Considerations in the Fire Assay of Copper Bullion

Matthew Cole

Analytical Chemist

Sabin Metal Corporation

Copper Bullion: Why is it important to our industry?

- Large lots melted with copper
- Copper acts as a “Friendly” metal
- Slags off organic compounds
- Copper ingot easy to sample and refine

Sampling Copper Bullion at Sabin

- Sampled while pouring ingots
- “Batting and Shotting”
- Copper shot to analyze
- Repeated 2x more
- Three disks poured to analyze via XRF

How do we analyze Copper Bullion?

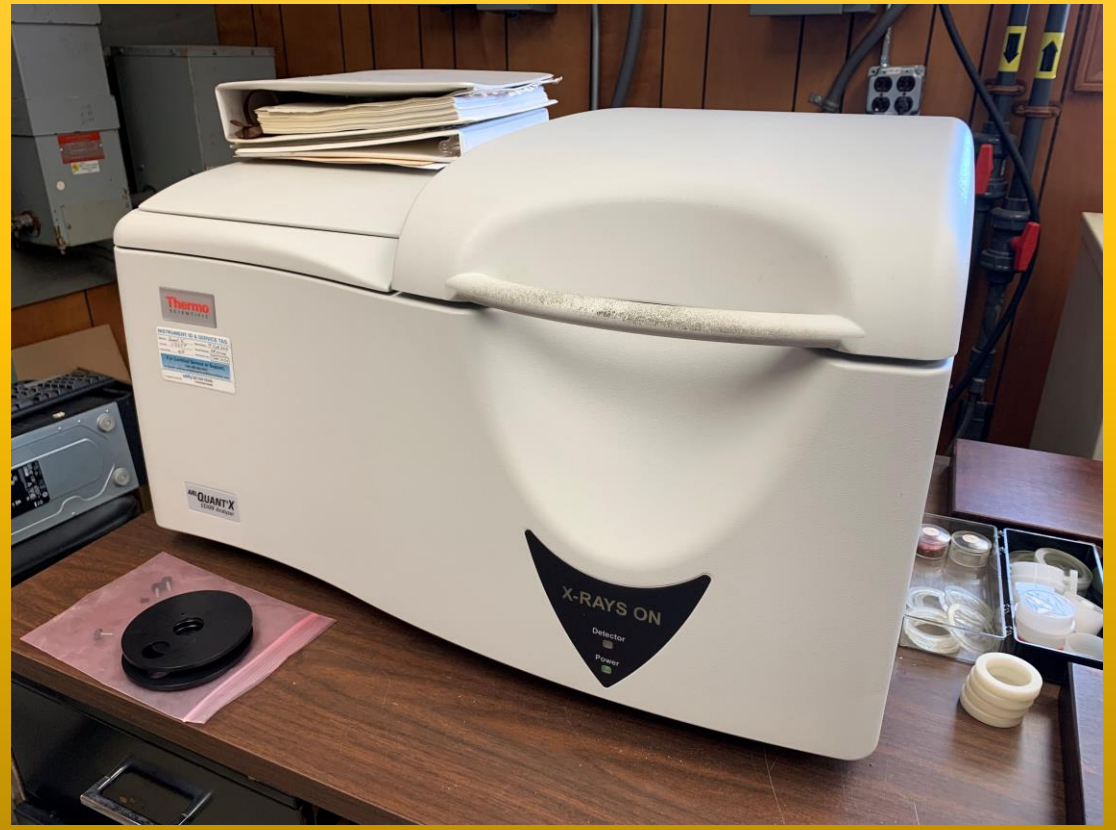
X-Ray Fluorescence

Wet Methods

Fire Assay

X-Ray Fluorescence: A Preliminary Analysis

- The discs are turned on a lathe
- Analyzed using XRF to determine preliminary concentrations
- Confirms homogeneity of melt



X-Ray Fluorescence: A Preliminary Analysis

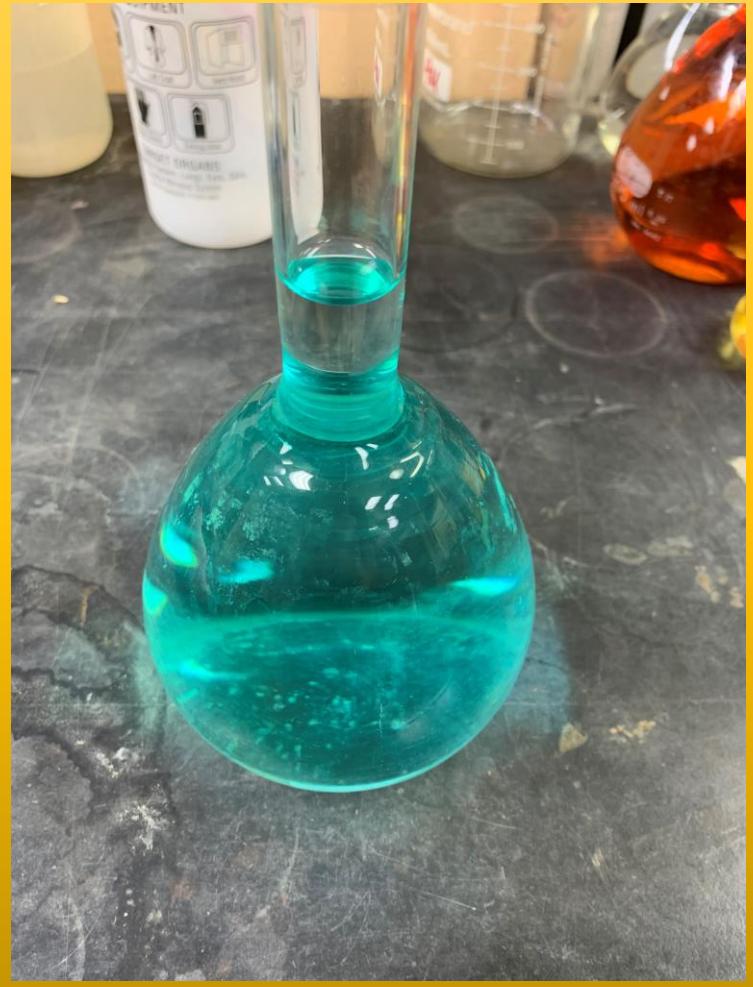
- Limited accuracy of XRF
- Limitations reading specific elements
- Limited to surface of disc
- Disc can segregate

Wet Lab Methods: Preliminary Assay Continued

- Confirm XRF this via Wet methods via ICP-OES
- Copper shot is dissolved in concentrated AR
- Can confirm the homogeneity of the melt even if the XRF cannot

Wet Lab Methods: Preliminary Assay Continued

- High copper solutions interferes with noble metals readings
- Results obtained should remain for internal use only
- Can matrix match standards



Fire Assay: the “Work Horse” of Sabin

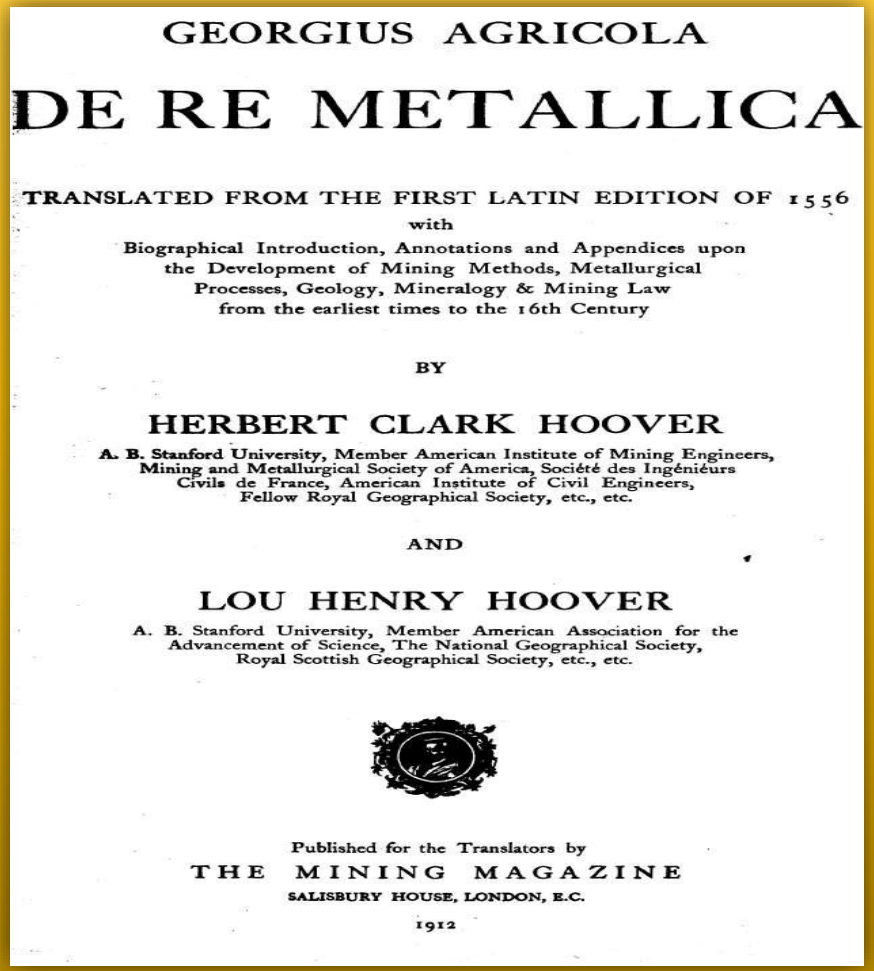
- Use XRF and Wet methods to help Fire Assay
- Fire assay separates precious metals from unwanted base metals
- Oldest form of precious metal chemistry
- Successfully separates noble metals with little to no loss

Fire Assay: the “Work Horse” of Sabin

- Dangers of Fire Assay:
  - Lead
  - Heat

Fire Assay of Low AuPdPt / High Cu Samples

A Brief History



GEORGIUS AGRICOLA

DE RE METALLICA

TRANSLATED FROM THE FIRST LATIN EDITION OF 1556
with
Biographical Introduction, Annotations and Appendices upon
the Development of Mining Methods, Metallurgical
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from the earliest times to the 16th Century

BY

HERBERT CLARK HOOVER

A. B. Stanford University, Member American Institute of Mining Engineers,
Mining and Metallurgical Society of America, Société des Ingénieurs
Civils de France, American Institute of Civil Engineers,
Fellow Royal Geographical Society, etc., etc.

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LOU HENRY HOOVER

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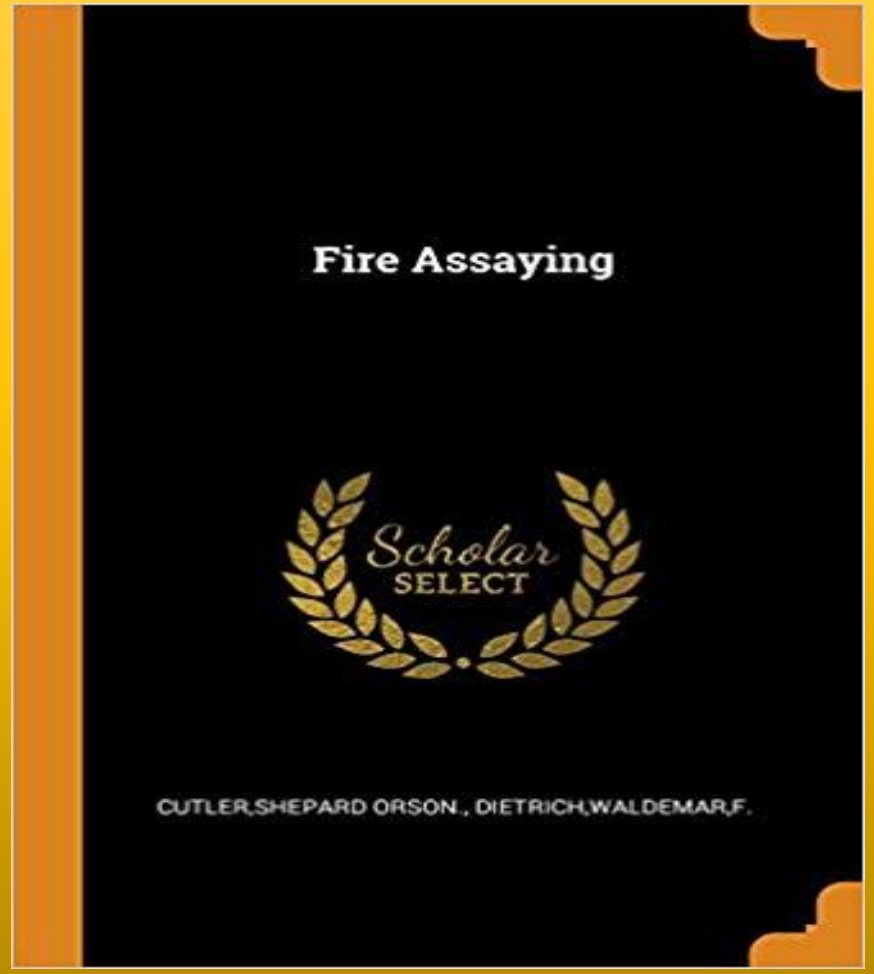
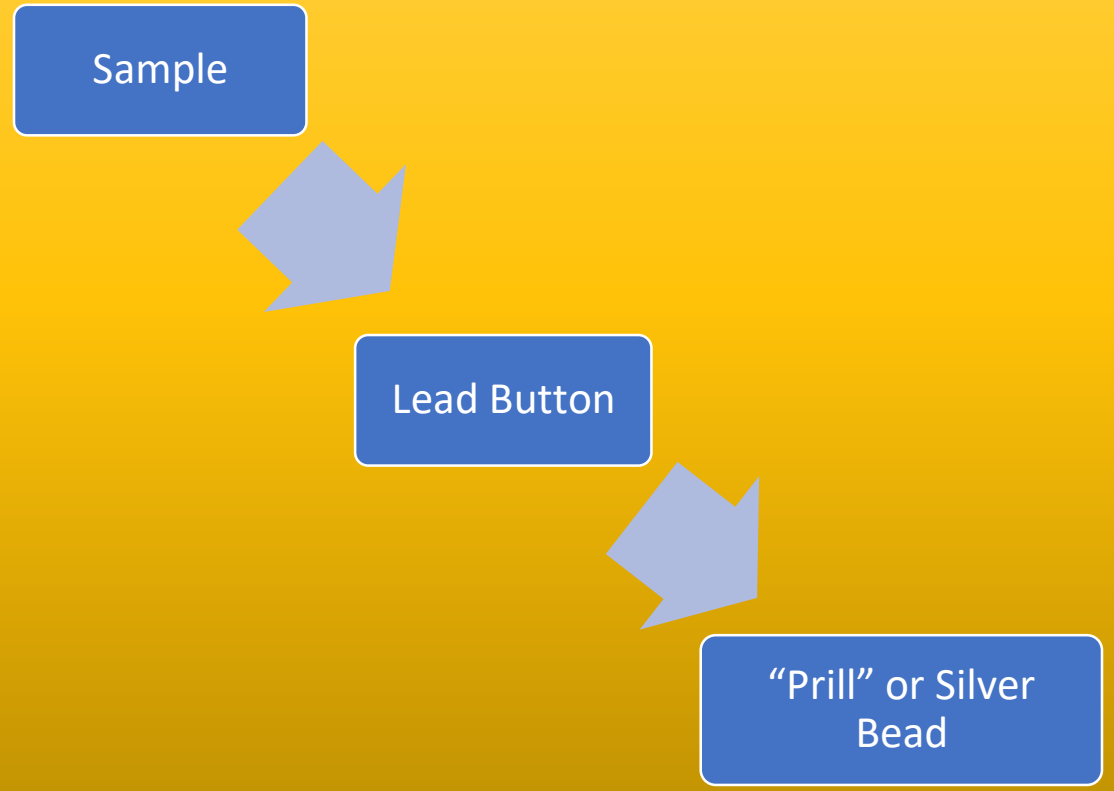


Fire Assay – A Basic summary

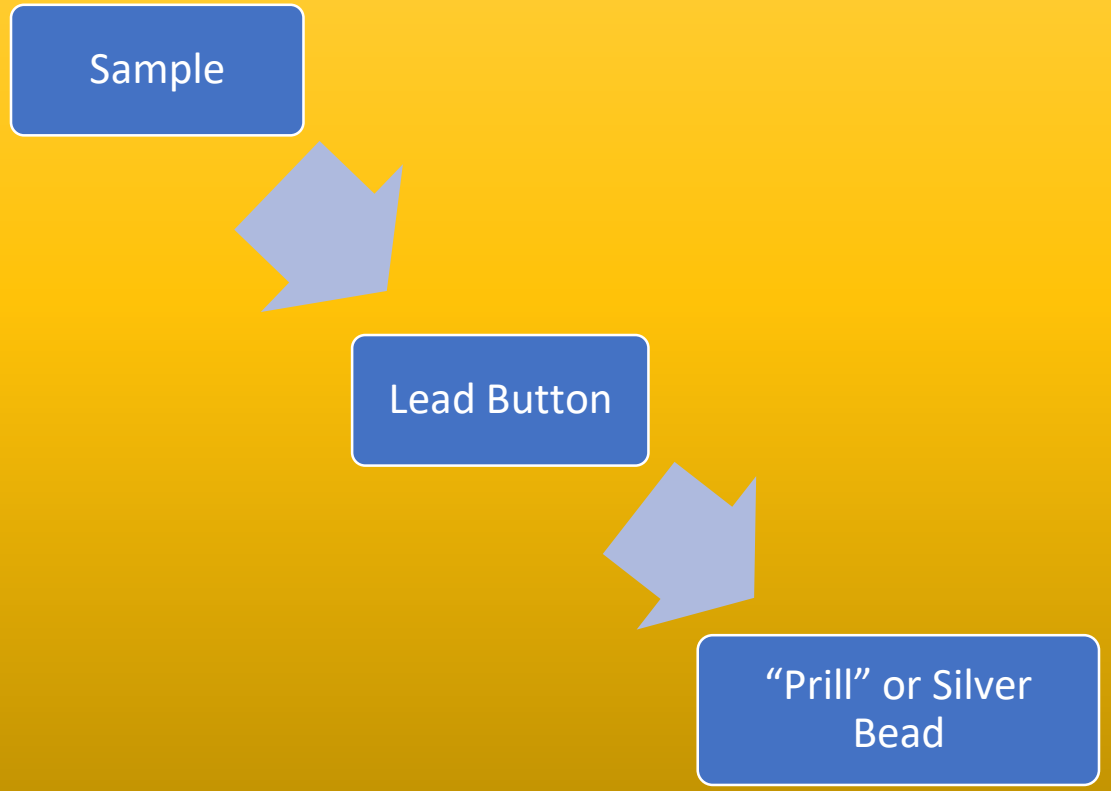
Fire assay is a two step process under extreme heat

1. Fusion
   - Noble metals are alloyed to lead button.
   - Base metal report to glassy slag
2. Cupellation
   - The lead button oxidized and absorbed into a cupel
   - Remaining “prill” or Ag bead contains only noble metals.



Problems Faced by Fusing High Copper Bullion in Fire Assay

- We encounter some problems fusing high copper bullion
- Copper will alloy with lead button



Is There Any Solution?!?!

- YES! Two specifically:
- Lead Button Scorification
  - Lead button is refused to remove copper
- Sulfur/Crucible Method
  - Added sulfur make lead fusible

Is There Scientific Precedent?

- We see that Shepard and Dietrich have studied both procedures and have come to a conclusion
- Shepard and Dietrich state; *“Scorification, thought at one time accepted as standard for smelters, is wasteful of time and materials and gives high silver losses unless slag and cupel corrections are made, because several scorifications are needed before the buttons are pure enough for cupellation.”*

Our Experiment

- To prove Shepard and Dietrich analytically
- To successfully analyze a low grade sample for Gold, Platinum, and Palladium containing high concentrations of copper
- To determine which method is the most efficient/effective for our lab

Fire Assay: Sampling

- Shot obtained is composited
- Preliminary analysis results:

0.085%Au, 0.144%Pd, 0.025% Pt, 92.4% Cu

- 7.5g of sample weighed into a 40g analytical Crucible
- 0.25g of Silver is added

Fire Assay: Flux Determination

- Flux used must compliment sample type and composition in order to have a successful fusion
- Acidic Samples
  - High Si and Al require a basic flux
- Basic Samples:
  - High amounts of oxides, carbon, and heavy base metals require an acidic flux

Fire Assay: Flux Ingredients

- Litharge
- Silica Sand
- Soda Ash
- With the addition of:
  - Borax
  - Flour
  - Sulfur

Litharge (Lead(II)Oxide)

- Basic
- Reduces into metallic lead when carbon is added
- Dissolves difficult to fuse metals (Cu)
- When used in excess can corrode crucible

Silica Sand (Silica Oxide)

- Strong Acid
- Combines with metal oxides to form fusible silicates
- Protects crucible integrity

Soda Ash (Sodium Carbonate)

- Basic
- It also acts as an oxidizing and desulfurizing agent.
- Increases Fluidity
- Very corrosive to the bone ash crucible

Flux Ingredients

SMC Flux:

- 8.5% Silica Sand
- 68.6% Litharge
- 22.9% Soda Ash

Sulfur Flux:

- 3.4% Silica Sand
- 93.6% Litharge
- 3% Soda Ash

Borax (Sodium Tetraborate)

- Strong acid
- Lowers fusing temp.
- Fluxes most metal oxides
- Increases fluidity
- In excess can stick to lead button and causes Ag to sink into cupel

Flour (Carbon Source)

- Reduces lead from lead oxide
- Most common carbon source
- 1g Flour = 11g Pb

Sulfur

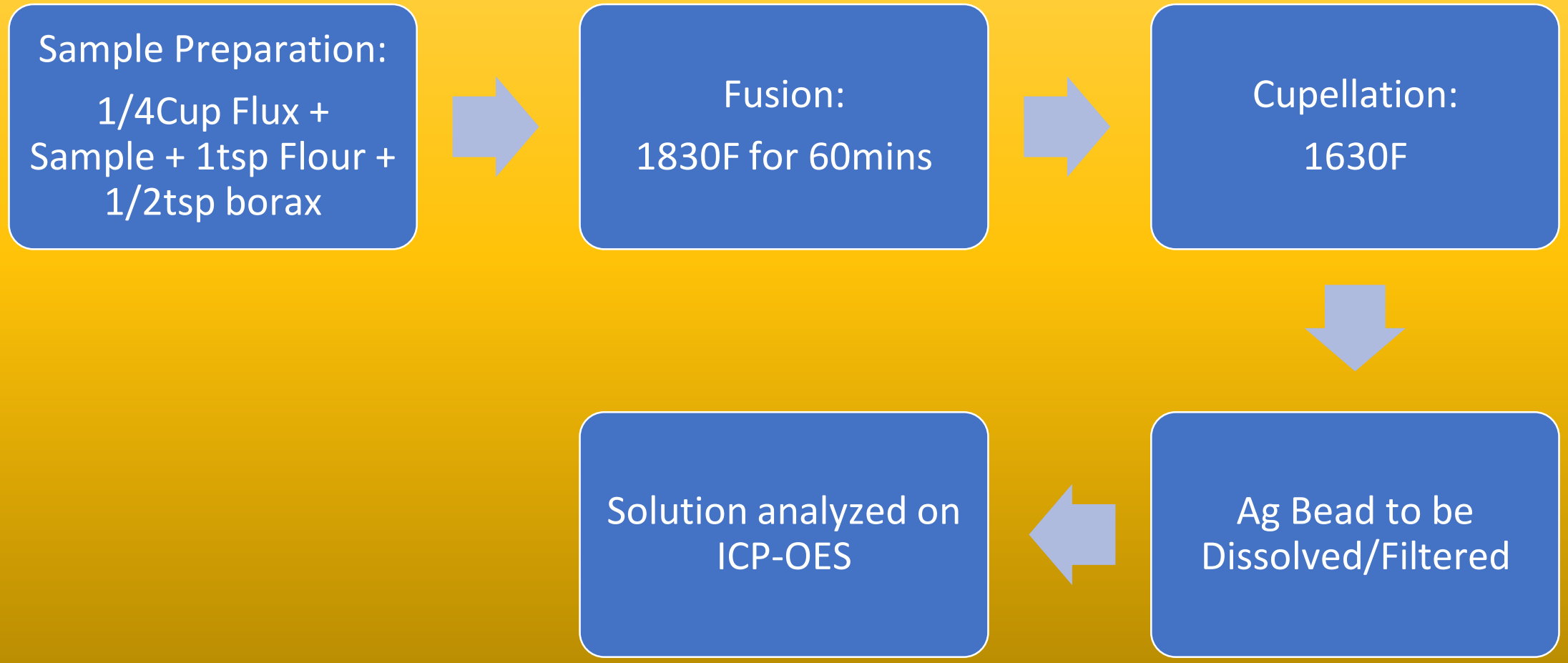
- Two step reaction
- Reacts with High Cu samples to create a Cu Sulfide matte
- When mixed with high litharge flux it is oxidized to form fusible sulfur oxide and copper oxide

Fire Assay: Preventing Loss

Although Fire Assay can be an affective method to analyze precious metals, loss can still occur. This loss can be minimized by:

- Refusing Slag
- Refusing Filter Paper

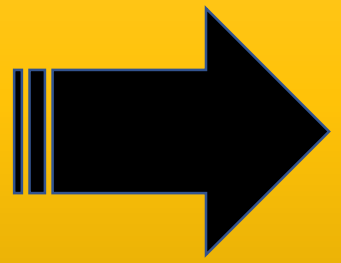
Traditional Fire Assay (no consideration for Cu)

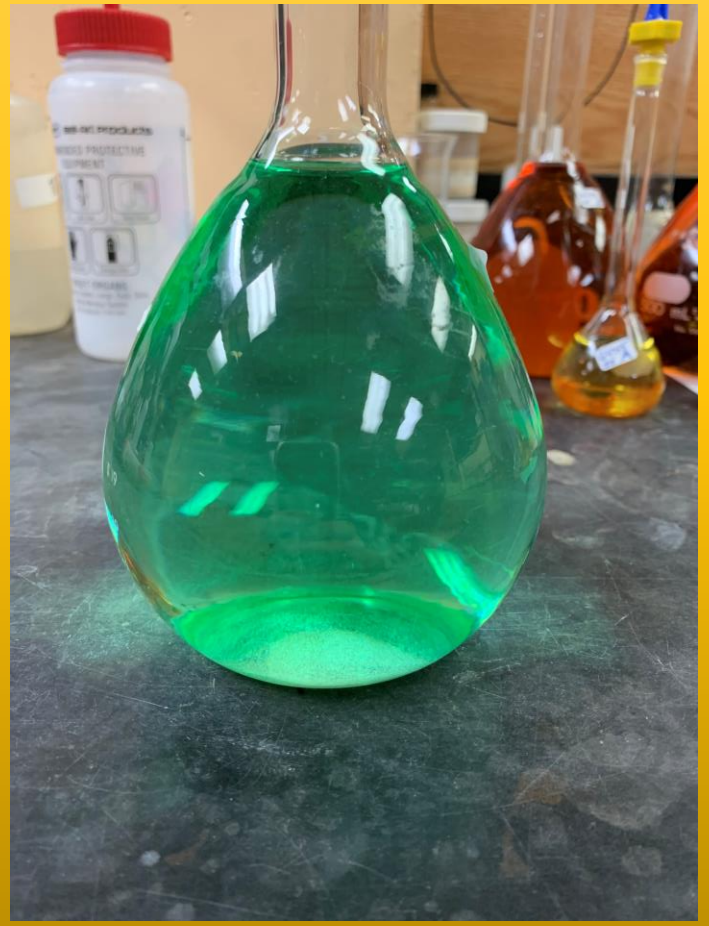


Traditional Fusion

Traditional Fire Assay Fusion Results

Traditional Fire Assay Cupellation/Wet Lab

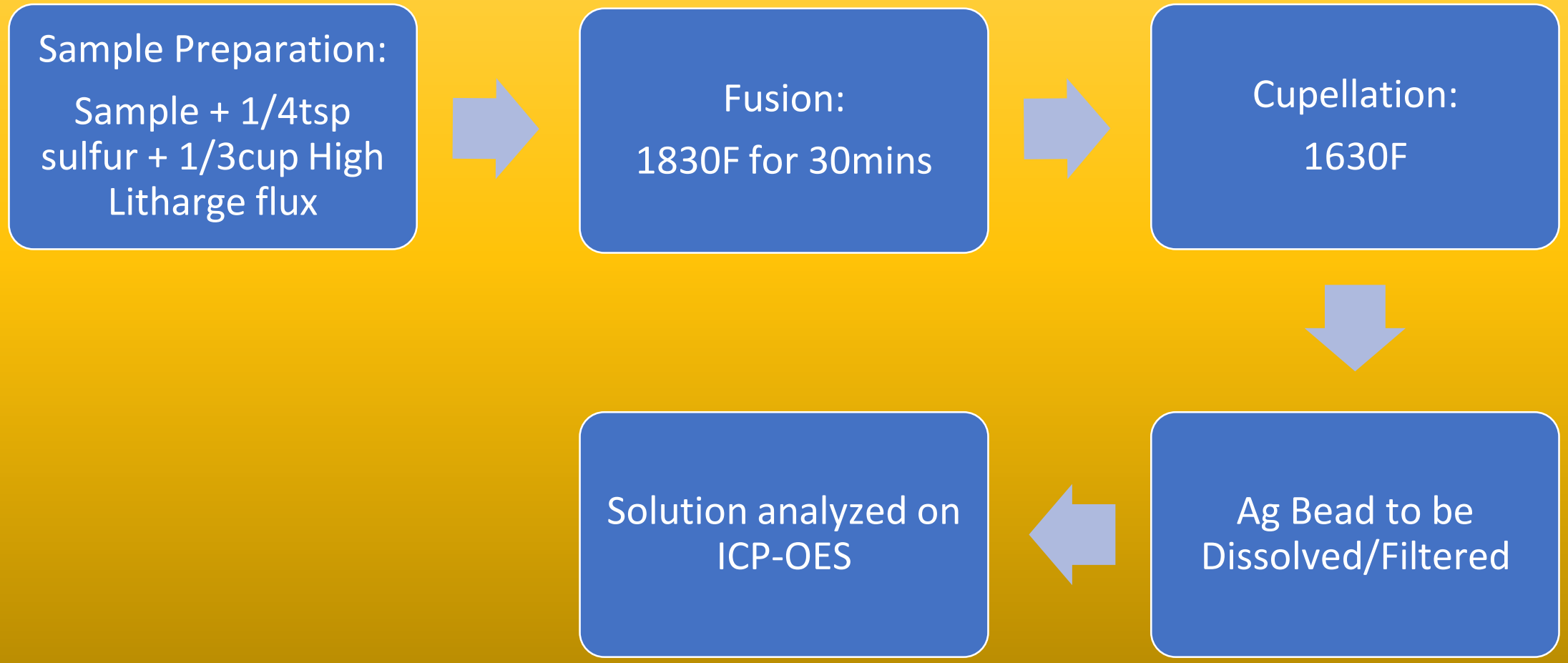




Traditional Fire Assay Results

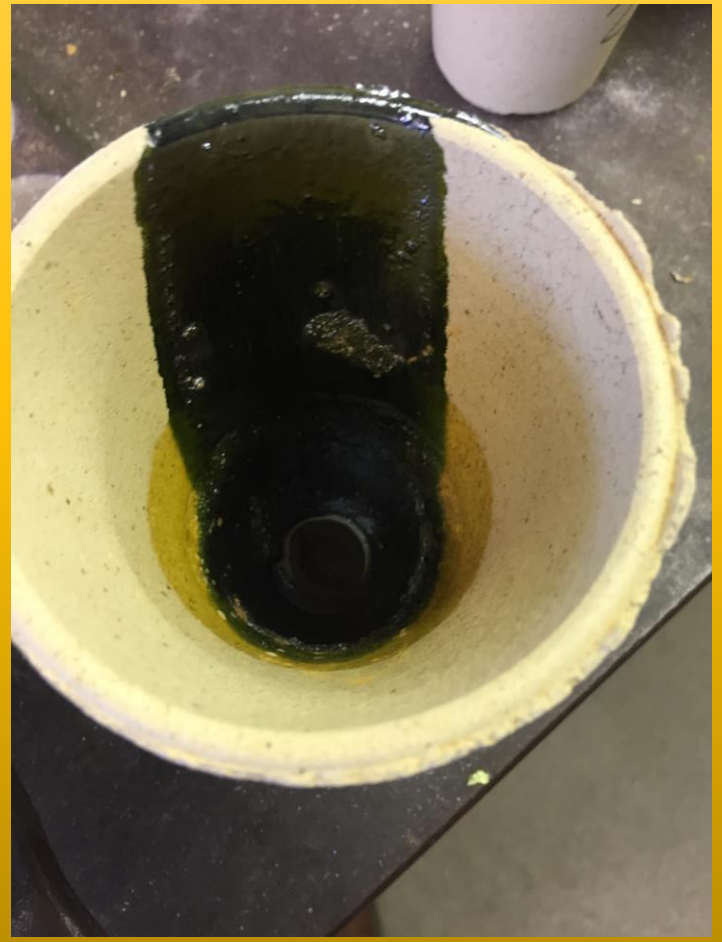
Sample	Au%	Pd%	Pt%
1	0.0779	0.1345	0.0248
2	0.0778	0.1349	0.0250
3	0.0753	0.1331	0.0247
4	0.0774	0.1328	0.0247
5	0.0758	0.1320	0.0249
6	0.0771	0.1342	0.0248
7	0.0752	0.1319	0.0247
8	0.0764	0.1320	0.0249
9	0.0759	0.1333	0.0246
10	0.0764	0.1336	0.0246
	(0.085%)	**(0.144%)**	**(0.025%)**
Average	**0.0765**	**0.1332**	**0.0248**
RSD	**1.31%**	**0.80%**	**0.57%**

Sulfur Method

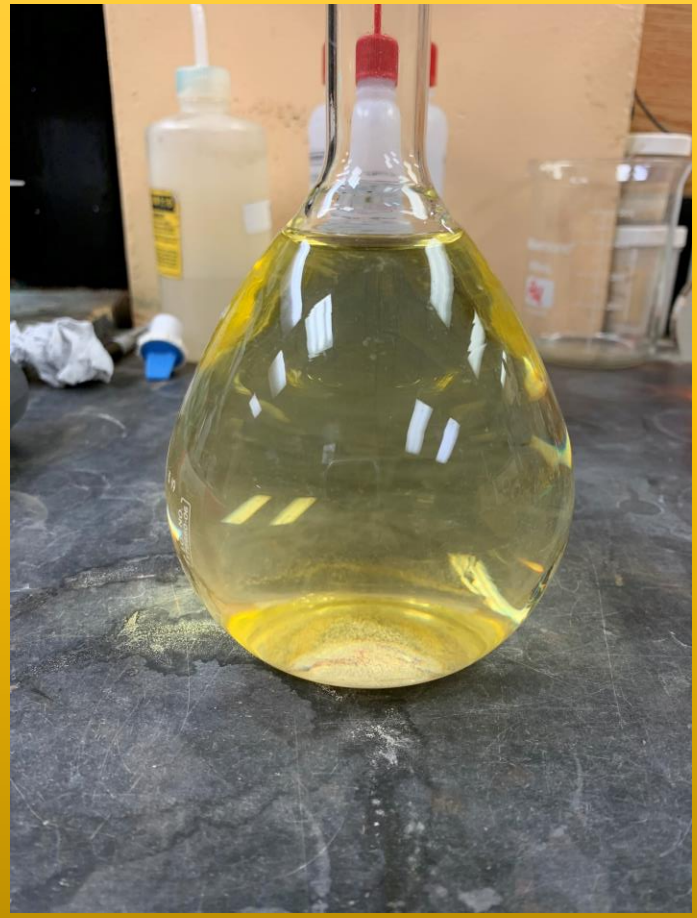


Sulfur Fusion

Sulfur Method Fusion Results



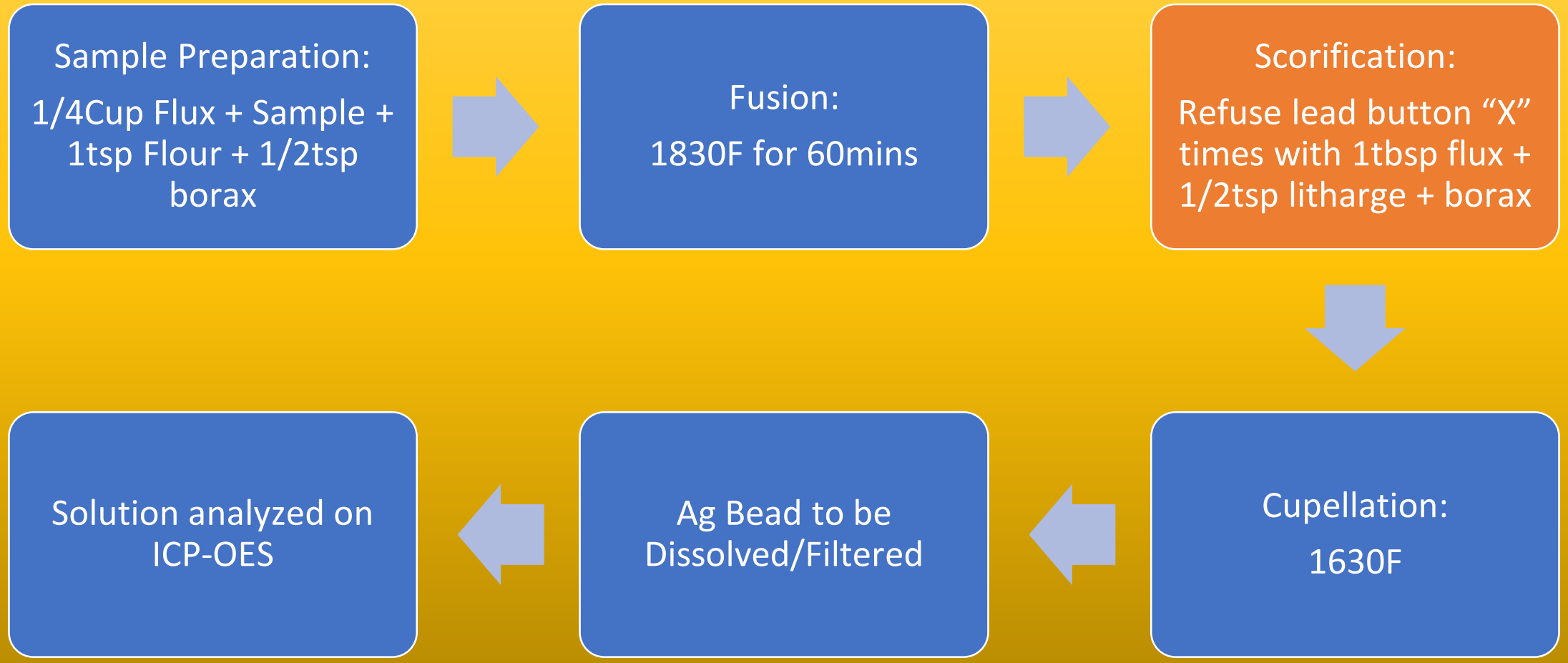
Sulfur Method Cupellation/Wet Lab



Sulfur Method Results

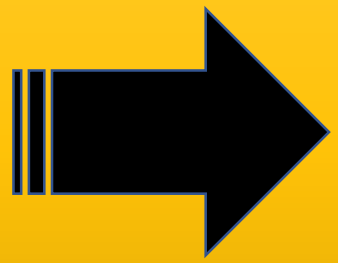
Sample	Au %	Pd %	Pt %
1	0.0853	0.1440	0.0246
2	0.0857	0.1446	0.0247
3	0.0852	0.1441	0.0246
4	0.0860	0.1453	0.0247
5	0.0858	0.1451	0.0247
6	0.0855	0.1443	0.0246
7	0.0860	0.1448	0.0247
8	0.0858	0.1449	0.0248
9	0.0858	0.1444	0.0246
10	0.0864	0.1455	0.0247
	(0.085%)	**(0.144%)**	**(0.025%)**
Average	**0.0857**	**0.1447**	**0.0247**
RSD	**0.41%**	**0.35%**	**0.27%**

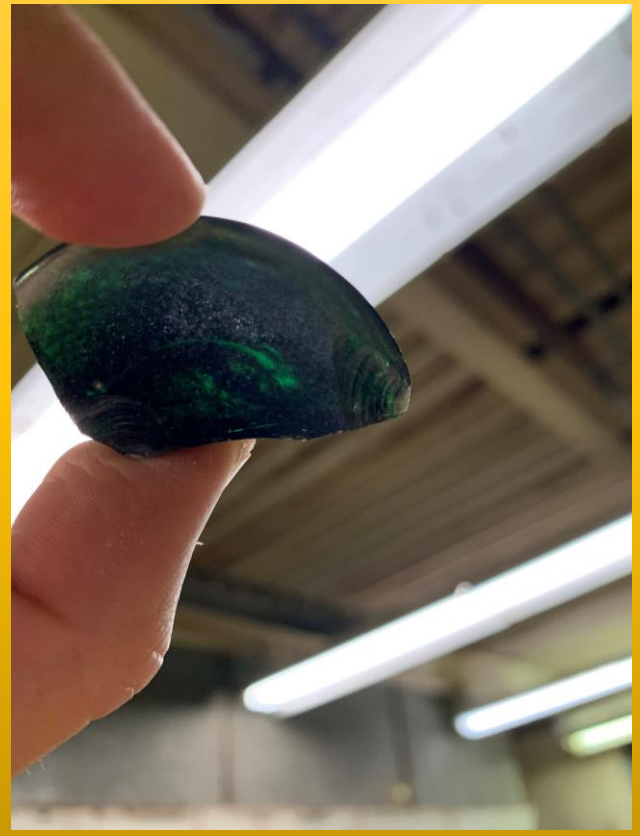
Scorification Method



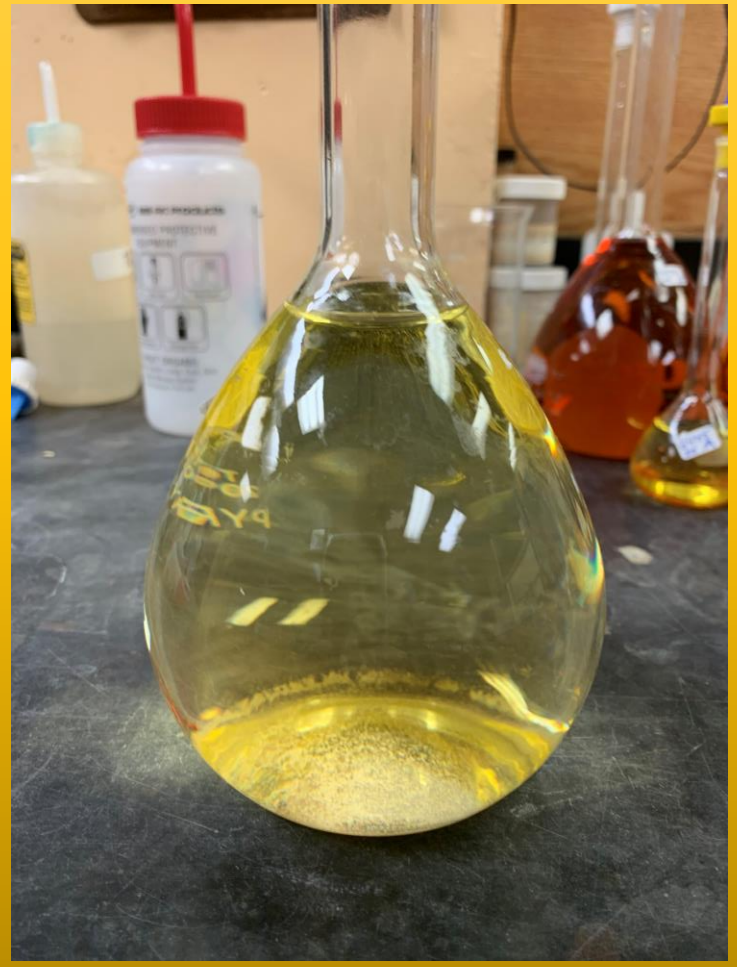
Scorification Method: Scorification

Scorification Method: Scorification Slag





Scorification Method Cupellation/Wet Lab



Scorification Results

Sample	Au %	Pd %	Pt%
1	0.0847	0.1449	0.0246
2	0.0849	0.1441	0.0246
3	0.0851	0.1449	0.0246
4	0.0848	0.1431	0.0244
5	0.0851	0.1442	0.0246
6	0.0849	0.1445	0.0246
7	0.0854	0.1450	0.0247
8	0.0852	0.1446	0.0246
9	0.0854	0.1453	0.0247
10	0.0848	0.1442	0.0245
	(0.085%)	**(0.144%)**	**(0.025%)**
Average	**0.0850**	**0.1445**	**0.0246**
RSD	**0.29%**	**0.44%**	**0.37%**

Results Compared

Method	Average Au	Average Pd	Average Pt
SMC	0.0765%	0.1332%	0.0248%
Sulfur	0.0857%	0.1447%	0.0247%
Score.	0.0850%	0.1445%	0.0246%
PA	0.085%	0.144%	.025%

Conclusions

- Traditional Fire assay methods are unreliable
  - Cu must be accounted for
- Both scorification and sulfur methods can yield accurate results
  - Scorification takes 10x longer
  - Each additional fusion increases risk of loss
  - Confirms *Shepard and Dietrich*
- Sulfur Methods must be done with care!
  - Risk of leaking crucibles
  - Break down furnace hearth and elements ($$$)

Questions?