the earlier authorities, or for heating it as recommended by Harkins,[1] by passing through it either cold or hot water.

In the preparation of standard mirrors for comparison, as recommended by Sanger,[2] as small a quantity as 0.005 mg. As_2O_3 may be standardized by means of this apparatus. The mirrors prepared vary distinctly with a variation of 0.005 mg. of As_2O_3 in the known solutions used and duplicate mirrors from equal quantities of As_2O_3 agree quite as well as those illustrated in Sanger's paper.

GEORGE WASHINGTON UNIVERSITY, WASHINGTON, D. C.

A COLORIMETRIC DETERMINATION OF MANGANESE IN THE PRESENCE OF IRON.

BY M. R. SCHMIDT.

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The following method has been used by the writer for some time, and has been found to give uniform satisfaction in the determination of small quantities of manganese in the presence of iron, in certain pharmaceutical preparations, such as peptonates, sirup of hypophosphites, etc. No originality can be claimed for the method, as it was proposed several years ago by Walter,[3] but it is believed that the details of manipulation here given will lead to the greatest rapidity in attaining accurate results The usual basic acetate method of separating manganese from iron requires, in order to effect complete separation, at least two precipitations of the iron, and subsequent evaporation of the filtrates in order to insure complete precipitation of the manganese by bromine or other reagents. This involves considerable loss of time, and when dealing with small quantities of manganese, the total experimental errors often amount to a considerable percentage of the weight of the manganese involved. The present method requires no previous removal of iron, and the results are accurate when the total manganese amounts only to a few milligrams Moreover, duplicate determinations can be made in one-half hour. The determination depends on the power of the persulphates to convert bivalent manganese into permanganic acid in the presence of silver nitrate.

A standard solution of manganese containing 2 mg. of manganese in each 10 cc. is found to be most convenient, and is prepared as follows: Run 182.1 cc. of exactly tenth-normal potassium permanganate solution into a liter flask, add about 100 cc. of 10 per cent. sulphuric acid and 12 cc. of 3 per cent. hydrogen peroxide. Warm the flask slightly until the oxygen is driven off, cool again and dilute to the mark. Prepare also a

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[1] THIS JOURNAL, **32**, 520 (1910).

[2] *Loc. cit.*

[3] *Chem. News*, **84**, 239 (1901).

solution of silver nitrate, containing 20 grams of the salt in a liter. 1 cc. of this solution must be used with each mg. of manganese present.

To make a determination, remove 5 cc. of the unknown solution to a small beaker by a pipette. Dilute to about 100 cc., add 2 or 3 cc. of ether, and precipitate with ammonium sulphide. Warm the solution on the steam bath, stirring vigorously, and after the precipitate is settled filter through a small folded filter and wash thoroughly with water containing a little ammonium sulphide. Dissolve the precipitate on the filter with the smallest possible quantity of dilute sulphuric acid, collecting the filtrate and washings in a small Erlenmeyer flask. Boil to expel hydrogen sulphide, add a little nitric acid to oxidize the iron and then add one drop of silver nitrate solution. If a turbidity occurs, which indicates imperfect washing of the sulphides, add silver nitrate in very slight excess, boil again, and filter through a small dense filter into a 100 cc. flask. *To this solution, which should be perfectly clear, add 1 cc.* of silver nitrate solution for each mg. of manganese which is present, followed by about 1 gram of ammonium persulphate, and place the flask on a steam bath. The contents soon turn yellow, then red, and in about one minute the purple color of permanganic acid is fully developed. Cool the flask, fill to the mark, stopper and shake well. Into another 100 cc. flask, introduce 10 cc. of the standard manganese solution, and about 20 cc. of dilute sulphuric acid. Add 2 cc. of the silver nitrate solution and 1 gram of ammonium persulphate, and proceed exactly as with the unknown solution. Fill a burette with the latter, and fill one of two comparison tubes, which must be of the same diameter, with a portion of the standard permanganic acid solution. Fill another burette with distilled water, run about 10 cc. of the unknown solution into the second comparison tube, and dilute with water until the color is equal in intensity to that of the standard solution, viewing the tubes from the side. Suppose, for instance, that 12 cc. of the unknown permanganic acid solution were taken and 18 cc. of water added. This solution, of the same intensity as the standard solution, is of the same strength, and contains 0.02 mg. in 1 cc. or, $(12 + 18) \times 0.02 = 0.60$ mg. in all. This is equivalent to 12 cc. of the original solution, which therefore contains 5 mg. manganese, derived from 5 cc. of the original preparation, giving a percentage of 0.10.

It is found at times that the oxidation of the manganese is only partial, and the solution, instead of becoming purple, is clouded by a brown precipitate of manganese dioxide. In such cases, add a few mg. of sodium sulphite or bisulphite, or a few drops of sulphurous acid solution, until the solution becomes clear again. Then add more silver solution, and another portion of ammonium persulphate and warm again.

The ether used when precipitating the sulphides has the effect of causing the precipitate to settle quite rapidly.

Following are results obtained in duplicate determinations of several preparations. In all of these preparations iron was present in amounts from 3–5 times the amount of manganese.

	1 cc. contains milligram Mn.
1	0.205
	0.243
2	0.1468
	0.1520
3	0. 734
	0. 723
4	0. 588
	0. 606

In order to test the accuracy of the method, 0.6316 gram of crystallized manganous sulphate, free from effloresced portions, was dissolved in 250 cc. of water, 10 cc. of this were taken, and after oxidation diluted to 200 cc.

On comparing with the usual standard, the 200 cc. were found to contain 6.392 mg. of manganese, corresponding to 0.6505 gram of manganous sulphate originally, or 102.9 per cent. To another 10 cc. of the original solution, 1 gram of iron alum was added and the manganese determined as before. In this case 6.178 mg. of manganese were indicated in the 10 cc., corresponding to 99.2 per cent. of theory. Here the concentration of the iron was roughly 20 times that of the manganese.

The method has already been applied to the determination of manganese in rocks,[1] and should prove excellent for determining manganese in waters.

LABORATORY OF SHARP & DOHME.

NOTES.

The Chemical Laboratory of the University of Washington.—The University of Washington on April 16, 1910, formally opened for use a new chemical laboratory which cost when completed a total of $230,000. This building is of concrete and steel construction and is three stories in height. The design of the building was made by the architects of the Alaska-Yukon-Pacific Exposition according to drafts prepared by the writer after his visit to all the modern laboratories of Europe and America. It contains certain features of construction which may be of interest to those chemists who are contemplating erection of buildings or equipment of laboratories. A drawing of the general floor plan of the building accompanies this note.

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[1] Hillebrand, *Bull.* **305**, U. S. G. S., p. 99 (1907).