mercuric chloride to 100 g. of the hydrochloric acid solutions, as is shown in the table above, and not to the amounts of hydrochloric acid contained in 100 g. of pure water as interpreted by Ley.

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[CONTRIBUTION FROM THE LABORATORY OF QUALITATIVE ANALYSIS, COLLEGE OF THE CITY OF NEW YORK.]

A STUDY OF THE SILVER ARSENATE TEST FOR ARSENIC.

BY L. J. CURTMAN AND P. DASCHAVSKY.

Received February 18, 1916.

The precipitation of arsenic in the form of an arsenate, by silver nitrate in a neutral solution is so striking that it recommends itself as an excellent confirmatory test. However, the results obtained by students in this laboratory have been so irregular that it was thought worth while to investigate this test for the purpose of ascertaining the conditions under which it could be relied upon to give constant results. It was also the object of the experiments recorded in this paper to determine whether this test is sufficiently sensitive to serve as a confirmatory test for amounts of arsenic from 1–5 mg.

Preparation of Standard Sodium Arsenate Solution.—41.6 g. $Na_2HAsO_4.7H_2O$ were dissolved in water and the volume made up to a liter. 20 cc. portions of this solution were run out from a buret and the arsenic determined gravimetrically by the method given by Treadwell.[1] The results of well-agreeing determinations gave the solution a value of 10 mg. of As per cc. By suitable dilution in volumetric flasks, solutions of lower concentrations were obtained.

Determination of the Sensitiveness of the Test.—By means of graduated pipets, definite amounts of the standard arsenate solution were introduced into a series of test tubes. 2 cc. of a silver nitrate solution of strength 50 mg. Ag per cc. were then added. After the addition of one drop of a 1% solution of phenolphthalein, the solutions were made alkaline with dilute NH_4OH and finally made just acid with 5% acetic acid. After standing for several minutes the tubes were examined and the results compared with a blank run under the same conditions. The following results were obtained:

TABLE I.

No.	As (mg.).	Result.
1	10.00	Heavy precipitate
2	5.00	Heavy precipitate
3	1.00	Small precipitate
4	0.50	Small precipitate
5	0.10	Faint precipitate
6–7	0.05	Faint precipitate on standing
8–10	0.02	Brown coloration. Limit
11–12	0.01	Negative

The above results show that the limit of the test (0.02 mg. in a volume of 3 cc.) represents a concentration of 1 part in 150,000. While this limit-

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[1] Treadwell, "Quantitative Analysis," translated by H. T. Hall (1904), p. 165.

ing amount of arsenic can be readily recognized when compared with a blank, decided and unmistakable results are obtained with 0.05 mg. which for routine qualitative work is the practical limit of the test. By the use of an indicator and a 5% solution of acetic acid, added drop by drop, a practically neutral solution is readily obtained.

Influence of Ammonium Salts.—In the above experiments care was taken to avoid the addition of an excess of NH_4OH. In systematic analysis the arsenate is formed by the solution of the sulfide in an excess of nitric acid and the test applied after the free acid has been neutralized by ammonia. This procedure results in the formation of ammonium salts which, it was thought, might diminish the sensitiveness of the test and thus account for the failure of students to detect from 1 to 5 mg. of arsenic. To ascertain whether or not ammonium nitrate exercises any influence on the test, the following experiments were made. The procedure was the same as that already described. The results are given in Table II.

TABLE II.

No.	As (mg.).	NH_4NO_3., g.	Total Vol., cc.	Test.
1–6	1.0	0.0–5.0	8.0	Good
7–12	0.2	0.0–5.0	9.0	Good

The above results show that 5 g. of NH_4NO_3 do not interfere with the detection of small amounts of arsenic by this test.

Sensitiveness of the Test in Systematic Analysis.—To determine whether small amounts of arsenic could be detected by this test in systematic analysis, the following test analyses were made. The procedure was as follows: The solution, the volume of which was about 20 cc., was neutralized with NH_4OH, 2.5 cc. conc. HCl added, heated to boiling and treated with H_2S. The volume was then made up to 100 cc. and again treated with H_2S till precipitation was complete. The precipitate was collected on a filter, transferred to a beaker, heated for 5 minutes with 10 cc. conc. HCl, diluted and filtered. The residue, after washing free from chlorides with hot water, was transferred together with the filter to an evaporating dish, boiled with 2 cc. conc. HNO_3 till no more fumes of the oxides of nitrogen were given off, diluted and the mixture filtered on a small filter. The latter was then washed five times with water,

TEST ANALYSES.

No.	As (mg.).	Sb (mg.).	Sn (mg.).	Result.
1	5	...	...	Reddish ppt.
2	5	5	5	Reddish ppt.
3	5	500	...	Small reddish ppt.
4	5	...	500	Small reddish ppt.
5	5	250	250	Small reddish ppt.
6	1	250	...	Small reddish ppt.
7	0.5	250	...	Reddish cloudiness
8	0.0	250	...	Pale yellow coloration

the combined filtrate and washings concentrated to 2 cc. and transferred to a test tube. The test was then carried out in the manner already described in the determination of the sensitiveness of the test. The preceding results were obtained.

The preceding results show that the silver arsenate test is capable of detecting as little as 0.5 mg. of arsenic in systematic analysis.

Summary.

1. With pure solutions of arsenate the test with silver nitrate is sensitive to 0.02 mg. of arsenic.

2. Ammonium nitrate, which is generally formed in carrying out this test, exercises no influence on the detection of 0.2 mg. of arsenic as sodium arsenate.

3. A procedure is given by which, in systematic analysis, the test is capable of detecting 0.5 mg. of arsenic with certainty.

New York, N. Y.

THE DETERMINATION OF ALUMINIUM AS OXIDE.

By William Blum.[1]

Received May 10, 1916.

I. Introduction.

Although a considerable number of precipitants have been proposed for the determination of aluminium, direct precipitation of aluminium hydroxide by means of ammonium hydroxide, followed by ignition to oxide, is most commonly used, especially if no separation from iron is desired, in which case special methods must be employed. While the general principles involved in this determination are extremely simple, it has long been recognized that certain precautions in the precipitation, washing, and ignition, are necessary if accurate results are to be obtained. While, however, most of these details have been studied and discussed by numerous authors, it is noteworthy that few publications or text books have taken account of all the factors. In the present paper it seems

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[1] Published by permission of the Director of the Bureau of Standards. This paper will appear in somewhat more detailed form as Scientific Paper No. 286, copies of which can be obtained upon request addressed to the Bureau of Standards, Washington, D. C.