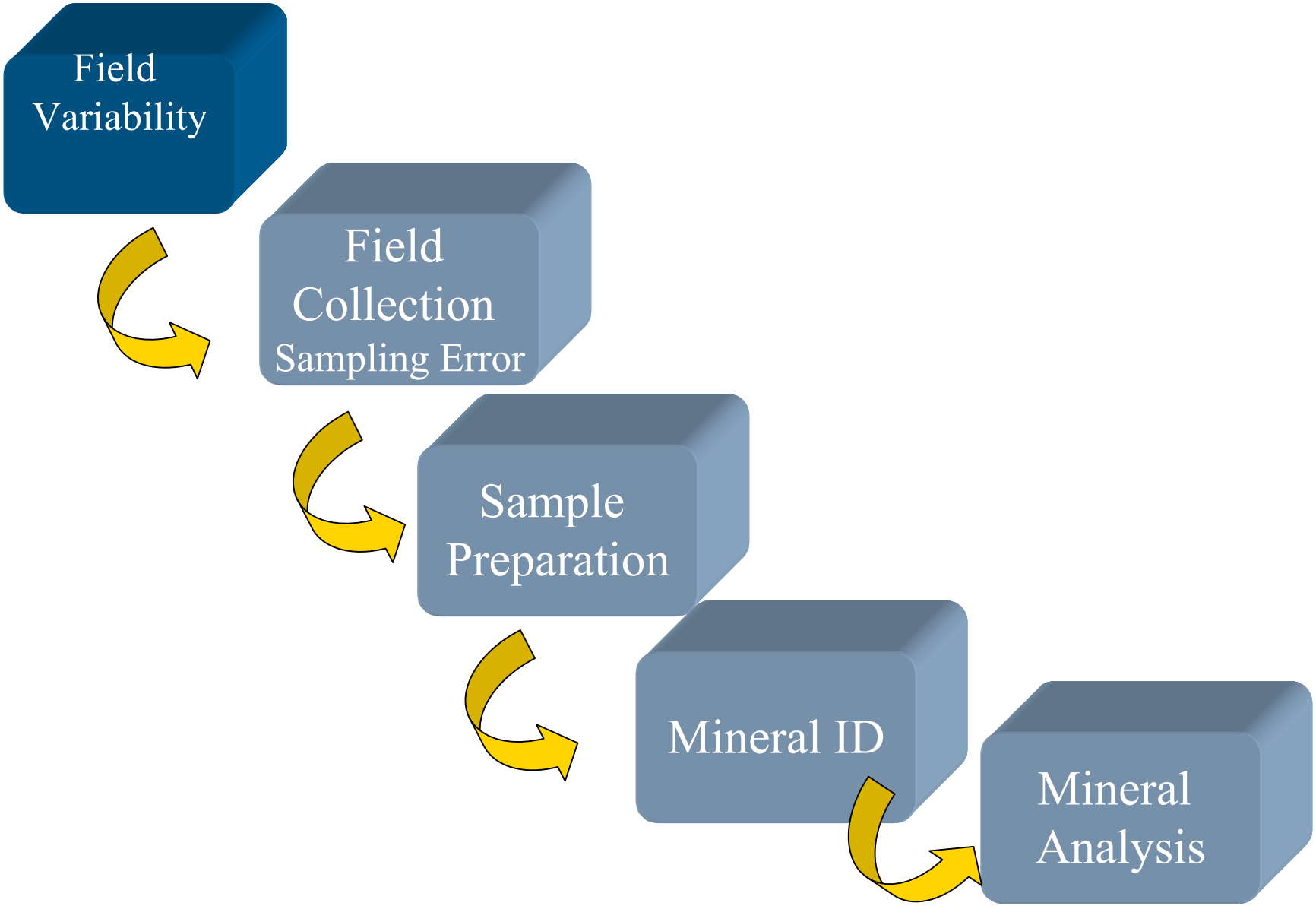
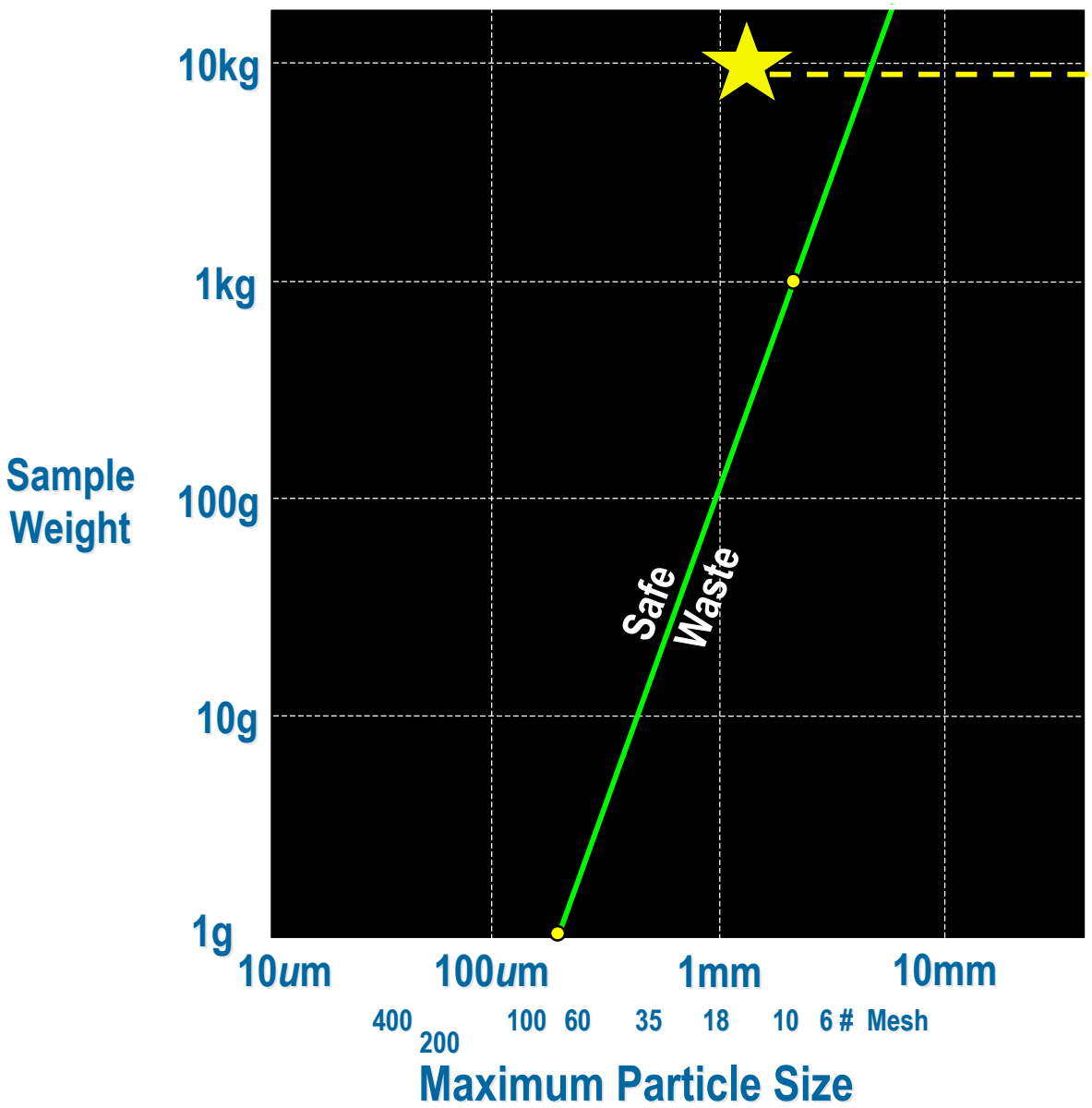
Define Sources of Error



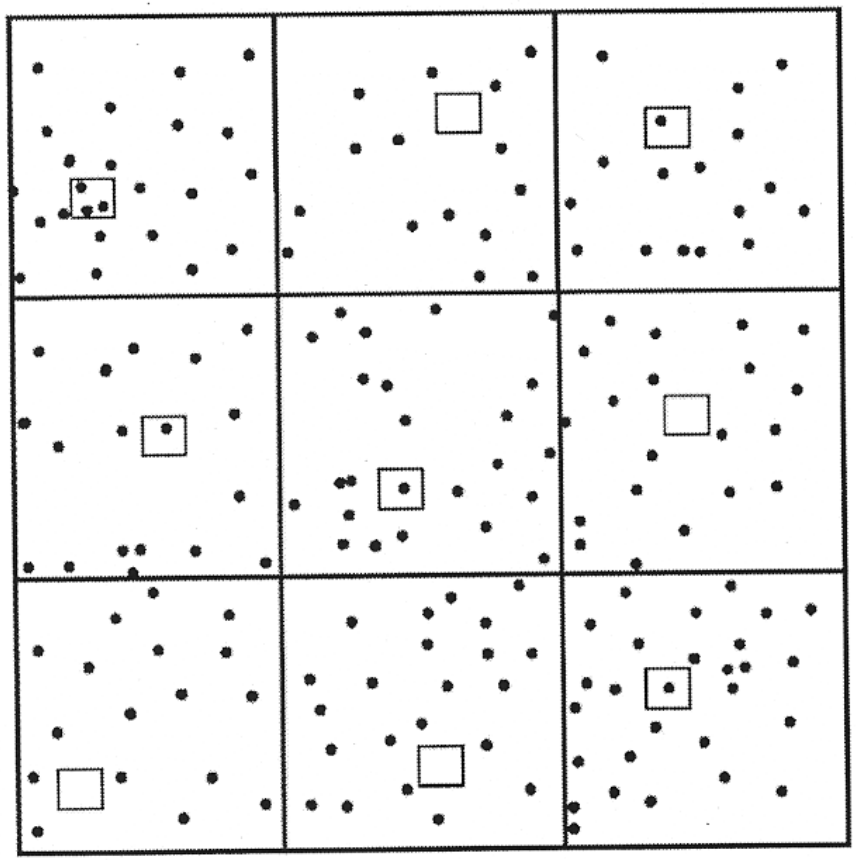
Gy's Safety Curve

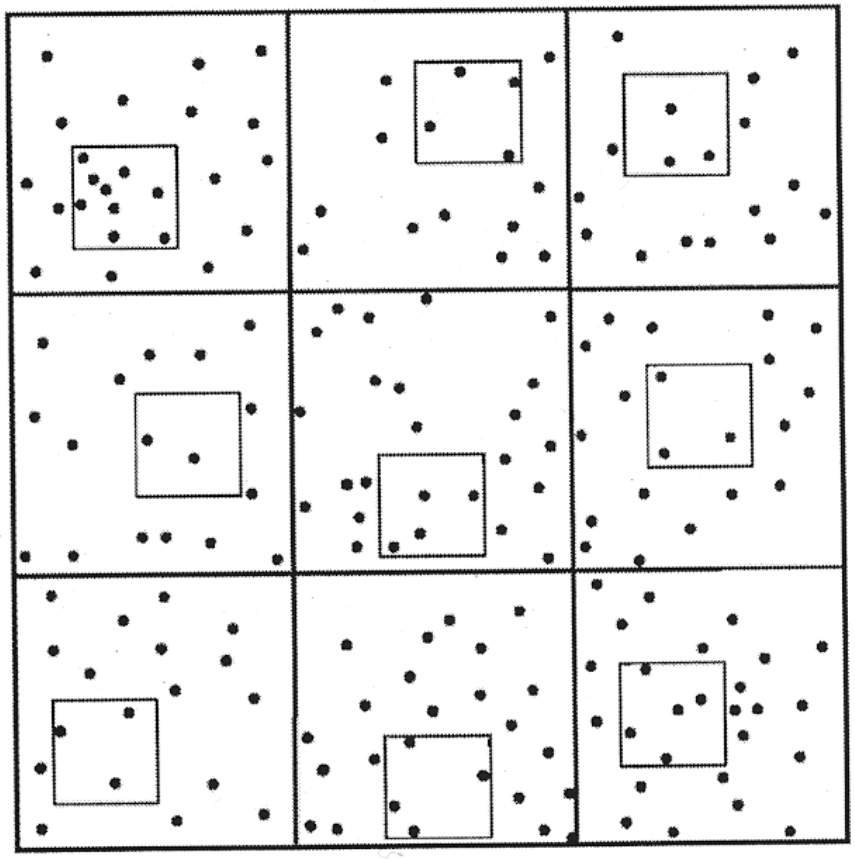


Assume:

- **Homogeneous sample**
- **Initial sample size**

Sample Heterogeneity





Sample A: 3,0,1,1,1,0,0,0,1

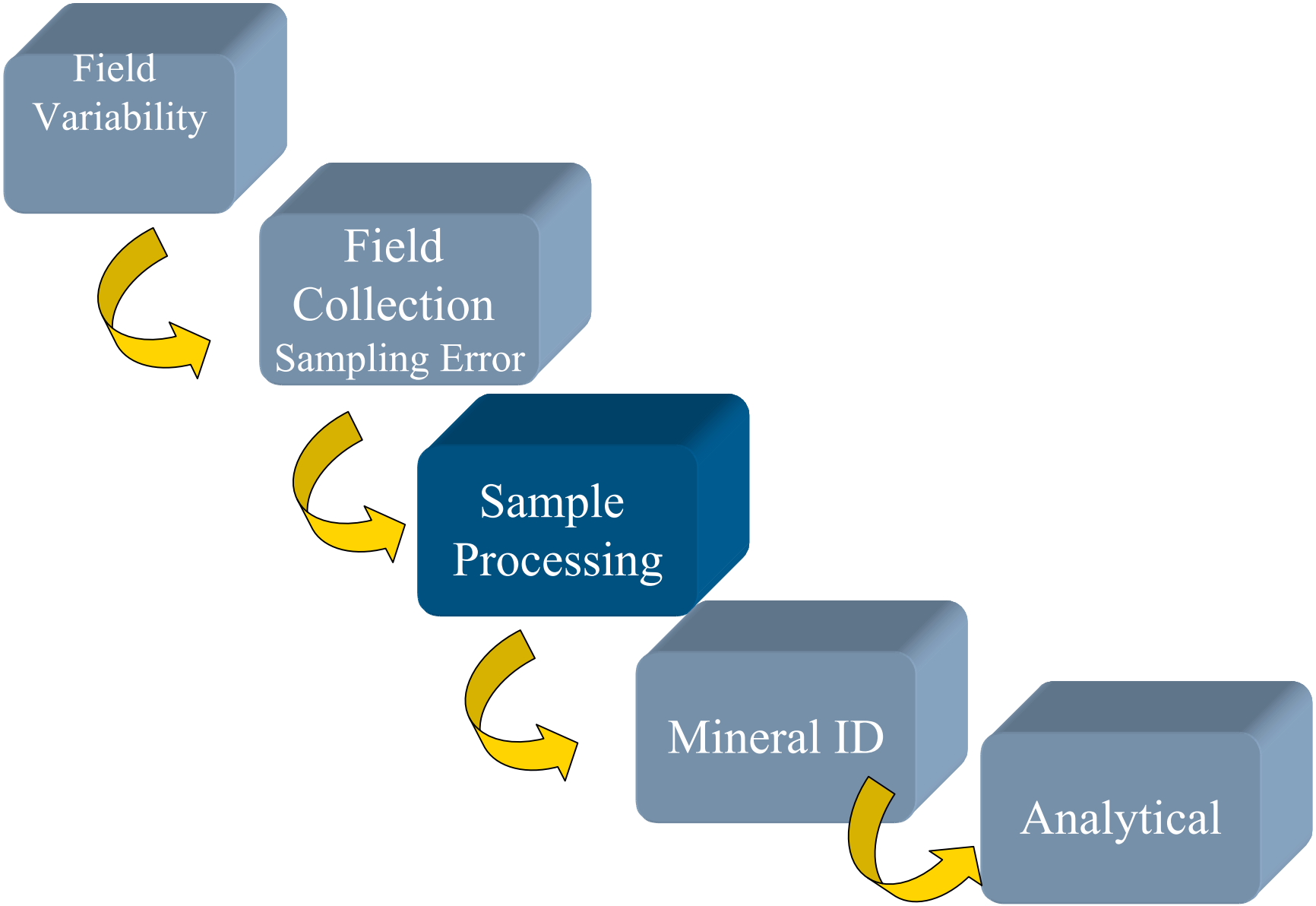
Answer: <1

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

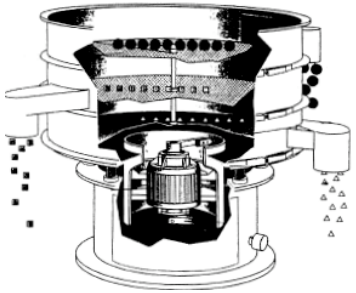
Answer : 5

(Pitard, 1993)

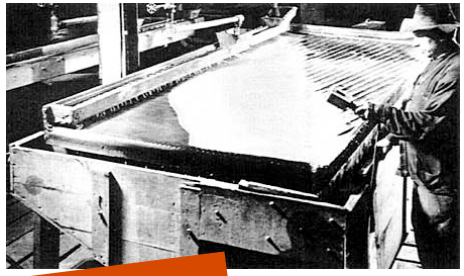
Define Sources of Error



Sample Processing



Sieve



Gravity Density Separation

Liquid Density Separation

Magnetic Separation

Mineral Picking

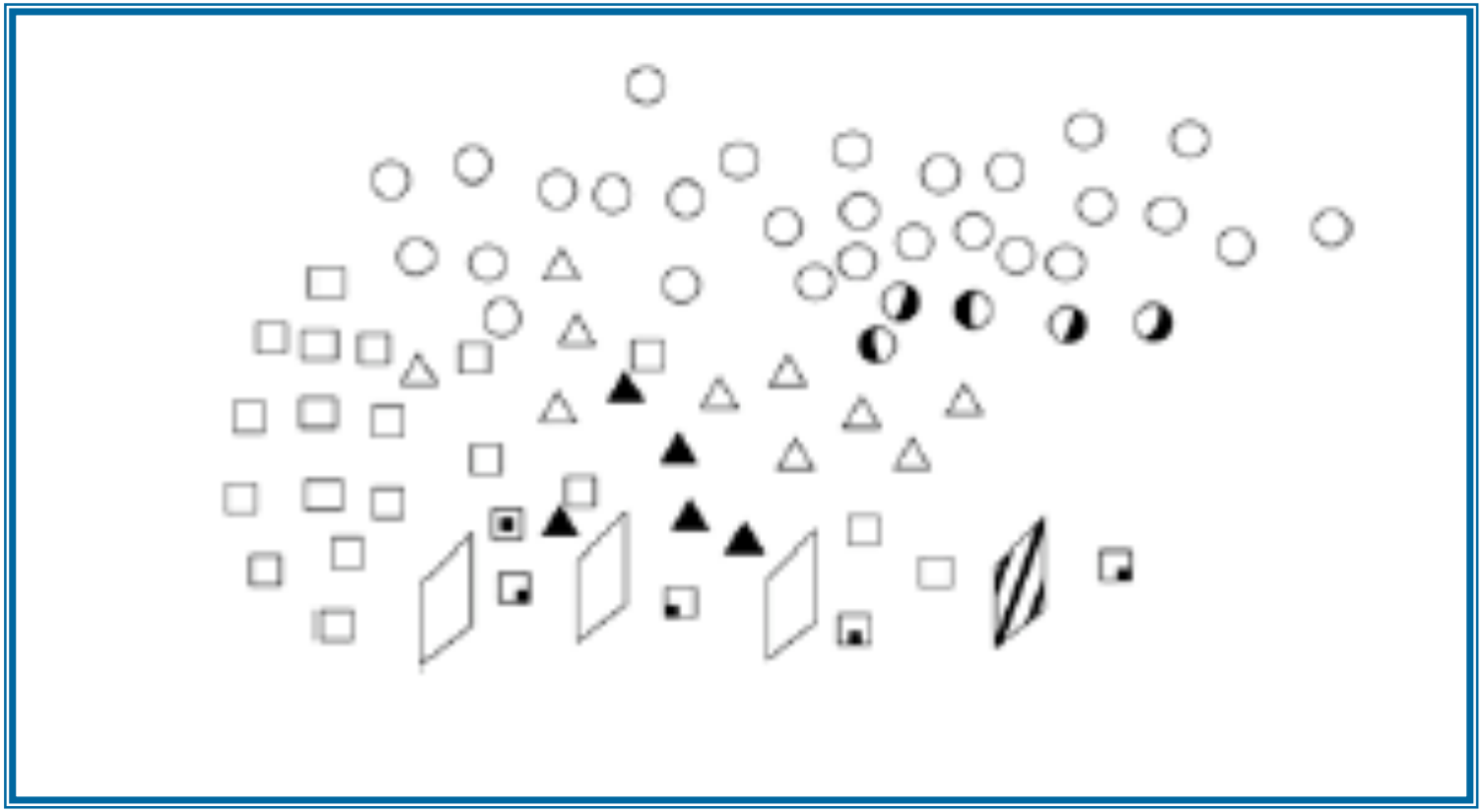
Define Sources of Error Processing

- 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."

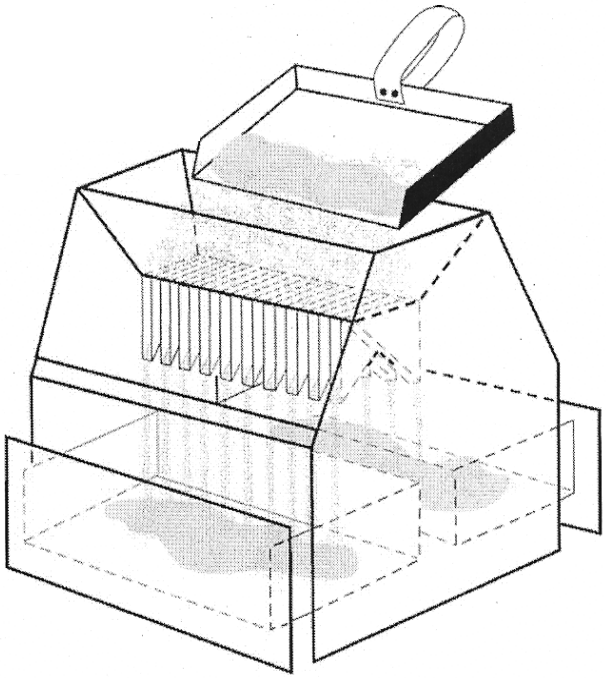
(Rocklabs)



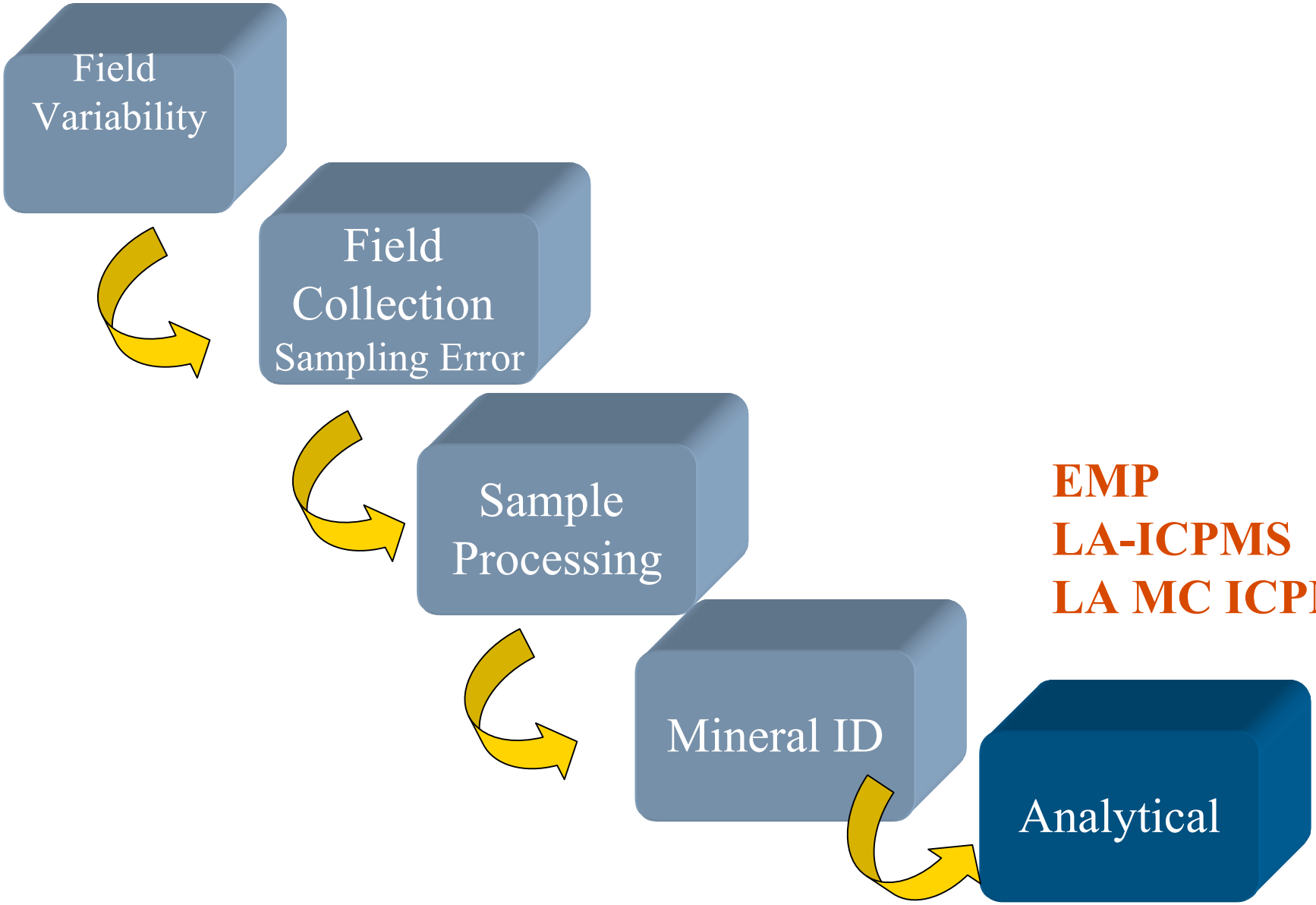
Split Error

- 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%

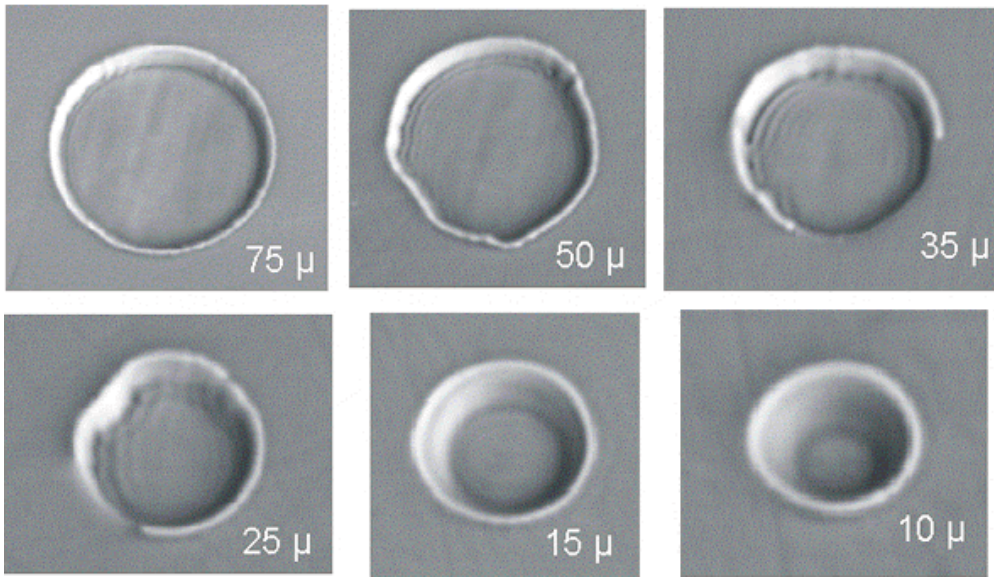
    (Allen and Kahn, 1970)
- Riffle may be as good if operator careful, less likely for larger samples requiring multiple splits.
- Rotary not too fast to segregate fines, more cones preferable.



Define Sources of Error



EMP
LA-ICPMS
LA MC ICPMS

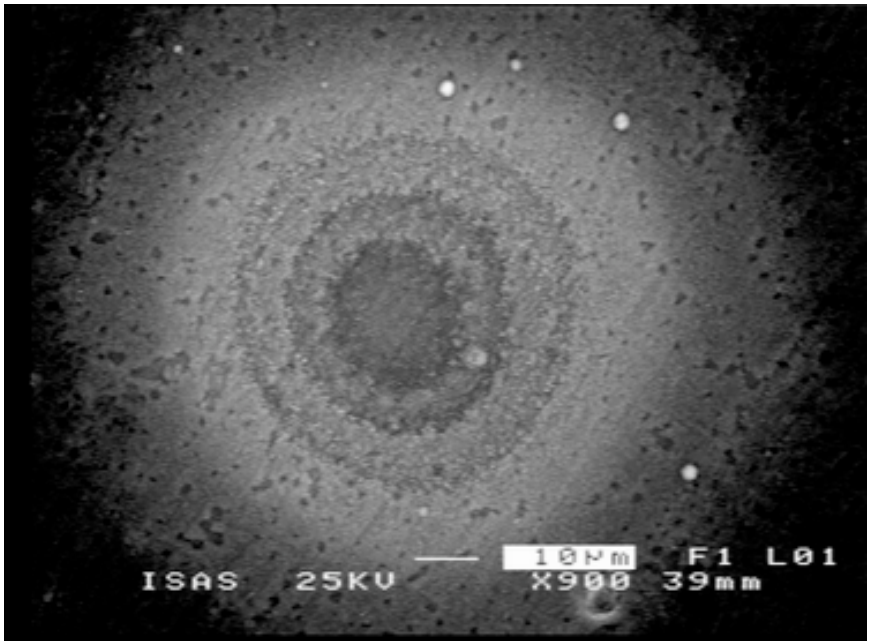


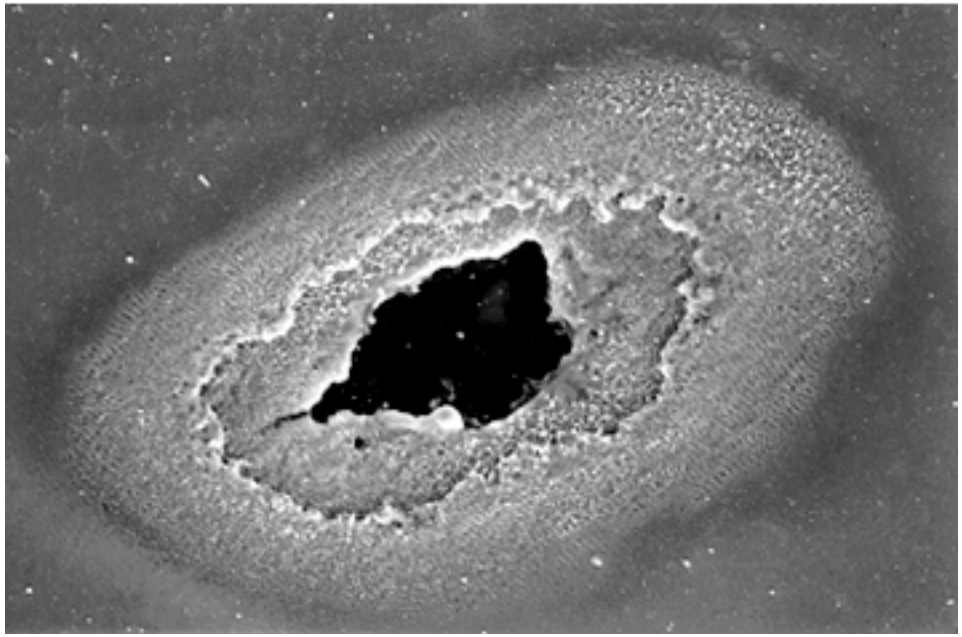
Ablation Error

- 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)

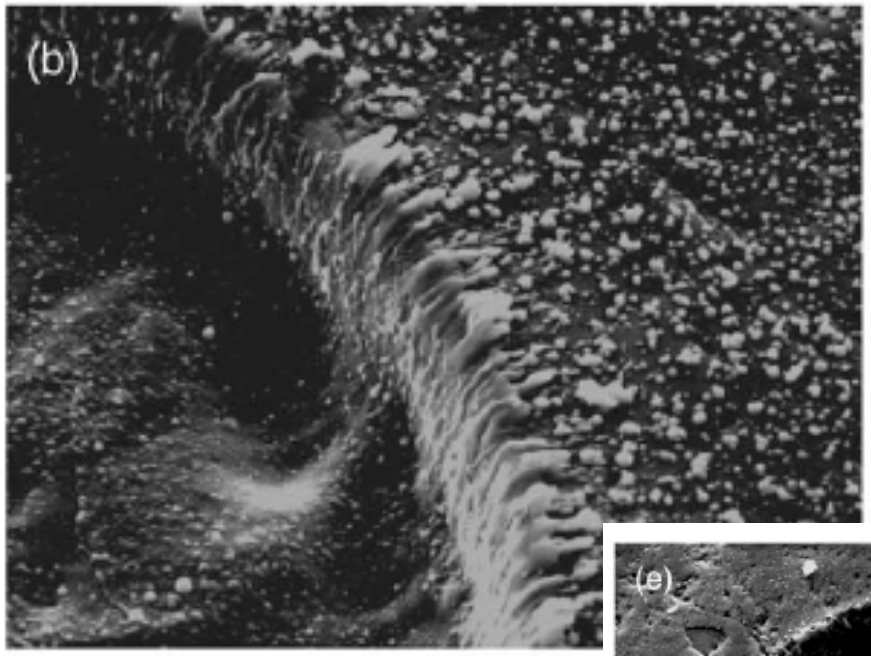
Ablation Error



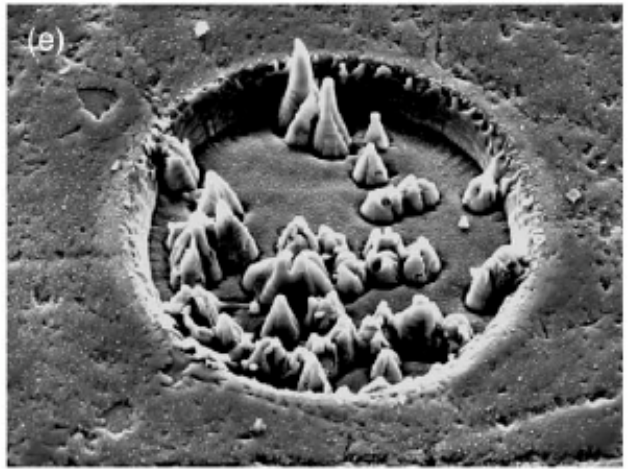


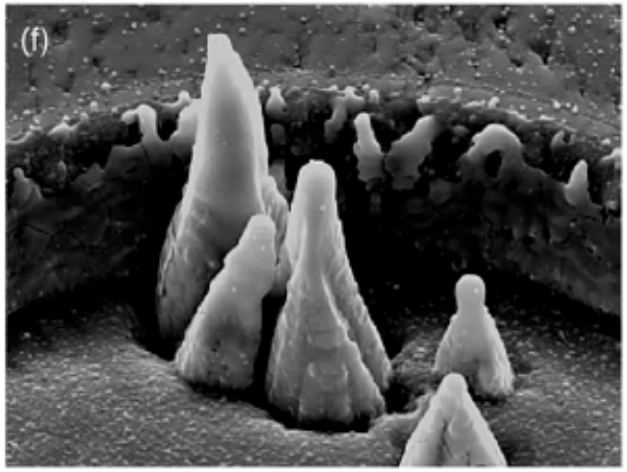
http://www.rsc.org/ej/JA/2001/b100016k/b100016k-f8.gif

Ablation Error Pyrite



- Melting and splatter
- Trulli

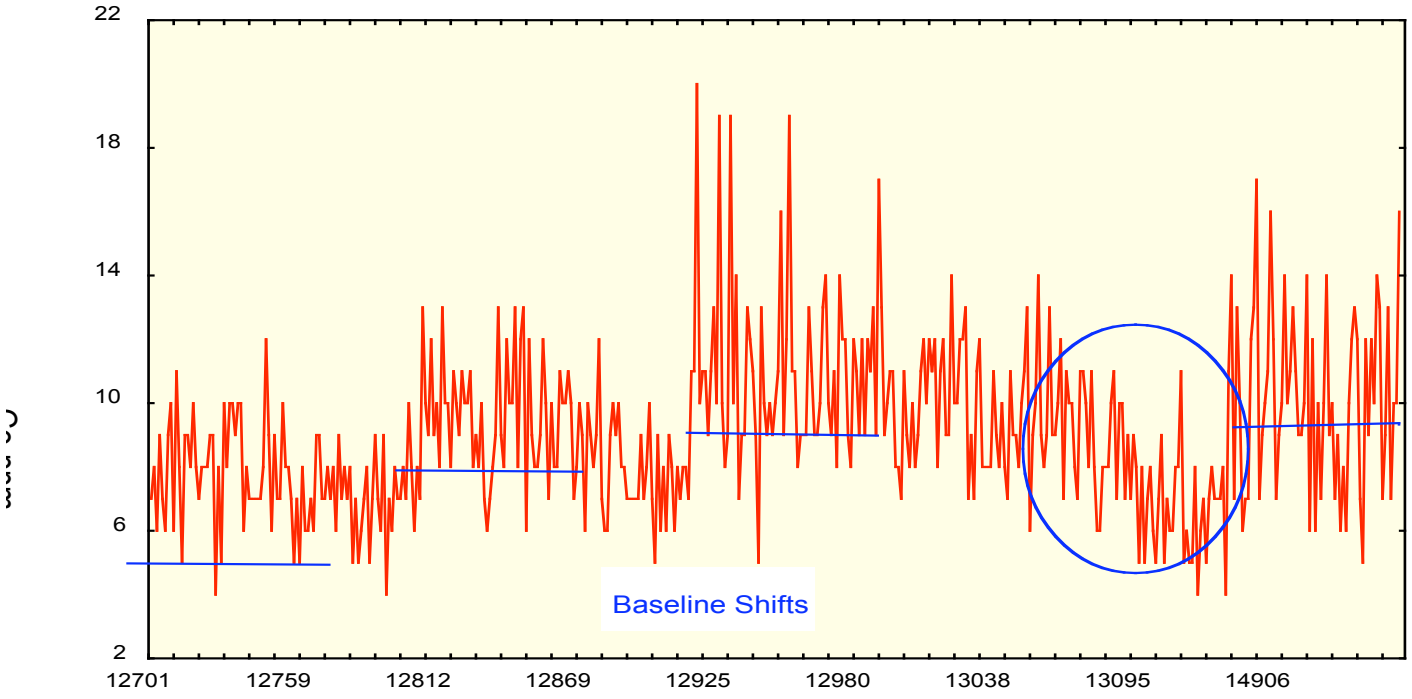




(Woodhead et al, 2009)

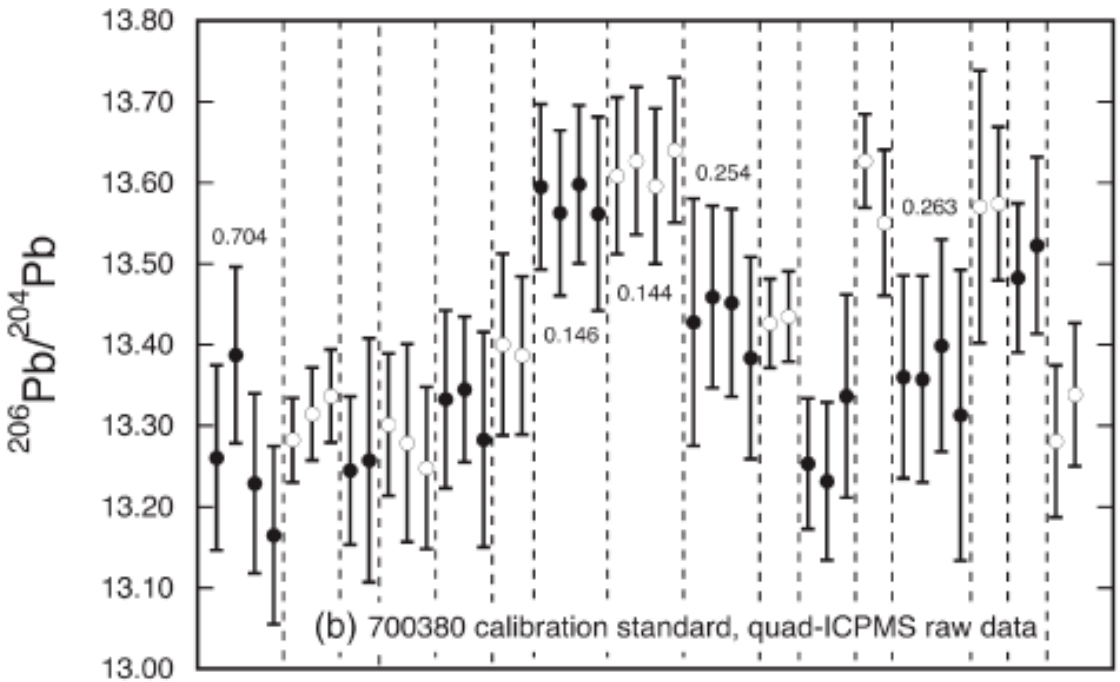
Pit ~100 µm diameter

ICP-MS Error



- **Batch Baseline Shifts**
- **Instrumental Drift**

ICP-MS Analytical Errors

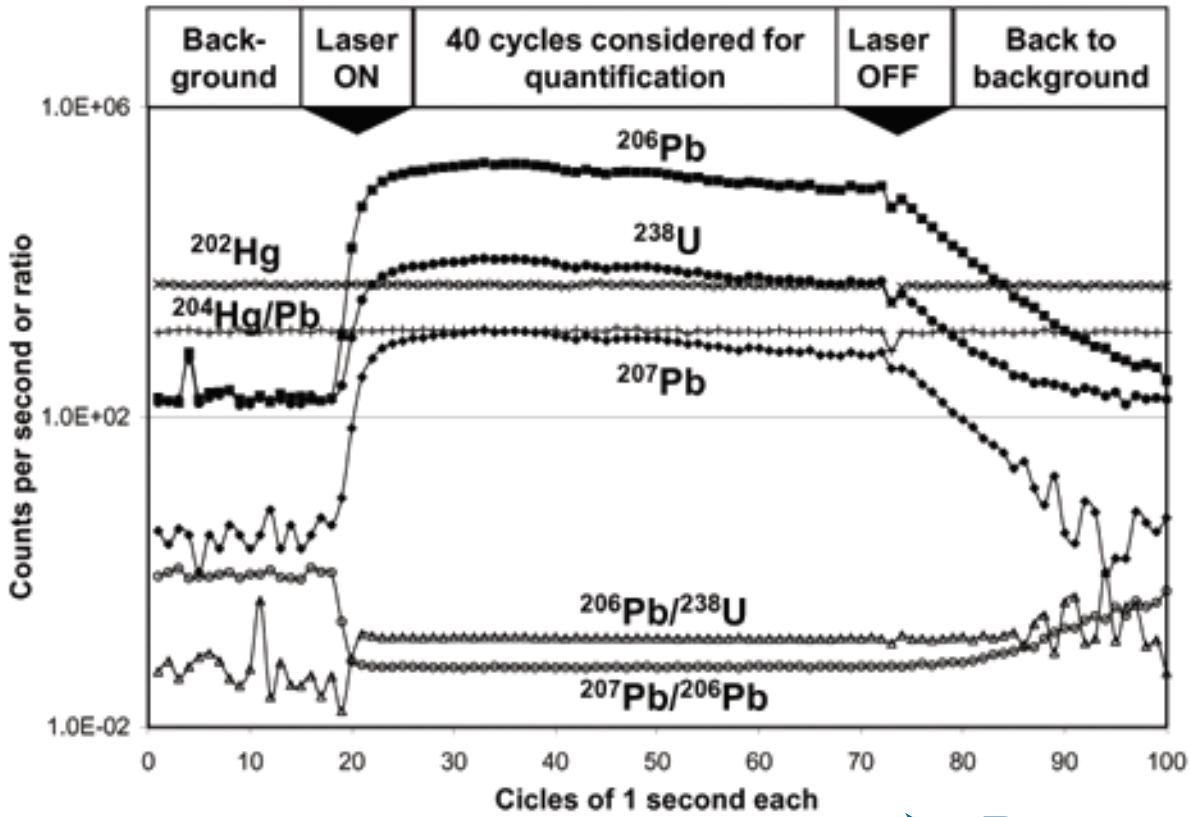


- **Standard Data**

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)

Quality Control Discussion

- Value of Quality
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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%

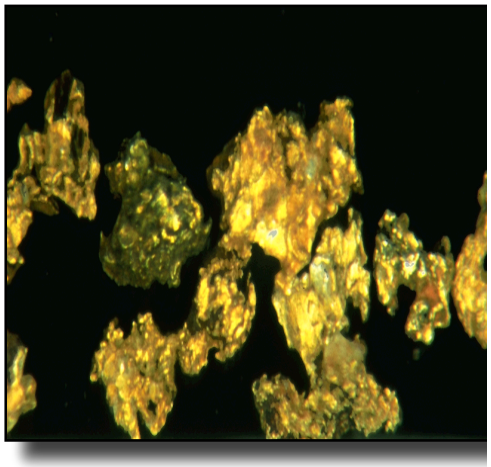
Measure and Monitor Quality Control

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:



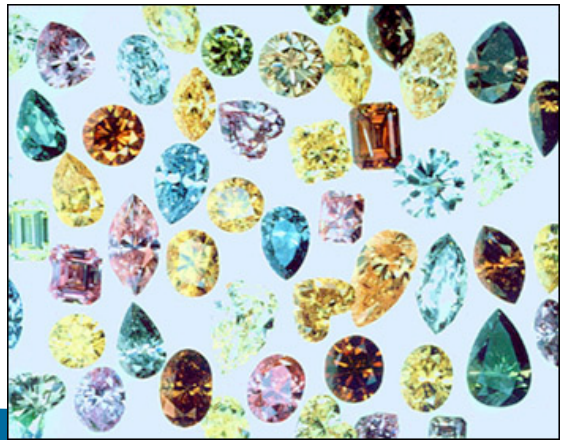
- **Multiple site replicates, Site "standards"**

  Set of samples taken from a known valuable field site

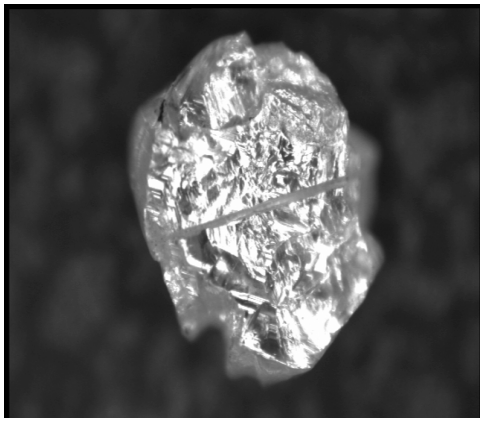
- **Spike samples**

  Known number of distinct grains

  - Unique color diamonds



Measure and Monitor Quality Control

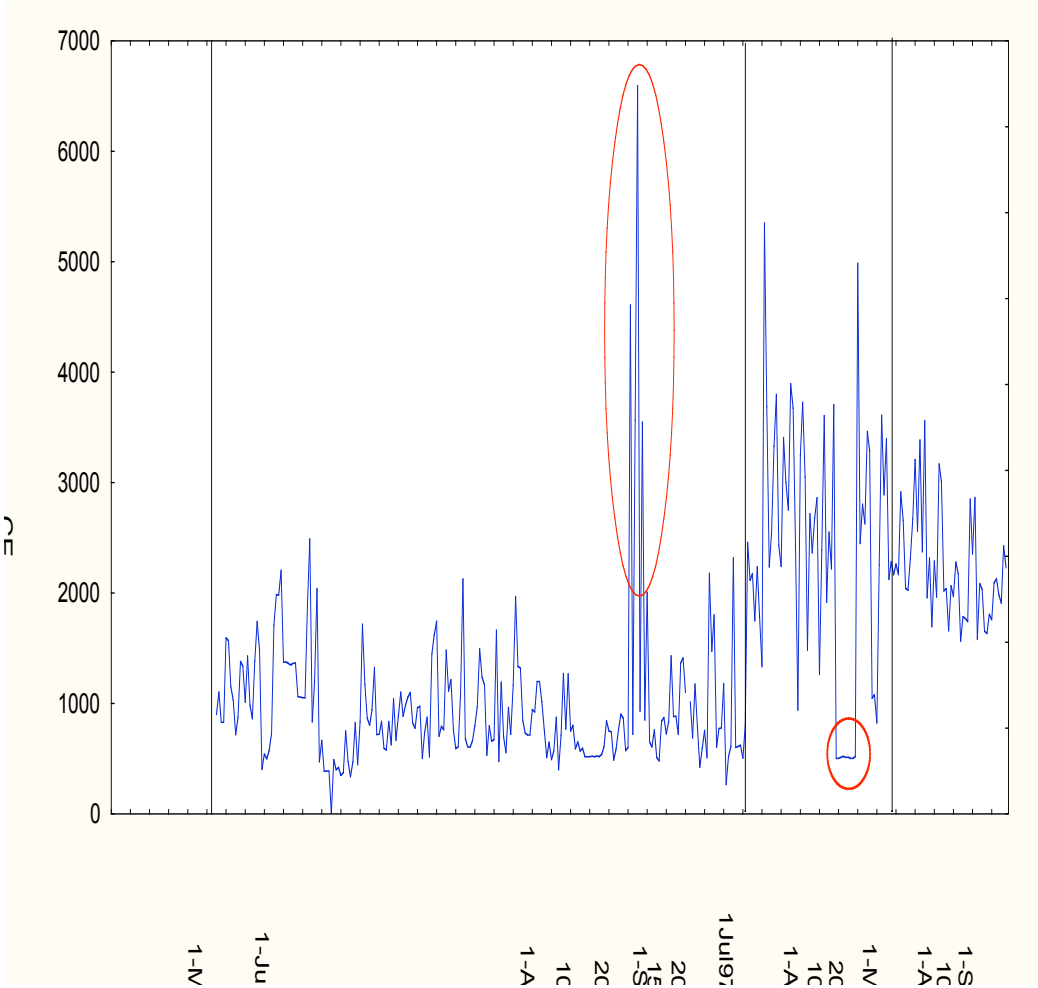


(photos from Whiteford)

Indicator Mineral Preparation and Identification:

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

Measure and Monitor Quality Control



ICP-MS Checks

Multiple Grain Re-analyses in batch and over time

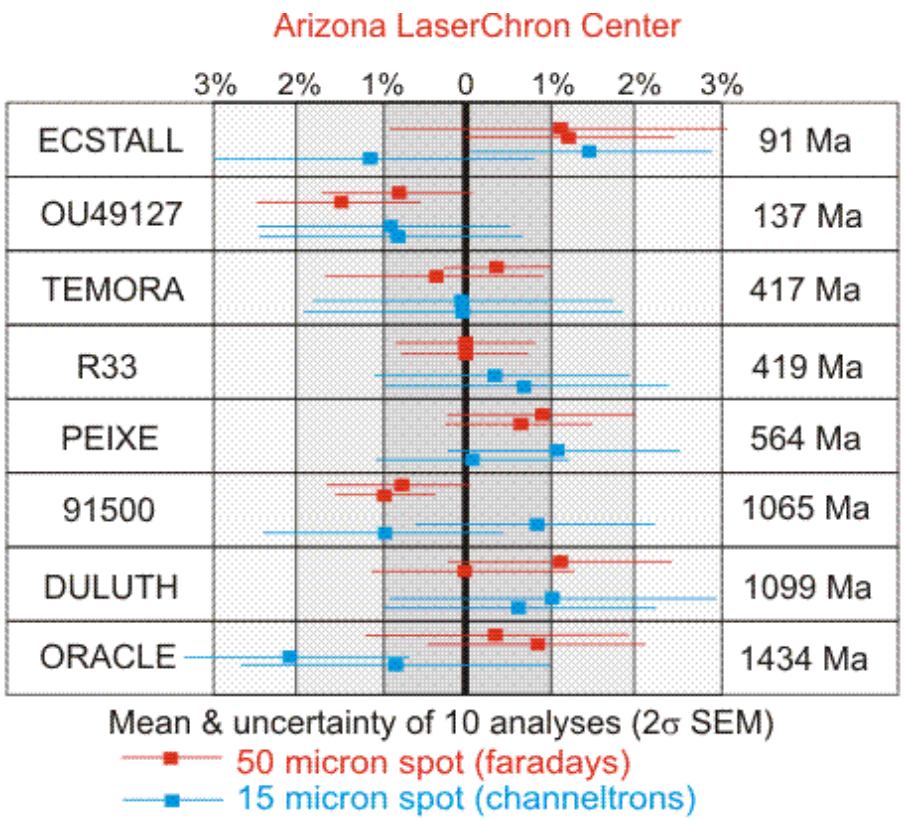
Ablation Standards

NIST Glass
USGS Standards

- **Matrix Matched standards**
- **Sulphide vs. Glass**
- **Calibration**

(USGS Standard Photo)

Ablation Standards



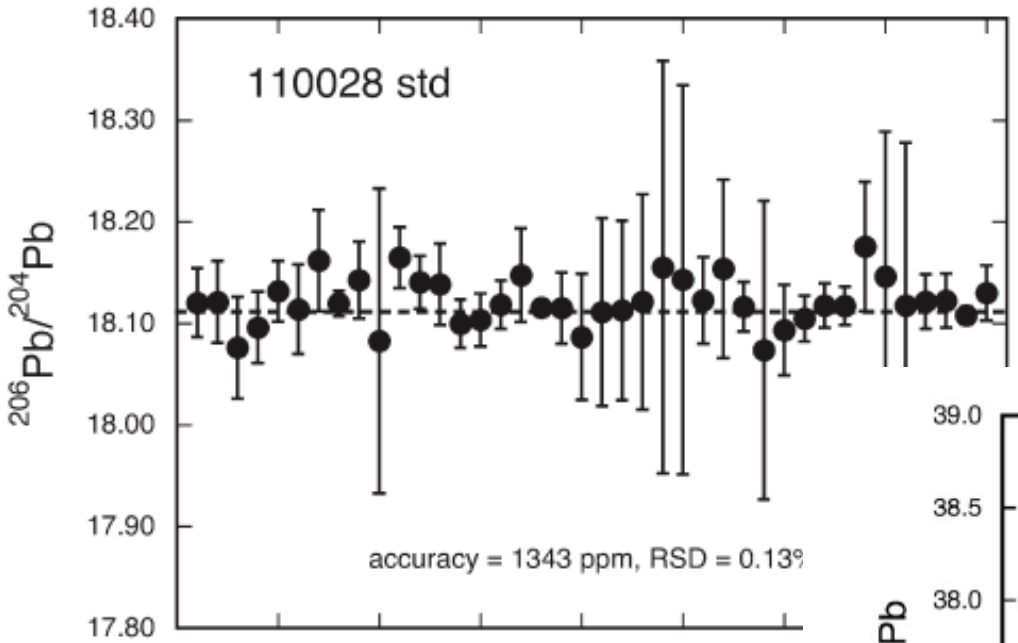
Zircon

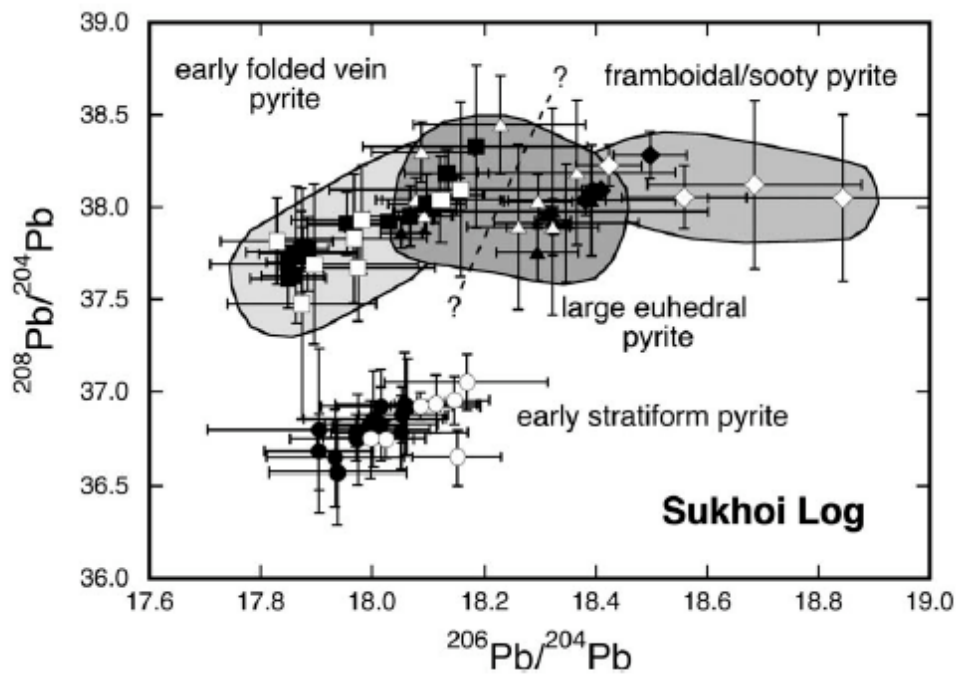
10 repeat analyses

- **Precision**
- **Accuracy**

(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%)

Quality Control Discussion

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Value of Quality

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

ALS Laboratory Group

ANALYTICAL CHEMISTRY & TESTING SERVICES

Certified System™

ISO 9001