

1910

## An investigation of certain methods for the determination of gold in cyanide solutions

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### Recommended Citation

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THESIS FOR THE  
DEGREE OF BACHELOR OF SCIENCE  
IN GENERAL SCIENCE.

*T209*

-SUBJECT-

AN INVESTIGATION OF  
CERTAIN METHODS FOR THE DETERMINATION  
OF GOLD IN CYANIDE SOLUTIONS.

-BY-

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10912

1910.

Approved:

*Durward Copeland*  
*per H. P. Mann.*

AN INVESTIGATION OF CERTAIN METHODS FOR THE  
DETERMINATION OF GOLD IN CYANIDE SOLUTIONS.

Some questions having been raised as to the reliability of the various methods for the quantitative determination of gold in cyanide solution, this work was undertaken with the view of determining the relative accuracy and convenience of these methods.

I prepared four solutions as follows:

- I. This solution contained 0.05 of 1% potassium cyanide (KCN) and 0.05 oz gold per ton.
- II. This solution contained 0.5 of 1% K C N. and 1 oz. gold per ton.
- III. This solution contained 0.5 of 1% KCN and 0.05 oz. gold per ton.
- IV. This solution contained 0.05 of 1% KCN and 1 oz. gold per ton.

These solutions were prepared by dissolving C P. KCN. in distilled water and adding the required amount gold in the form of the chloride. The gold chloride was prepared by dissolving fine gold in aqua regia, evaporating off the excess acid and adding the gold

AN INVESTIGATION OF CERTAIN METHODS FOR THE  
DETERMINATION OF GOLD IN CYANIDE SOLUTIONS.(continued)

chloride to the KCN. solution. With each solution for  
comparison, assays were made, using <sup>five</sup> assay ton samples  
and five assays were made using five assay ton samples.

The solutions were accurately measured, a standard  
burette being used, for this purpose. The samples  
taken were assayed by each of the following methods:

- I. The evaporation on lead foil or the lead boat method.
- II. The A. F. Grosse, or the silver nitrate method.
- III. The litharge method.
- IV. The aluminium sulphide method.
- V. The lead acetate method.
- VI. The Walter H. Vigoe, or the copper sulphate method

These methods with the results obtained will be  
described in detail.

-LEAD BOAT METHOD- (Continued)

Ounces per ton.

I assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.05	0.05	0.05	0.97
2		0.95	0.05	0.97
3	0.04	0.95	0.04	1.00
4	0.06		0.05	0.98
5	0.04	0.90	0.05	0.99
aver- age.	0.0475	0.925	0.05	0.982

Ounces per ton.

5 assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.26	4.97	0.25	4.90
2	0.24	4.99	0.24	4.95
3	0.24	4.96	0.25	4.97
4	0.24	4.97	0.24	4.95
5	0.26	4.98	0.24	4.98
aver- age.	0.25	4.98	0.244	4.96

-LEAD BOAT METHOD-

I. For this method a lead boat or box is made by turning up the edges and ends of a piece of lead foil. For 1 assay ton of solution about 20 gms. of lead foil was used, and for 5 assay tons of solution about 75 gms. of lead foil was used. After the solution has been measured into the lead boat, the boat is placed on a hot plate and the solution slowly evaporated to dryness. After complete evaporation enough silver was added to inquart the gold, the lead boat was rolled into a ball and cupelled at the ordinary cupellation temperatures. The resulting silver-gold bead or button is brushed to clean.

The button is then placed in a porcelain crucible, and 5 to 10 cc. of dilute nitric acid ( $\text{HNO}_3$ ), one acid to three of water is added. This nitric acid solution dissolves the silver and leaves the gold as an insoluble residue. This residue is then washed three times with distilled water, dried, annealed, cooled and weighed. The results obtained by this method are shown in the following tables:

-A.F. CROSSE, or the SILVER NITRATE METHOD-

III. This method consists of precipitating the gold *with Silver Nitrate*. The samples were measured as in the preceding cases. The silver nitrate solution was prepared by dissolving 25 gms. of silver nitrate in 200 c.c. of distilled water. This solution was added until no more precipitate formed, then a slight excess was added. The solution was heated, filtered and the precipitate dried, wrapped in lead foil, scorified and cupelled. The silver-gold button was treated in the same manner as in the preceding cases.

The results obtained by this method are given in the following tables.

Ounces per ton  
1 assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.05	1.00	0.04	0.70
2	0.05	I.	0.05	0.75
3	0.06	0.96	0.06	
4	0.04	0.99	0.05	0.85
5	I.	0.99	0.04	0.87
aver- age.	0.05		0.989	0.793

-A.F. GROSSE, or the SILVER NITRATE METHOD-(Continued)

Ounces per ton  
5 assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.23	4.70	0.24	4.50
2	0.27	4.87	0.25	4.00
3	0.24	4.96	0.26	4.70
4	0.26	4.95	0.23	4.50
5	0.19	4.89	0.21	
aver- age	0.22	4.87	0.23	4.45



-THE LEAD OXIDE or LITHARGE METHOD-

II. In this method a measured amount of solutions is evaporated in a casserole or evaporating dish, almost to dryness. The lead oxide is then added to absorb the remaining solution which contains the gold. The lead oxide is then carefully removed from the casserole or evaporating dish, enough silver is added to inquart the gold. The litharge is then charged into a crucible with a suitable flux and reducing agent, and fused. The resulting lead button is cupelled and the gold-silver bead treated in the same manner as in the lead boat method.

The results obtained by this method are given in the following tables.

Ounces per ton

I assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.04	0.97	0.05	0.96
2	0.03	0.95	0.05	0.95
3	0.03	0.96	0.06	0.96

-THE LEAD OXIDE or LITHARGE METHOD-(Continued)

Ounces per ton  
I assay ton samples.

	Sol.No.I.	Sol.No.II,Sol.No.III.	Sol.No.IV.
4		0.87	0.05
5	0.04		0.04
aver- age.	0.0325	0.937	0.05

Ounces per ton  
5 assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol. No.III.	Sol. No.IV.
1	0.26	4.00	0.23	4.00
2	0.25	4.80	0.24	4.25
3	0.26	4.68	0.25	
4	0.24	4.50	0.25	4.00
5	0.20	4.75	0.26	3.70
aver- age.	0.242	4.56	0.25	3.98

-THE ALUMINIUM SULPHIDE METHOD-

In this method the gold is precipitated from the KCN solution with aluminium sulphide. The aluminium was prepared by fusing 60 gms. of finely powdered aluminium with 300 gms. of galena (Pbs). Aluminium sulphide is very unstable and should be kept in a closed vessel, so that the air cannot come in contact with it, as the moisture from the air will form with the aluminium sulphide, hydrogen sulphide. The aluminium is added in lump form, two or three grams being sufficient to precipitate the gold, then the solution is allowed to stand until all action has ceased, filtered and the precipitate wrapped in lead foil with enough silver to inquart the gold, scorified, and the lead button expelled, the silver-gold bead is treated in the same manner as before.

The results obtained by this method are given in the following tables.

Ounces per ton			
I assay ton samples.			
Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.90	0.04	0.70

-THE ALUMINIUM SULPHIDE METHOD- (Continued)

Ounces per ton  
I assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
2	0.04	1.00	0.04	0.75
3	0.04	0.94	0.03	0.85
4	0.06	0.95	0.05	0.90
5	0.7	0.96	0.04	0.90
<i>Average</i>	0.05	0.95	0.04	0.83

Ounces per ton  
5 assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.19	4.50	0.23	4.50
2	0.20	4.45	0.23	4.70
3	0.21	4.70	0.24	4.87
4	0.17	4.80	0.25	4.70
5	0.17	4.50	0.23	4.80
<i>average</i>	0.19	4.59	0.24	4.71

-THE LEAD ACETATE METHOD-

v. This method consists of precipitating the gold with zinc dust and then adding lead acetate to form a lead sponge which acts as a collector for the gold. The zinc is then dissolved with hydrochloric acid.

A 10% solution of lead acetate was prepared by dissolving 10 gms. of lead acetate crystals in 100 c.c. of distilled water. About 1/4 gm of zinc dust was added to the KCN solution. Then 10 c.c. of the 10% solution of lead acetate was added to the one assay ton samples and 50 cc to the 5 assay ton samples. This caused a lead sponge to form which collected the gold. Hydrochloric acid was then added to dissolve the excess <sup>zinc</sup>. The lead sponge is allowed to remain in the solution until it is attacked by the acid. The lead sponge is then removed, being careful not to tear it apart. Enough silver is added to inquart the gold and the sponge is then wrapped in lead foil and cupelled.

The silver-gold button is treated as in the preceding methods.

The results obtained are given in the following tables.

-THE LEAD ACETATE METHOD- (Continued)

Ounces per ton  
I assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.05	0.95	0.05	0.97
2	0.04	0.90	0.04	0.95
3	0.05	0.94	0.05	0.90
4	0.07	0.97	0.05	0.90
5	0.04	0.99	0.04	0.90
aver- age	0.05	0.95	0.046	0.90

Ounces per ton  
5 assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.14	4.50	0.25	4.75
2	0.21		0.26	4.50
3	0.19	9.80	0.26	4.60
4	0.19		0.25	
5		4.70	0.24	4.70
aver- age.	0.18	4.66	0.254	4.64

-THE WALTER H. VIGOR, or the COPPER SULPHATE METHOD-

This method consists of precipitating the gold from cyanide solution with copper sulphate. The copper sulphate solution, which was prepared by dissolving copper sulphate crystals in distilled water, was added until the precipitate ceased to form and then a few drops in excess were added. The solution was filtered and the precipitate was wrapped in lead foil with enough silver to inquart the gold. The precipitate was then scorified, the resultant lead cupelled and the silver-gold bead treated in the same way as in the preceding methods.

The results obtained by this method are given in the following tables.

	Ounces per ton			
	I assay ton samples.			
	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.06	0.97	0.05	0.90
2	0.05	0.98	0.06	0.94

-THE WALTER H.VIGOE, or the COPPER SULPHATE METHOD-(Continued)

Ounces per ton  
1 assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
3	0.07	0.99	0.04	0.90
4	0.04	0.96	0.04	0.89
5	0.05	1.00	0.05	0.95
aver- age	0.053	0.98	0.049	0.92

Ounces per ton  
5 assay ton samples.

	Sol.No.I.	Sol.No.II.	Sol.No.III.	Sol.No.IV.
1	0.06	4.50	0.24	3.70
2	0.05	4.60	0.25	4.00
3	0.05	4.80	0.25	3.00
4	0.06	4.65	0.24	
5	0.06	4.70	0.26	3.25
aver- age.	0.06	4.65	0.25	3.49



-COMPARISON of the DIFFERENT METHODS-

The lead boat method is accurate on almost all of the solutions compared. Better results were obtained on solutions No. III. and IV. These solutions contained 1% 0.5 of KCN. and 0.05 oz. gold per ton and 0.05 of 1% KCN and 1.00 oz. gold per ton. Fairly good results were on the other two solutions. The low results on these solutions were probably due to some mechanical error. Both the 1 assay ton samples and the 5 assay ton samples gave good results.

The silver nitrate, which is a precipitating method, is not good in weak KCN solutions. On solution No. I. very poor results were obtained with one assay ton samples of the same solution very poor results were also obtained. On the strong KCN and weak gold good results were obtained, while on the strong KCN and strong gold poor results were obtained. On 5 assay ton and weak KCN and strong gold the average for the 5 assay was just 0.796 oz. per ton. The solution actually containing 1.00 oz. per ton.

By the litharge method poor results were obtained on all the solutions, except No. I, II and III. of the I assay ton samples.

The aluminium sulphide method gave good results on solutions I, II, and III, for the I assay ton samples. Also fairly good results were obtained on solution No. III with the 5 assay tons samples.

The lead acetate method gave good results on solutions I, II and III of the I assay ton samples, also on solution, No. III of the 5 assay ton samples.

The copper sulphate method gave good results in solutions I, II and III of the I assay ton samples, also on solution No. III, of the 5 assay ton samples. The other results obtained were very poor. Very little precipitate was formed in solution No. I of 5 assay ton samples.

All the methods are easy of operation. In the aluminium sulphide method it takes sometime to filter the precipitate. The lead oxide method is hindered, on account of the fact that the dried litharge tends to stick to the casserole.

The time of operation is short in all the

precipitation methods.

The evaporating methods are slow because it takes some time to evaporate the 5 assay ton samples to dryness.

The lead boat method is probably the best method for all solutions, ~~also~~<sup>care</sup> has to be taken in constructing the boat for they are apt to leak. The lead acetate method probably requires less time, and is almost as accurate as the lead boat method.

All the precipitating methods are inaccurate in weak KCN solutions, especially, so in weak KCN. and strong gold it seems that there is not sufficient precipitate formed to collect all the gold.

Any of the methods compared are good on strong KCN solutions ~~or~~<sup>and</sup> weak gold.