

lish blue color. Still another variety can be obtained, by crystallizing from an ammoniacal solution, as a deep brownish yellow substance. All of these forms are anhydrous.

10. The precipitated tellurites are all flocculent and amorphous, and with the exception of the magnesium salt, show practically no tendency to become crystallin.

11. These precipitates are very apt to occlude other salts as impurities, which are very difficult to remove. This tendency is most marked in the barium salt, which will occlude large quantities of barium chloride. In the case of lead tellurite, which was precipitated from a solution of lead nitrate, the precipitate contained nitrates, but these were not so difficult to remove as the chlorides.

12. With the exception of the silver salt, these precipitates are all definit hydrates, which do not readily give up their water.

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## THE VOLUMETRIC DETERMINATION OF GOLD.

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It has been shown<sup>1</sup> that sulfurous acid is capable of reducing auric chloride to aurous in the presence of certain other salts. Through the agency of this reagent it is possible to estimate gold volumetrically easily and accurately.

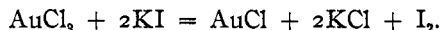
In the following work a sulfurous acid solution was prepared by evolving sulfur dioxide from sodium acid sulfite by means of hydrochloric acid and, after washing the gas, conducting it into repeatedly distilled water. The strength of the sulfurous acid necessarily varied from time to time, due to its gradual oxidation. Its strength varied from 0.005 gram Au to 0.002 gram Au per cubic centimeter. Sulfurous acid solution requires frequent standardization, owing to its ability to slowly oxidize by absorbing oxygen from the air in the container and pass over into sulfuric acid. After a large amount of experimentation under widely differing conditions the idea of using a permanganate solution as a convenient standardizing solution was abandoned. Working under the most favorable conditions, it has not been found possible to obtain results having an error lower than 1% between permanganate and sulfurous acid.

The most reliable method of standardizing sulfurous acid is through the agency of iodine. It is entirely immaterial for the methods to be described whether the sulfurous acid solution is standardized on a known weight of iodine in potassium iodide or whether solid potassium iodide is dissolved in water acidulated with sulfuric or hydrochloric acid and a

<sup>1</sup> THIS JOURNAL, 35, 546 (1913).

definit amount of standard permanganate introduced. As a precaution both of the methods indicated were used continuously and in addition a gold solution whose content had been determined gravimetrically was also used as a standardization check on the sulfurous acid solution.

1. Auric chloride when treated with potassium iodide liberates iodine according to the equation.



The iodine thus liberated can be titrated in presence of excess of potassium iodide by means of standard sulfurous acid. Starch can be used as the indicator.

2. Auric chloride can be treated with excess of a strong solution of magnesium chloride, then with potassium iodide, and the liberated iodine titrated with sulfurous acid.

3. Auric chloride when treated with potassium bromide yields bromine thus:



The liberated bromine can be titrated with a solution of sulfurous acid.

4. Auric chloride treated with magnesium chloride and potassium bromide similarly liberates bromine, which can be titrated with sulfurous acid.

5. Auric chloride when treated with magnesium chloride gives a yellow color, which can be decolorized by titration with sulfurous acid.

6. Auric chloride and sodium chloride give a yellow color which by sulfurous acid can be titrated to the colorless or aurous state.

In order to test the efficiency of these principles auric chloride solutions were prepared by solution of weighed amounts of metallic gold in hydrochloric acid and potassium chlorate and the excess of chlorine removed by the addition of ammonia until the formation of a permanent precipitate, which was then dissolved in hydrochloric acid. Unless precautions of this character are taken the gold solution will either contain some free chlorine on the one hand, or some metallic gold will separate on the other when an attempt to remove the free chlorine by evaporation to dryness or nearly to dryness is made. Evaporation of a pure auric chloride solution, even on the water bath, causes the production of a small amount of aurous chloride which on subsequent dilution gradually changes to metallic gold and auric chloride. By the treatment of metallic gold with hydrochloric acid and potassium chlorate followed by the ammonia and hydrochloric acid treatment, an auric solution can be satisfactorily obtained.

#### Series 1.

Definit amounts of gold chloride prepared in the manner indicated and acid with hydrochloric acid were treated in a dilution of 100 cc. with

about 3 grams of potassium iodide and the iodine liberated was titrated with a standard solution of sulfurous acid with the following results:

No.	Au taken. Gram.	Au found. Gram.	Error.
1	0.0395	0.0395	0.0000
2	0.0439	0.0438	-0.0001
3	0.0878	0.0876	-0.0002
4	0.1098	0.1092	-0.0006
5	0.1107	0.1108	+0.0001
6	0.2289	0.2289	0.0000
7	0.4395	0.4397	+0.0002
8	0.7301	0.7302	+0.0001

### Series 2.

Gold chloride solutions were treated with an excess of a saturated solution of magnesium chloride, potassium iodide was added and the titration by means of sulfurous acid effected in a dilution of 100 cc.

No.	Au taken. Gram.	Au found. Gram.	Error.
9	0.0439	0.0440	+0.0001
10	0.0878	0.0879	+0.0001
11	0.1061	0.1063	+0.0002
12	0.2653	0.2652	-0.0001
13	0.5305	0.5298	-0.0007

### Series 3.

Known amounts of gold chloride solution were treated with excess of a saturated solution of magnesium chloride. The yellow color of the auric chloride is considerably deepened by the addition of magnesium chloride. When sulfurous acid is added from the buret, the solution is bleached progressively until colorless. No other indicator is necessary.

No.	Au taken. Gram.	Au found. Gram.	Error.
14	0.0439	0.0438	-0.0001
15	0.0439	0.0440	+0.0001
16	0.2289	0.2289	0.0000
17	0.2289	0.2289	0.0000

In numbers 14, 15 and 16, 25 cc. portions of auric chloride solution containing a small amount of hydrochloric acid were treated with sufficient excess of concentrated magnesium chloride to bring the volume to 100 cc. and titrated. In No. 17 conditions were the same except that 20 cc. of concentrated hydrochloric acid were present, showing that a considerable amount of free hydrochloric acid does not materially affect this titration of auric chloride in presence of magnesium chloride.

### Series 4.

50 cc. of the slightly acid solution of auric chloride were brought to a volume of 100 cc. with a saturated solution of sodium chloride and titrated to a colorless state by means of sulfurous acid.

No.	Au taken. Gram.	Au found. Gram.	Error.
18	0.0439	0.0438	-0.0001
19	0.2289	0.2289	0.0000

In all of the above titrations a decided excess of alkaline salts was present and at this point the reaction of sulfurous acid was studied on auric chloride solutions containing free hydrochloric acid but no salts. In such a gold solution sulfurous acid not only does not show a distinct bleaching effect but metallic gold is thrown out of the solution with ease.

#### Series 5.

Gold chloride containing a small quantity of hydrochloric acid was brought to a volume of 100 cc., 3 to 5 grams of potassium bromide were added and the red color of the free bromine was titrated to colorless by means of sulfurous acid.

No.	Au taken. Gram.	Au found. Gram.	Error.
20	0.0439	0.0439	0.0000
21	0.0439	0.0440	+0.0001
22	0.0439	0.0438	-0.0001
23	0.0878	0.0876	-0.0002
24	0.2289	0.2289	0.0000

#### Series 6.

Auric chloride solutions were treated with 3-5 grams of potassium bromide and made up to 100 cc. with a strong solution of magnesium chloride. The red color of the free bromine was titrated to colorless by sulfurous acid.

No.	Au taken. Gram.	Au found. Gram.	Error.
25	0.0439	0.0440	+0.0001
26	0.1061	0.1063	+0.0002
27	0.2653	0.2655	+0.0002

In the application of the above methods, the gold is originally usually in the metallic form. In order to dissolve the metal either chlorine water or hydrochloric acid and potassium chlorate are convenient solvents. The excess of the oxidizing agent cannot be removed by evaporation on account of the production of some aurous chloride. The free chlorine can be advantageously removed by addition of ammonia until a permanent precipitate forms, acidulating with hydrochloric acid and heating to redissolve the precipitate formed by the ammonia. On cooling, 3 grams of potassium iodide are added and titration is effected by a standard solution of sulfurous acid, using, if desired, starch as the indicator; or, instead of using potassium iodide, the solution can be treated with potassium bromide or with excess of sodium chloride and titrated by sulfurous acid.