STATE OF ALASKA
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# LABORATORY NOTES NO. 3

# AAS Analyses for Gold and Silver in High Antimony Samples

By

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# AAS ANALYSES FOR GOLD AND SILVER IN HIGH ANTIMONY SAMPLES

## INTRODUCTION

Samples that are high in antimony present special problems of AAS analysis because the addition of water to the aqua regia digestion liquor causes the antimony to hydrolyze and form a dense, milky jell precipitate of antimony hydroxide (?). The jell makes centrifuging or filtering more difficult and the extraction of gold into MIBK extremely difficult. Tests were conducted to see whether water must be avoided by bringing the liquor to volume with acid or whether it is permissible to add water and let the jell form and filter or centrifuge to remove the jell with the other undigested solids.

## EXPERIMENTAL

A sample of ore containing better than 85 percent stibnite was crushed, ground to -100 mesh and thoroughly mixed by rolling. Three, 10-gram samples were digested by the "high sulfide" method which uses nitric acid to oxidize the sulfide. One sample used only HCl to bring to 100 ml volume, one sample used about 25 ml of water, and the third sample used about 50 ml (50:50::acid:water) of water to bring to 100 ml volume. The three samples were then centrifuged and the aliquots taken, followed by filtering and aliquots taken. The above two aliquots allows a test of the centrifuged versus filtered liquor. Filtering results in an essentially clear liquor while with centrifuging considerable hydrolyzed antimony does not go to the bottom and remains in suspension in the liquor. Fifty ml of each of the above aliquots was extracted of its gold into MIBK for the gold analysis. Finally, the precipitate on the filter paper from the 50 percent water sample was dissolved in HCl and the gold and silver determined to see if the jell adsorbs silver or gold ions from the solution. A diagram of the experimental design is shown on the following page.

# RESULTS

The samples on the preceding page were analyzed by AAS in the normal procedure by comparing against standards. The following values were obtained:

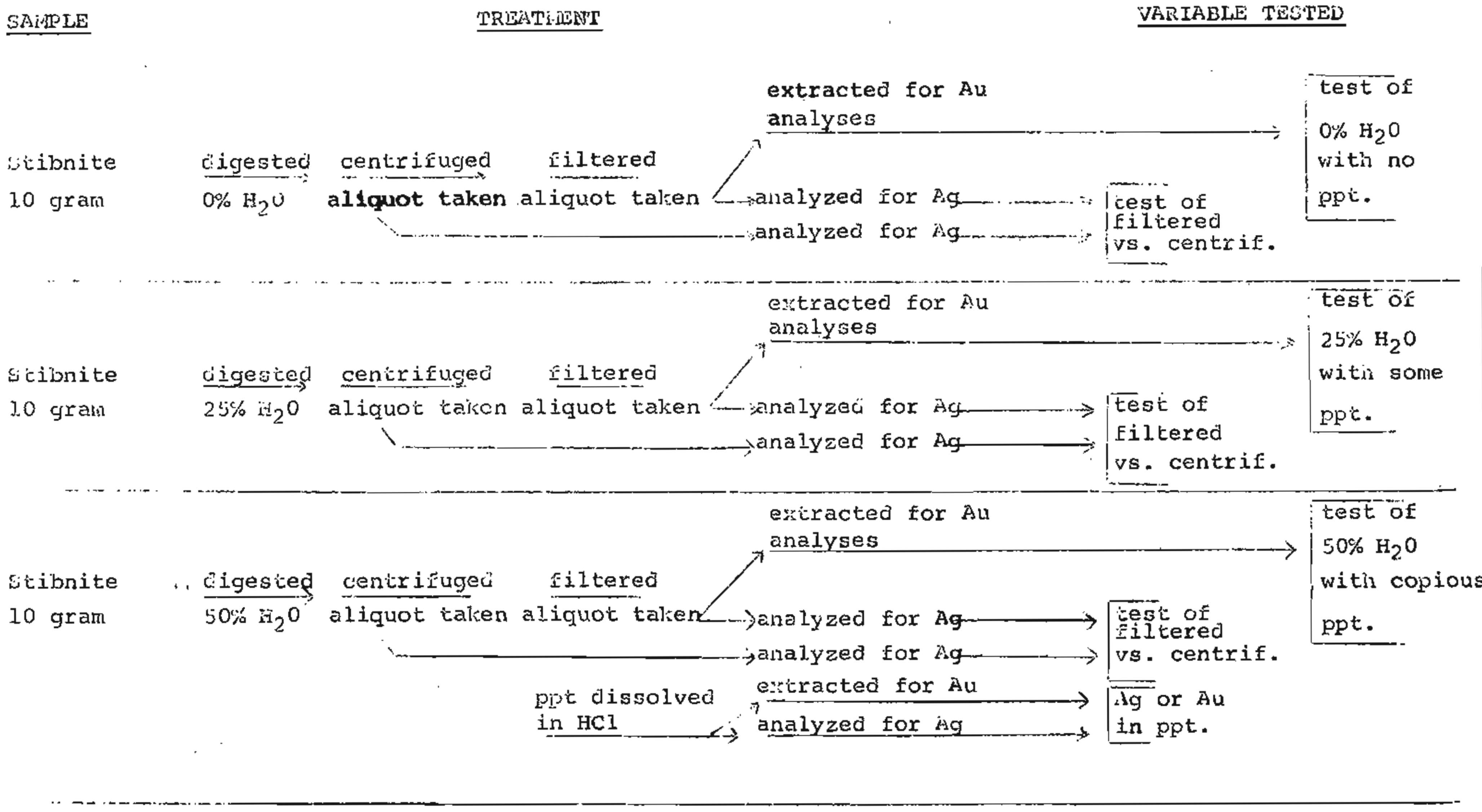
| SAMPLE | SILVER PPM SAMPLE | GOLD PPM SAMPLE |
|---|---|---|
| 0% $H_2O$ centrifuged | 1.50 | ---- |
| 0% $H_2O$ filtered | 1.65 | 1.06 |
| 25% $H_2O$ centrifuged | 1.55 | ---- |
| 25% $H_2O$ filtered | 1.65 | 1.04 |
| 50% $H_2O$ centrifuged | 1.90 | ---- |
| 50% $H_2O$ filtered | 1.85 | 1.40 |
| Hydrolyzed antimony dissolved in HCl | 0.20 | 0.08 |

# DISCUSSION

Looking first at the precipitate, the silver showed 0.2 ppm Ag and 0.08 ppm Au. The amount of solids this represents is estimated to be no more than about 1 gram and so the amount of metals relative to the original sample is about 1/10 of the amount shown, or an essentially insignificant amount. This indicates that the jell does not adsorb the Ag and Au ions, and, therefore, adding water and allowing the jell to form is not delaterious to the analysis. Also, allowing the jell to form and then getting rid of it by filtering or centrifuging leads to a much easier MIBK extraction of the gold because then the jell has already been removed and does not form upon adding dilute HCl to wash the MIBK of iron. It is surprising that the higher, and not the lower, values were obtained from the 50 percent water samples. The differences observed are large enough to be significant analytically and probably indicates more silver and gold in solution from the 50 percent $H_2O$ samples, though I don't understand why.

There was not a significant difference in Au-Ag values between the filtered and centrifuged methods of preparation and so either could be used.

# EXPERIMENTAL DESIGN



Based on these experiments, I make the following recommendations for high antimony samples:

1. Bring the digestion liquor volume to about 50 ml, by evaporation or by the addition of HCl, just prior to bringing to volume.

2. Bring the liquor to 100 ml volume by the addition of $H_2O$.

3. Either filtering or centrifuging may be used to remove the solids.

4. During the extraction of gold into MIBK, add about 50 ml of 0.1N HCl to wash the MIBK, but shake this first wash very little or none. Most of the iron will come out of the MIBK without shaking, unhydrolyzed antimony will hydrolyze in this dilute acid and is easily removed from the MIBK if it is not shaken.. If the funnel is shaken it will be difficult or impossible to recover the MIBK. Wash the MIBK a second time with mild shaking, say four or five strokes, drain off the aqueous portion and wash again if necessary. Usually the MIBK does not form a continuous organic layer, but a large number of sphercial droplets of MIBK surrounded by an aqueous fraction. This is not a problem if a half hour or an hour's time is allowed before the analysis. Slowly the MIBK will coalesce and the water will settle away from the MIBK. Just take care that during the analysis only the MIBK and not any of the aqueous bottom portion is asperated into the analytical flame.