

GC-6 - Analysis of Ores, Feeds, Tailings, and Concentrates: Silver and Gold Assay

Scope This method describes the determination of silver and gold in samples of varying types found in the copper mining industry. The method is also applicable to the determination of lead, zinc, copper, molybdenum, and other elements.

Reagents

- Hydrochloric acid, HCl, concentrated.
- Hydrochloric acid, HCl, 10% (v/v) in deionized water.
- Hydrochloric acid, HCl, 15% (v/v) in deionized water.
- Nitric acid, HNO₃, concentrated.
- Methyl isobutyl ketone (MIBK).

Standard Solutions Standard solutions should be prepared by dilution of the stock standard solutions described under the Standard Conditions for the appropriate element. All standards should contain 25% (v/v) HCl.

Sample Preparation

Ores, Drills, Mill Feeds and Mill Tailings

Weigh 10 g of sample into a 400-mL beaker, add 25 mL concentrated HCl, cover and place on a medium hot plate. After 15 min, **cautiously** add 15 mL concentrated HNO₃. Digest for 20 min and then add 25 mL concentrated HCl and 25 mL deionized water.



Caution

Do not allow the solution to go to dryness after the addition of HNO₃, as some of the gold present may be reduced and not recovered.

Cover and boil to expel nitric acid digestion gases and dissolve all soluble salts. Cool and transfer the solution to **100-mL Nessler color tubes or graduated cylinders**, diluting to 100 mL with cold deionized water. Mix well and filter through **No. 2 Whatman filter paper** into **125-mL Erlenmeyer flasks with screw cap** fittings. The solution may be analyzed at this point for silver, lead, zinc, copper and molybdenum, or may be stored for analysis at a later time. To determine gold, proceed as follows.

Pipet 75 mL of filtrate into a 125-mL separatory funnel. Add 15 mL MIBK, stopper and shake vigorously for 15 seconds. Allow to stand until layers separate, and then draw off and discard the aqueous portion.

NOTE: Formation of a frothy emulsion may prevent a clean separation of organic and aqueous layers. Addition of a drop or two of HF with one or two swirls of the flask (**DO NOT SHAKE**) will normally break the emulsion.

Add 35 mL of 10% (V/V) HCl and shake. Allow the phases to separate and again draw off and discard the aqueous layer. If a yellow (iron) color persists, extract the organic layer with an additional 35 mL of 10% (v/v) HCl. Drain the ketone layer into a 25-mL Erlenmeyer flask or small bottles equipped with tight fitting caps containing a polyethylene liner.

Copper Shipments and Final Concentrates

Weigh 10 gram samples in duplicate into 800-mL beakers, as well as a previously assayed sample of similar composition for use as a standard. Add 25 mL of concentrated HCl to each and evaporate to dryness. Cover and add 80 mL of concentrated HNO₃ in small increments until violent action ceases. Heat on a medium hot plate until the sulfur floating on the acid is light in color. Add 30 mL of concentrated HCl and continue boiling for 20 min.

NOTE: If dark sulfur balls are present, remove the cover, take to dryness, and add 15 mL of concentrated HNO₃. Replace the cover and heat for 15 min. Add 30 mL of concentrated HCl and heat for an additional 20 min.

Cool, make to one liter with a 15% HCl solution and mix. Pipet duplicate 75-mL aliquots of each sample and standard solution into 125-mL separatory funnels, add 15 mL of MIBK and proceed as described under ores, drills, etc., above.

Analysis

For ores, drills, etc., analyze the sample extracts directly versus similarly extracted standards and a blank using the Routine Procedure given in the General Information section. Refer also to the section on Organic Solvents for proper operating conditions.

For copper shipments and concentrates, analyze versus the previously assayed sample. The average of all results should be used as gold may be very unevenly distributed in this type of sample.

Calculations

Gold (oz/ton) = ($\mu\text{g/mL}$ in extract) (0.0583)

Other elements (wt %) = $\frac{(\mu\text{g/mL in solution}) (0.01)}{(\text{g sample})}$

References

1. F. M. Tindall, At. Absorpt. Newsl. 4, 339 (1965).
2. F. M. Tindall, At. Absorpt. Newsl. 5, 140 (1966).
3. F. Tindall, Perkin-Elmer Atomic Absorption Application Study No. 317.