

---

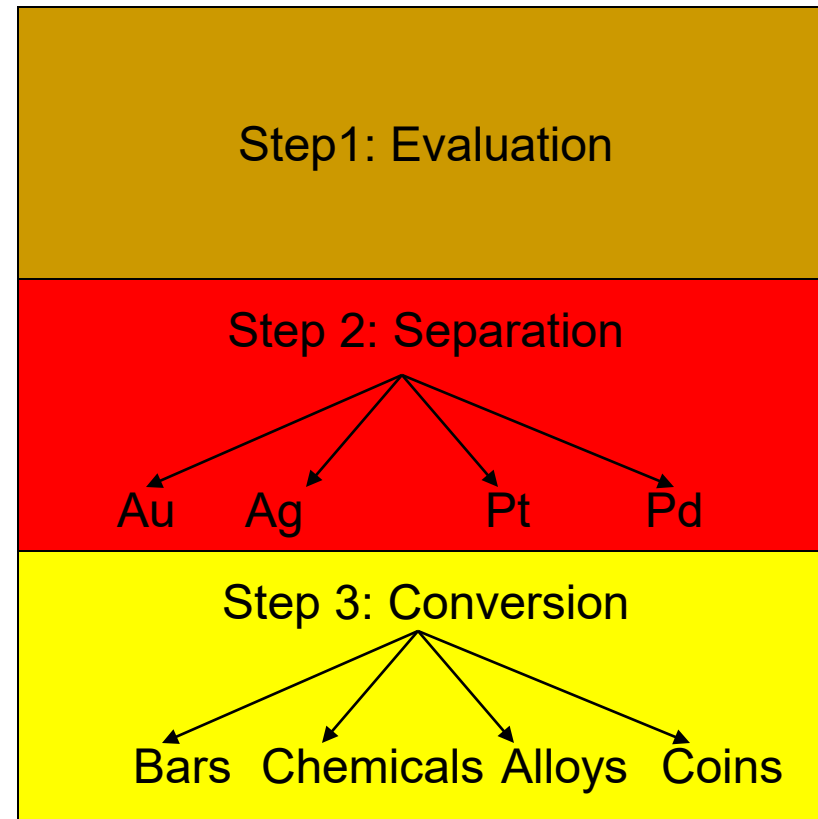
# **Sampling Precious Metals in the Liquid and Solid States - A Review of Practices and Suggestions for Improvement**

---

**Michael B. Mooiman**

Franklin Pierce University & Argo Advisors International

# Refining – 3 Key Steps



# Precious Metals Evaluation

**Sampling** + Weighing + Assaying



**Precious Metals Content**

- The challenge! Only 0.5 gram analyzed so....
- 0.5 g sample had better be representative of the lot

# What Standards Apply?

- AS 3515.2:2019 Gold and Gold Bearing Alloys, Part 2: Determination of gold content (30% to 99.5%) – Gravimetric (fire assay) method
- ISO 11596:2021 Jewellery and precious metals — Sampling of precious metals and precious metal alloys
- ASTM E1335-08(2017) Standard Test Methods for Determination of Gold in Bullion by Fire Assay Cupellation Analysis
- ASTM B562-95 (2021) Standard Specification for Refined Gold
- ASTM B413-97a (2021) Standard Specification for Refined Silver

# In Summary:

- Liquid sampling better than solid sampling
  - Vacuum pins
  - Dips
  - Graining
- Solid sampling
  - Drilling
  - Sawing
- Patterns - partial or all the way through

---

# Theory of Sampling Teachings - Gy's 7 sampling errors

1. Fundamental error
2. Grouping and segregation error
3. Long range non-periodic heterogeneity error
4. Long range periodic heterogeneity error
5. Delimitation error
6. Extraction error
7. Preparation error

---

# Theory of Sampling Teachings

Gy's 7 sampling errors

- Material variation
- Tools and techniques
- Periodic/process variations

---

# Reduce sampling errors

Due to material variation

- Mixing
- Particle size reduction
- Increase mass of sample
- Take many random increments and combine



# Reduce sampling errors

Due to tools and techniques

- Reduce sampling dimension when possible
- Take correct sample - every part of lot has equal chance of being sampled
- Use correct sampling tool and use it correctly
- Preserve integrity of sample

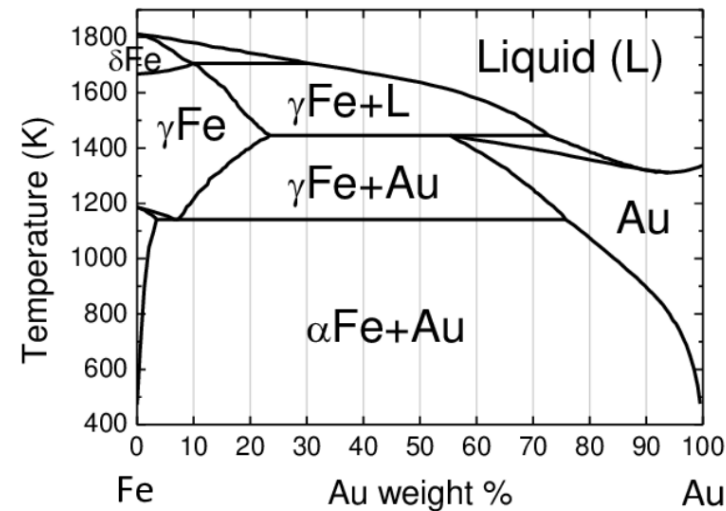
# Review of Sampling Approaches - Liquid

- High temperature to melt and homogenize all components
- Flux - borax
- Well mixed
- Sampling techniques
  - Vacuum tube
  - Dips
  - Graining

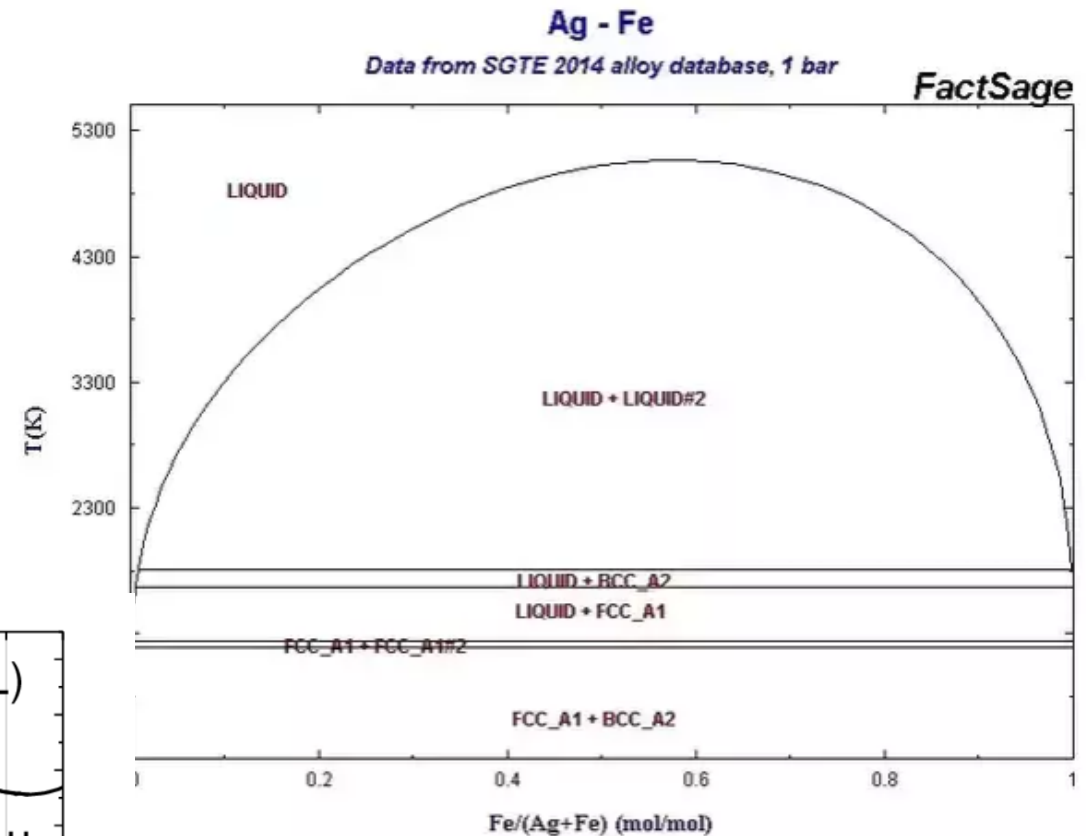


# Liquid Sampling Concerns

- Liquid immiscibility
- Iron and nickel
- Temperature
- Copper addition
- Subsampling



Phase diagram of Au-Fe system.



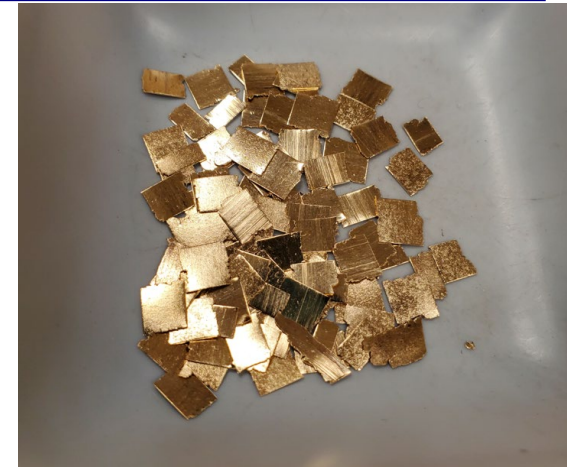
# Subsampling

1. How do we split the sample between customer, refiner and umpire?
2. How do we split the sample to get the 0.5 g analytical aliquot?



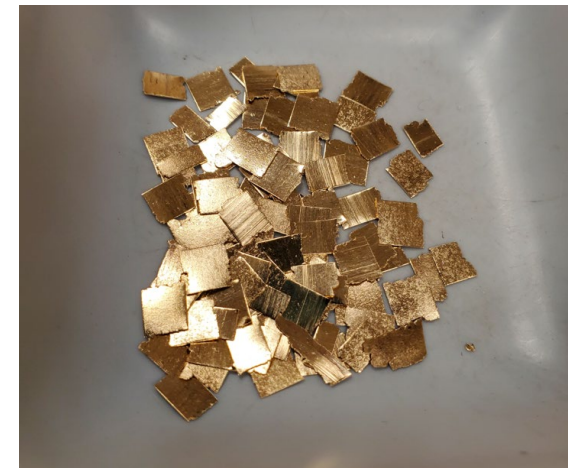
# 1. First 3-way split

- Everybody gets a pin is a common approach
- Better approach is to roll out all pins, chop into shorter segments and split the sample
- Grain sample split is better in this regard



## 2. Lab Subsampling

- Roll out pin and cut ribbon to get 0.5g
- Chop up whole ribbon & choose randomly from pieces for replicates – back to grab sampling
- Ideal would be roll out whole sample and chop into small pieces and to riffle split to 0.5 grams
- Difficult to do – complicated and costly
- Introducing sampling error



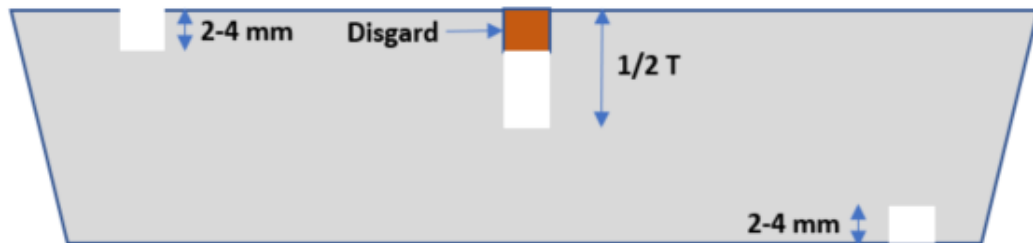
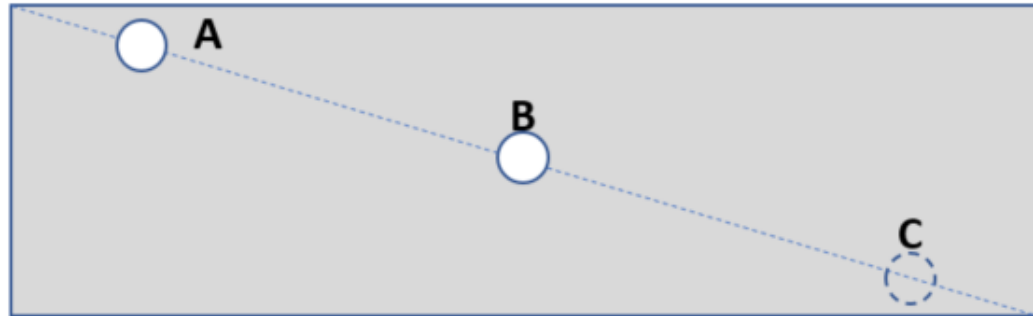
# Solid/Bar Sampling

- Often applied to smaller lots
- Slag layer too thick
- Concerns:
  - Segregation during cooling
  - Top to bottom and sides to center
  - Inclusions form – magnetic
  - Contamination from drilling/cutting tool.
- Drilling, sawing, chipping



# Drilling Patterns

ISO 11596:2021E



AS3515.2:2019

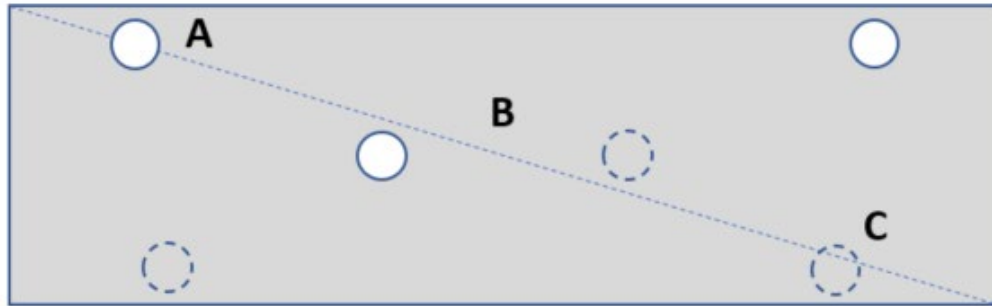


Thin Bars - 15-20 mm - Sample A  
Thick Bars - 30-80mm - Sample A, B, C  
Intermediate - Sample A and B



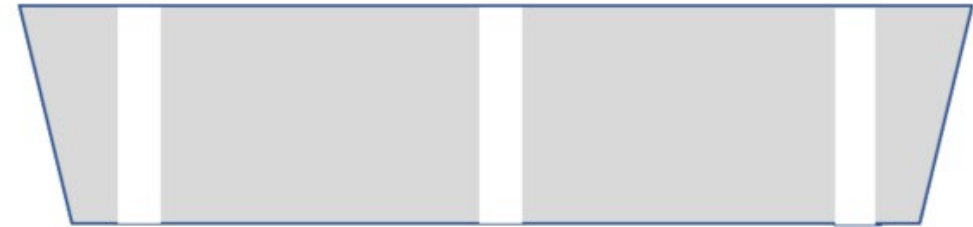
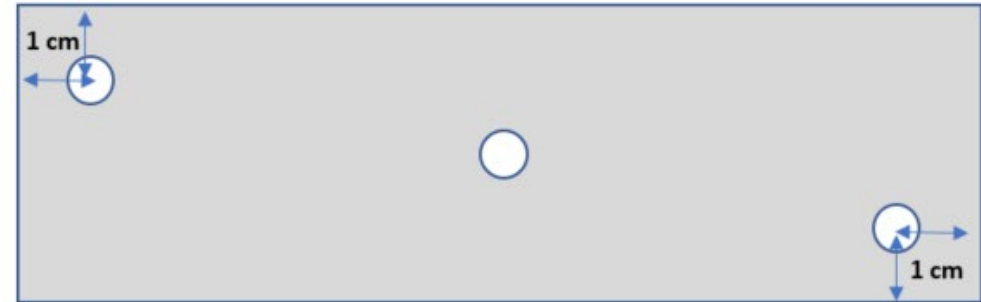
# Drilling Patterns

ASTM B413 - Refined Silver

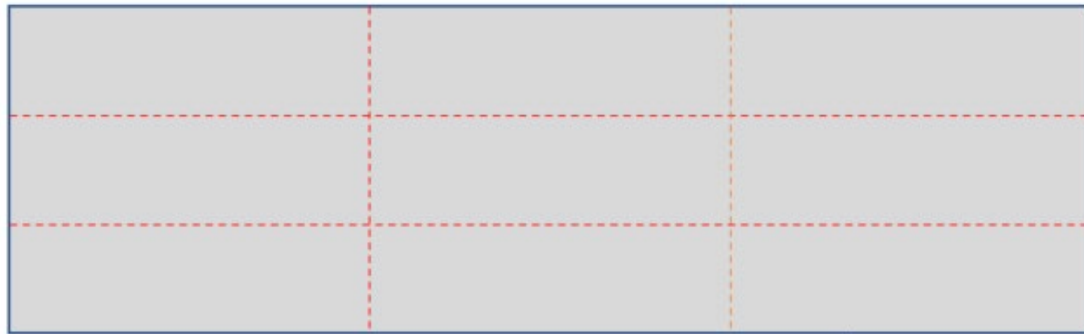


Single Bar - at least 6 drillings  
Multiple Bars - 1 drilling per bar

Alex Stewart International (Andrew Smith)



# Drilling Patterns - Random



Even with random pattern – need to drill through bar or from both top and bottom

---

# Best Practices in Alignment with TOS

- Drill/saw randomly – do not be pattern driven
- Drilling/saw all the way through
- Drilling swarf and cutting filings make it easier to subsample
- Mix drillings from several holes or filings from several cuts

---

# Conclusions

1. Liquid sampling always preferred
2. Issue is subsampling between customer and refiner and then in lab
3. Recommend rolling out, cutting and mixing sample before splitting
4. Random solid sampling – avoid patterns
5. Drill all the way through – combine samples and split