

Total Gold in Solution by Zinc-Lead Precipitation (Chiddy) (Assay Preparation Module)

Contributed by:

Jim Force

Newmont Gold Company, Carlin

Scope

The objective of this procedure is collection of gold from cyanide solution onto a lead "sponge" and preparation of the lead for cupellation. Sample and silver inquarts are measured into a beaker, cyanide is added to ensure dissolution of precious metals, water and lead solution are added. After heating, zinc powder is added to precipitate the lead and precious metals by an electrochemical displacement process. An excess of hydrochloric acid is added to dissolve zinc, completing the precipitation of precious metals onto the lead sponge. The lead sponge is rinsed with water for removal of zinc chloride, compressed into a cube, wrapped in lead foil, and dried. The lead is then cupelled and analyzed for gold content.

Apparatus and Equipment

- Beakers, glass 1000 mL, graduated
- Pipet, class A 100 mL
- Dispenser, 1 mL
- Balance, 2 decimal places
- Hot plate
- Stir sticks
- Thermometer
- Lab timer
- Beaker tongs
- Watch glass

Reagents and Materials

- Deionized (DI) water
- Silver cyanide solution: in 100 mL, 1 g NaOH, 10 g NaCN, 0.75 g AgNO₃
- Lead acetate solution: in 1 L, 400 g Pb(C₂H₃O₂)₂•3H₂O
- Zinc metal, powdered
- Hydrochloric acid (HCl), concentrated
- Lead foil (optional)

Hazards and Precautions

- Personal Protective Equipment required
- Steel toed shoes
- Safety glasses
- Disposable latex gloves (for handling acids, bases, lead and cyanide)
- Dust mask (for handling cyanide solids)
- Reagents: read MSDS for each chemical used in procedure, especially cyanide and lead.
- Refer to procedures for handling solids and solutions.
- Refer to MSHA training for cyanide safety.
- Refer to emergency response procedures.
- Know the locations of safety shower and eyewash station.

Procedure

1. Preheat hotplate.
2. Label clean 1000-mL beakers for control solution and samples.
3. Transfer 200 mL of control or sample to the appropriate beaker.
4. Add a clean plastic stir stick to each beaker.
5. Bring each solution beaker to approximate volume of 600 mL by adding deionized water.
6. Add 1 mL of silver cyanide solution.
7. Wearing gloves, add 50 mL of lead acetate solution to each beaker and stir.
8. Transfer beakers to hot plate under fume hood and insert thermometer into a central beaker.
9. Allow solutions to reach a temperature of 85° to 97° C.
10. Weigh out 20 ± 0.1 g of zinc powder.
11. Add weighed zinc to each beaker and stir vigorously for 10 seconds.
12. Set timer for 5 minutes.
13. Stir gently without compressing sponge as lead precipitates onto zinc.
14. After 5 minutes, carefully add HCl until the total volume reaches 900 mL.
15. Scrape all zinc and lead from the beaker walls with stir stick.
16. Allow sufficient time for total dissolution of zinc, approximately 30 minutes.
Quality Note: Any zinc remaining in the lead will result in scoria during cupellation, causing loss of gold. Excessive heating time will cause dissolution of the lead sponge, again resulting in gold loss.
17. After 30 minutes, remove beakers from hot plate with beaker tongs.
18. Decant solutions into acid drain in fume hood, flushing with tap water.
19. Rinse sponge twice with tap water, using about 300 mL each time.
20. Compress and then firmly cube the sponge.
21. Place cubes on hot tray and dry for 30 minutes. The sponge may be placed in lead foil to prevent loss of small particles.
22. Before cupelling, test the lead for dryness with a watch glass.
23. Cupel the lead, then part or digest the doré and finish by appropriate measurement technique.