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ANALYSIS OF COPPER-PALLADIUM-GOLD-SILVER CONCENTRATES

By C. W. Davis<sup>1</sup>

A copper-palladium-gold-silver ore is being mined at Salt Chuck, Prince of Wales Island, Alaska, from which a concentrate is produced that contains 40 to 50 per cent copper, and considerable amounts of palladium, gold and silver. There has been much difficulty in obtaining assays for palladium, gold and silver from the mine, smelter, sampler, and umpire that check within reasonable limits. While the results for copper content have not been unusual, the results for palladium have varied as much as 0.8 oz. on 3 oz. material, results for gold differed by 0.1 oz. on 1.6 oz. materials, and for a silver content of 7 oz., results differed by as much as 4.0 oz.

The Bureau of Mines was requested to determine the causes of variation in the results of the different assayers, and to develop a satisfactory procedure for analyzing such ore.

Three samples of concentrate were received for analysis, two small lots that had previously been assayed by the various laboratories, and one large sample. The large sample was dried and ground in a ball mill, put through a 150-mesh screen, and then returned to the ball mill for thorough mixing. The small samples were ground to pass a 150-mesh screen and then mixed on a mixing wheel.

The large sample (No. 1) was analyzed by five different procedures, one of the small samples (No. 2) by three procedures, and the other (No. 3) by two procedures (the two small samples being too small for further tests). The different procedures tried and the results obtained are presented below. The samples of concentrate were from ore mined and milled by the Alaska Palladium Co. at Salt Chuck.

Procedure No. 1, "Combination Assay"

Samples of one assay ton were roasted at 500° C. for two hours, then digested with (1:10) H<sub>2</sub>SO<sub>4</sub>; a little NaCl was added with stirring, followed by a few drops of KI solution; lead acetate was introduced with stirring,

1 - Associate chemist, Rare and Precious Metals Experiment Station, (Reno, Nev.), Bureau of Mines, Department of Commerce.  
In cooperation with the Mackay School of Mines, University of Nevada, Reno, Nev.

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and after settling over night the solution was filtered. The filter papers with residue were placed in assay crucibles to which 2 assay tons of PbO had been added, and the filter paper was ignited at a low temperature; silver chloride was added to all but one of the charges, followed by borax glass, soda, flour, and silica. After thorough mixing and covering with borax glass, the charges were fused and poured as usual. The lead buttons were then scorified and cupelled.

Procedure No. 2, "Scorification Assay."

Samples of 0.2 assay ton were mixed with 70 gm. Pb and 5 gm. of borax glass and covered with 35 gm. of Pb in scorifiers and a pinch of silica added. (Silver chloride was added to all but five of these charges.) The charges were scorified as usual. The lead buttons were scorified together until each button represented an assay ton sample; the scorifications being repeated with the addition of test lead and silica until the scorifiers showed only a trace of green color. The final buttons were cupelled.

Procedure No. 3, Fusion with Excess of Litharge.

Samples of 0.5 assay ton were mixed with 250 gm. of PbO, borax glass, and sodium carbonate (a preliminary test showed that it was best to use no niter), and covered with borax glass. Silver chloride was added to all but two of the charges. The charges were fluxed and poured as usual. The lead buttons were first scorified singly; then in pairs to make each represent an assay ton sample and then the scorification repeated till free from copper. This was followed by cupellation.

Procedure No. 4, Fusion with Small Excess of Litharge.

This was performed as in No. 3 except that 90 gm. of PbO was used instead of 250 gm. The first lead button was much smaller than in No. 3, and was too brittle to pound. Lead was added and the buttons scorified twice singly and then in pairs, followed by a single scorification and then by cupellation.

Procedure No. 5, Double Fusion with Excess of Litharge.

The same charge was used as in No. 3 and after pouring, the lead button was hammered and then melted for 10 minutes with a charge consisting of 250 gm. PbO, with a little soda and silica, then poured again. The resulting lead buttons were first scorified together, then singly, and then cupelled.

Treatment of the Beads

The beads were weighed and parted as usual with dilute nitric acid. It was found that the gold from those samples to which no silver had been added contained palladium so that they were inquartated and again parted. The gold beads were annealed and weighed. The silver was precipitated from the solutions with HCl, filtered and washed with hot water. (A test showed that the silver chloride did not occlude more than a trace of palladium). The filtrates were evaporated to dryness with HCl twice on a water bath, cooled, moistened with a couple of drops of HCl and a little water added, then filtered and a little dimethylglyoxime solution (1% dimethylglyoxime in 95% alcohol)

added in the cold with stirring. After settling for about 5 minutes, the precipitates were collected on cut-down ashless filter papers (using cut-down funnels) and washed with cold water. The filter papers and contents were heated, gently at first and then ignited in air and finally reduced by hydrogen in an annealing cup using the clay tube of a rose crucible, but with a nickel cover through which the clay tube passed snugly. In using this arrangement the clay tube was first passed through a hole in the nickel lid, then the hydrogen issuing from it was ignited; and then the end of the tube was put into the annealing cup containing the palladium, the nickel cover fitting over the top; heat was applied to the bottom of the cup for a few minutes and then removed; finally after about 5 minutes the hydrogen was turned off. This permitted the palladium to cool in a reducing atmosphere and thus prevent reoxidation. Several tests showed that if the ignited palladium were not reduced with hydrogen, the results under the conditions used would be about 0.03 mg. too high per mg. of palladium present, on account of oxidation. The ignited palladium was blue on the surface, whereas the reduced palladium was steel gray.

An explanation recently given by R. A. Cooper (Jour. Chem. Met. & Min. South Africa, p. 296, April, 1935), for the separation of palladium from platinum by dimethylglyoxime is worth repeating.

Palladium and platinum both form compounds with dimethylglyoxime that are insoluble in dilute HCl. These compounds are only formed, however, when the elements are in the reduced condition. Palladium is reduced by evaporating to dryness with HCl on a water bath, while platinum is not reduced. Heating to a higher temperature, as on a hot plate, or heating a solution containing dimethylglyoxime will partly reduce platinum and cause it to be partly precipitated by the glyoxime, hence the reasons for the details of the procedure.

Solutions from the beads of 10 - assay ton samples of concentrates were tested for platinum. Only a trace (not a determinable amount) was found by a delicate color reaction.

Of course, since appreciable platinum is absent, the palladium may be precipitated directly as the metal by boiling with formic acid or reduction with magnesium or other metals.

The following table gives results obtained by using the different procedures.

Results Obtained in Assays by the Different Procedures.

SAMPLE No.	Procedure No.	Test No.	Ag. Oz. Per ton	Au. Oz. Per ton	Pd. Oz. Per ton	Remarks
1	1	1	4.01	0.78	1.61	Ag added & weight deducted
1	1	2	4.92	.77	1.65	do.
1	1	3	5.81	.73	.90	No Ag added.
1	2	4	4.83	.75	1.77	Ag added & weight deducted
1	2	5	4.97	.72	1.75	do.
1	2	6	5.55	.79	1.60	No Ag added.
1	3	7	4.62	.73	1.75	Ag added & weight deducted
1	3	8	3.91	.71	1.70	do.
1	3	9	5.30	.73	1.67	No Ag added.
1	4	10	4.95	.75	1.78	Ag added & weight deducted
1	4	11	5.55	.78	1.72	No Ag added.
1	4	12	5.50	.79	1.76	do.
1	5	13	4.53	.74	1.62	Ag added & weight deducted
1	5	14	5.03	.72	1.59	No Ag added
2	1	15	6.68	1.11	2.33	Ag added & weight deducted
2	1	16	7.90	1.13	2.10	No Ag added.
2	2	17	7.80	1.12	2.68	Ag added & weight deducted
2	2	18	9.84	1.14	2.35	No Ag added.
2	3	19	6.42	1.13	2.51	Ag added & weight deducted
3	1	20	4.37	1.48	2.40	do.
3	1	21	5.51	1.50	2.87	do.
3	2	22	3.22	1.52	2.82	do.
3	2	23	5.21	1.49	2.62	No Ag added.
2	--	---	5.7	1.12	2.33	Range of variation
2	--	---	9.6	1.14	3.04	in returns from mine,
3	--	---	4.7	1.48	2.59	refiner, sampler and
3	--	---	5.4	1.63	3.44	umpire.

Results

The results shown in the table indicate the following points:

1. With the sample ground to pass 150 mesh and thoroughly mixed, all the procedures tried give checking results for gold.

2. All the procedures give fair results for silver if no silver be added to the tests, but procedures No. 3 and 5 are inclined to give low results.

3. When silver is added to the test and its weight deducted, the results for silver are erratic and low.

4. Unless silver be added to the tests, the results for palladium are apt to be low.

5. Procedures No. 1 and No. 5 give low results for palladium.

6. Procedure No. 3 gives fair results for palladium when silver is added at the start.

7. Procedures No. 2 and No. 4 give satisfactory results for palladium when silver is added.

#### Conclusions

Procedure No. 4 seems to be the most satisfactory method of those tested for the analysis of the concentrate examined. For the determination of palladium about 10 mg. of silver per mg. of palladium, preferably as the chloride, should be added to the charge at the start. A separate sample to which no silver has been added should be run for silver.

Procedure No. 2 apparently gives satisfactory results but is much more tedious than No. 4. -- Reports of Investigations, Bureau of Mines, Department of Commerce.