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Digestion of Heavy Sulfide Ores for AAS Analyses

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DIGESTION OF HEAVY SULFIDE ORES FOR AAS ANALYSES

I N T R O D U C T I O N

Heavy sulfide ores such as a high concentration of lead, zinc, antimony or copper sulfide minerals offer special problems of digestion. The sulfide must be oxidized to prevent its forming a thick scum of black elemental sulfur on the surface of the digest liquor. Such a scum will not centrifuge to the bottom of the tube, is difficult to filter, and may adsorb metallic ions from the digest liquor. Following a series of experiments such as roasting and acid oxidation, the detailed procedure given below has been found to be the most satisfactory for these kinds of samples.

P R O C E D U R E

1. Place the weighed, powdered sample in a 400 ml beaker. The beaker will most conveniently have approximate volume marks. Add 25 ml of concentrated HCl.

2. Allow reaction near the boiling point until the HCl has nearly all evaporated. Do not take completely to dryness, but to a thick liquid sludge.

3. Add slowly, in two or three ml intervals, 10 ml of concentrated HNO_3. After the dense fumes have stopped add 50 ml more of HNO_3. Allow this liquor to digest and oxidize until all the floating elemental sulfur has become yellow.

4. Add 25 ml of concentrated HCl to form aqua regia. Digest for 20 minutes.

5. Add a few grains of solid $KMnO_4$. This insures the maintenance of gold in the chloroaurate which is then extractable into MIBK.

6. Add 10 ml of a water solution of 5 percent (W/V) urea to destroy the liquor's nitrate. Nitrate leads to flame instability during the AAS analysis. Allow three to five minutes for the evolution of the nitrate.

7. By evaporation or the addition of HCl bring the liquor volume as indicated by the beaker's volume marks to about 50-60 ml.

8. Transfer the liquid and solids to a volumetric vessel (nessler or centrifuge tube). If the sample is high in antimony there may be a precipitate that is hard to recover from the bottom of the beaker. Add 15 ml HCl and boil for a couple of minutes to dissolve the precipitate. Add this liquid to the digest liquor. Bring to 100 ml volume by adding water after the liquid has cooled to near room temperature. If filtering, a Whatman #2 filter paper is recommended.

9. In the case of antimony ore a milky precipitate will form. This has been found to not interfere with the analysis. The above liquor (50 percent acid) will hold better than 50 ppm of silver. If the solution contains more than 50 ppm silver a new digestion must be made using a smaller sample. It also helps keep the silver in solution if one uses a higher acid concentration by making to volume 25 ml concentrated HCl and the remainder water. This centrifuged or filtered digest liquor is ready to be analyzed directly for silver or the gold extracted by MIBK.