3. The original solution shows the presence of 0.01326 gram of aluminum as Al_2O_3 per cc.

The original solution, freed from lead by means of H_2S, shows the presence of 0.01296 gram aluminum as Al_2O_3 per cc, thus indicating that the results are practically identical whether the aluminum is determined in the presence or absence of lead.

4. The amount of aluminum as Al_2O_3 found —0.01326 gram per cc in the presence of lead and 0.01298 gram per cc in the solution freed from lead by H_2S—proves that the NATIONAL FORMULARY standard, "each milliliter of solution of aluminum acetate corresponds to not less than 0.01126 Gm. nor more than 0.01376 Gm. of aluminum oxide (Al_2O_3)," is correct.

RESEARCH AND ANALYTICAL LABORATORIES
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THE DETERMINATION OF TOTAL IRON IN PHARMACEUTICAL PREPARATIONS.

BY J. FORBES WILLIAMS.

There are numerous official syrups, elixirs, tinctures and pills which contain iron in various forms. In most of these preparations no assay is given for the iron determination. Besides the official preparations there are hundreds of tonics on the market, the majority of them containing iron in some form. An effort has been made to establish an assay which gives accurate results in preparations of this class, but which can be performed in a reasonable length of time.

A short résumé of the methods tried and in some degree found unsatisfactory, might be given:—Those depending upon the quantitative liberation of I from KI in the presence of HCl, can be used only when the iron is in the ferric state. Those depending upon the precipitation as $Fe(OH)_3$ are not quantitative when sugar or certain organic acids are present. Those by igniting the sample, taking up in acid and precipitating as $Fe(OH)_3$; the residue after ignition, in the cases where sugar and sodium hypophosphite were present, was only dissolved with difficulty in acids. That, by precipitating as the sulphide, dissolving in HCl, oxidizing with HNO_3 and precipitating as $Fe(OH)_3$, igniting and finally weighing as Fe_2O_3, in nearly all determinations gave accurate results, but required about three hours to run, and when manganese was present in considerable quantity, gave high results.

The results in the last-mentioned method having been for the most part satisfactory, some modification whereby it could be used in the presence of manganese and by which the time required would be reduced, was sought.

By dissolving the FeS in dilute H_2SO_4 and titrating with $N/10$ $KMnO_4$, it was found that Mn did not interfere, besides eliminating a drying, ignition and weighing.

An outline of the assay finally decided upon is given.

To 10 cc of the sample in a 125-cc beaker, add 10 cc. H_2O, 5 cc HNO_3, and boil for 5 minutes. Add 5 cc of 10% NH_4Cl solution, then add NH_4OH until neutral or only slightly acid. Cool and run in H_2S for 1 minute; add excess of NH_4OH and continue to pass in H_2S until com-

pletely precipitated. Filter through a small Büchner funnel connected as in Fig. 1 and previously prepared by fitting in a filter paper and overlaying with a thin layer of asbestos filter media. Wash the precipitate with three 15-cc portions of H_2O containing 1% NH_4S and 1% NH_4Cl. Disconnect flask, discard filtrate, rinse flask and reconnect to funnel. Pass five 15-cc portions of hot 10% H_2SO_4 through the funnel, disconnect and boil filtrate until free from H_2S (shown by the vapors not darkening a piece of filter paper moistened with lead acetate solution). Cool and titrate with $N/10$ $KMnO_4$.

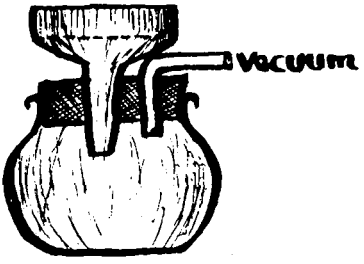

Fig. 1.

A number of preparations were assayed giving results that checked well, but as no other good checking method was available and as the actual iron content was not known, it was thought advisable to repeat the experiments using preparations made by dissolving exact amounts of iron of known purity in the required acids, etc., and then adding the other ingredients.

For the sake of convenience in tabulating and comparing, the iron content in all the liquids was made at one percent instead of the percentage required by formulas.

RESULTS OBTAINED.

	Percent actual.	Percent found.
Solution of Ferric Hypophosphite	1.000	1.005
	1.000	1.005
Elixir Ferric Phosphate	1.000	1.010
	1.000	1.005
Syrup Iron and Manganese Iodide	1.000	1.016
	1.000	1.010
Beef, Iron and Wine	1.000	0.980
	1.000	0.988
Pills of Ferrous Iodide (1 pill)	0.0400 Gm.	0.0405 Gm.
	0.0400 Gm.	0.0410 Gm.

SUMMARY.

An assay has been outlined for the determination of iron in pharmaceutical preparations, which in a number of analyses gave an error not exceeding 0.002 gram.

The time required by the writer, to complete one analysis, was 25 minutes, six analyses having been completed within an hour.

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THE ASSAY OF SALICYLIC ACID AND OFFICIAL SALICYLATES.*

BY ABRAHAM TAUB AND HARRY TAUB.

The present U. S. P. assay of the alkali salts of organic acids having in certain cases proved unsatisfactory, as reported from many authoritative sources, the authors undertook, at the suggestion of Dr. H. V. Arny, Chairman of the Sub-Committee on Inorganic Chemicals, U. S. P. Revision Committee, a study of the

* Read before New York State Pharmaceutical Association meeting, 1922.