

PROCEEDINGS  
OF THE  
CHEMICAL SECTION  
OF THE  
FRANKLIN INSTITUTE.

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[*Stated meeting, held Tuesday, May 17, 1892.*]

HALL OF THE FRANKLIN INSTITUTE,  
PHILADELPHIA, May 17, 1892.

Dr. Wm. H. Wahl, President, in the chair.

A paper on "An Improved Method of Determining Small Percentages of Gold and Silver in Base Metal," by Mr. Cabell Whitehead, of Washington, D. C., was read by Dr. D. K. Tuttle. It was followed by an interesting discussion.

The paper was referred for publication in the *Journal*.

Mr. F. Lynwood Garrison then gave a number of views of the recently conducted armor-plate tests at Indian Head proving ground. Mr. Garrison discussed the results of these tests from the metallurgical standpoint. The complete report of Mr. Garrison to the Institute will appear in the *Journal*.

Adjourned.

WM. C. DAY, *Secretary*.

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AN IMPROVED METHOD OF DETERMINING SMALL  
PERCENTAGES OF SILVER AND GOLD IN BASE  
METALS, MATTES, ETC.

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BY CABELL WHITEHEAD,  
Assayer to the Mint Bureau, Washington, D. C.

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[*Read at the stated meeting held May 17, 1892.*]

Any one having frequent occasion to report the values of silver and gold in such material as crude copper, copper mattes or ores, metallic iron, zinc, etc., will appreciate the advantages of a method which affords accurate results with little expenditure of time. The large production of pure copper by electrolytic methods from silver- and gold-bearing copper ores, renders some such method very desirable in

view of the various transfers of, (1), ores to the smelters; (2), mattes to the refiners; and last, the crude copper to the electrolytic refinery, each of which transfers carries a commercial charge of the bullion contents.

While the proposed method is available for a great variety of base products, it will perhaps best be illustrated by its use in determining the silver and gold contents in crude copper or the mattes resulting from bullion-bearing copper ores. The adaptability of the method to other base metals will readily suggest itself to the experienced assayer.

Given a crude copper or matte carrying from thirty ounces of silver and  $\cdot 10$  of an ounce of gold, upward, the usual method would be scorification. It is usual to take for assay from  $\cdot 05$  to  $\cdot 1$  assay ton to each scorifier, with which are used from forty to fifty grammes of test lead, one-half of which is mixed with the ore in the scorifier and the remainder used as a cover. The scorifier is now introduced into a hot muffle and the door closed until the lead is melted, when it is opened and about one gramme of borax glass placed on the lead. Oxidation begins at once and the lead and base metals are rapidly scorified off until the slag covers the metal, when the assay is poured into a mould. If the lead button still retains much copper, or is hard or brittle, it is again scorified, with the addition of more test lead if necessary. When the lead button is soft and of proper size, it is cupelled and the button weighed and parted. Several scorifications are sometimes necessary before the button is ready for the cupel.

The two objections to this method are, first, the loss of silver during scorification and cupellation, the latter due chiefly to the copper remaining in the lead which it is practically impossible to remove in the scorifier, and which takes silver into the cupel. These losses I found to amount to from 2.33 to 2.78 per cent. of the silver present in the copper. My experiments were made with pure copper and pure silver, the silver being added in the proportion of 100 ounces per ton of alloy.

The second objection is the small amount of bullion which can be operated upon, which necessitates the use of

many scorifiers where small amounts of gold are to be estimated, which is usually the case.

To avoid the doubtful results of scorification processes, the following method has been adopted at the copper works of a firm having large interests at stake:

One assay ton of copper is dissolved in nitric acid, diluted and allowed twenty-four hours to settle. The solution is now filtered from the slight sediment containing the gold. A few drops of hydrochloric acid are added to the filtrate and the solution again allowed twenty-four hours for silver chloride to deposit, when it is filtered, washed and the two filters dried, combined, scorified with test lead and cupelled. The chief objections to this are, (1) the time required; (2) the small amount of precipitate obtained and the danger of loss in scorifying silver chloride; (3) the greater solubility of silver chloride as compared with the bromide.

While the method used at these works is an advance over previous practice, yet the time employed and the uncertainties indicated, leave much to be desired. The protracted time allowed for settling is necessitated by the exceedingly fine condition of the gold resulting from the solution of a metal carrying possibly from .10 to 5 ounces of gold per ton of metal. It would pass through a filter unless allowed to aggregate by standing.

The larger quantity (say 100 ounces) of silver per ton will give but a slowly subsiding cloud (impossible to filter) when newly precipitated as chloride. The scorification of these combined precipitates, one of them being silver chloride, cannot give results quite satisfactory, despite the time involved, some forty-eight hours at least.

The method which I will now briefly describe was devised to meet just such cases, and is suitable for the bullion valuation of zinc, iron, nickel, etc., as well as the sulphides and arsenides of iron and copper. To illustrate the proposed method, which has been found effective and useful, the treatment is detailed for a crude metallic copper carrying both silver and gold. It will be understood that sodium bromide is chosen as a precipitant because of the greater insolubility of silver bromide over the chloride, and also

that the soluble lead salt is added in order that the heavy precipitate of lead bromide may envelop and promptly carry down the silver bromide cloud, which otherwise would require long waiting for its subsidence, as well as to furnish the lead necessary for cupellation.

Weigh out from one to four a. t., depending upon its richness, place it in a beaker of 500 cc. capacity and add gradually enough nitric acid to dissolve it completely, heat until red fumes cease to come off, dilute with water and add fifty grammes of lead acetate, stir, and when dissolved, add one cc. dilute sulphuric acid and allow the lead sulphate to settle. Filter into a 1,000 cc. flask and fill to the mark with distilled water.

The filter contains the gold which has been collected and carried down by the sulphate of lead. The filter paper and precipitate are dried, the paper burned and the ash and lead sulphate scorified with test lead. The button is cupelled and the gold, with any trace of silver it may contain, is weighed. Add silver and part the gold in the usual way. Note the amount, if any, of silver found and include the proper proportion of the amount with that found in the solutions as about to be described.

In order that there may be a control assay, the solution is divided into two equal parts, to each of which a saturated solution of sodium bromide is added with constant stirring as long as a precipitate is produced. The precipitates settle quickly and filter and wash well. Cold water only should be used, and the washing be continued until the washings are free from copper. Any bromide of lead dissolved during the washing, may be neglected, as I have never found it to contain more than a trace of silver, even when assaying ores carrying many thousand ounces.

The precipitate when dry can be brushed from the paper without difficulty, and thus the trouble of burning the filter is avoided. The bromides are now mixed with three times their weight of carbonate of soda and a small amount of flour or other reducing agent, placed in a small crucible, covered with borax glass and melted down in the muffle. The button should weigh about two grammes and be free

from copper and other injurious impurities. This button is cupelled at a low temperature, so that the cupel "feathers" nicely. The time required is from three to five minutes. Duplicate assays usually agree within two-tenths of an ounce per ton.

The following examples are from copper bullion:

	Oz.	Oz.	Oz.
No. 1, . . . . . silver	61.30	61.20	gold .10
No. 2, . . . . . "	63.45	63.30	" .17

Had the gold button contained silver, one-half of its silver contents would have been added to the silver recovered from the solutions, since the gold is estimated on the whole weight, while two determinations of silver are made on the same weight.

The following is an example from a silver-bearing cast iron from the U. S. Mint at Philadelphia. It was dissolved in nitric acid, diluted and precipitated with lead and sodium bromide. After washing, the bromides were dried and brushed from the filter. No. 1 was mixed with ten grammes of litharge, a little flour, the usual amount of soda and covered with borax. Melted down and cupelled, the button gave 30.80 ounces per ton. No. 2 was mixed with test lead and borax and scorified. It gave 29.90 ounces per ton, .90 of an ounce less than No. 1. However, this would be considered a very close agreement by the old method.

The following is an ore from Colorado, very base and with a gangue of sulphate of barium. It proved very unsatisfactory to assay by scorification, duplicates not agreeing at all. The ore was treated with nitric acid and filtered, the residue being assayed separately.

The following are my results:

	No. 1. Oz.	No. 2. Oz.
Residues, . . . . .	32.50	35.95
Solutions, . . . . .	454.90	453.30
As a total, . . . . .	487.40	489.25
Difference of . . . . .	1.80	

Gold was not separated in the residues. The highest assay gotten by scorification was about 467 ounces, which shows twenty ounces in favor of the new method, with a reasonably close agreement in different assays.