0.05 molar iodate while according to (5) it would require 40 cc. The results were as follows:

Conc. of HCl (% by vol.)	26	31	36	40	60	65	70	90
Cc. 0.05 *M* KIO_3	26.2	28.2	29.6	32.2	40.4	39.9	40.2	40.0

The above results show, as with the cadmium, that with maximum oxidation only one-third of the sulfur is oxidized to sulfuric acid; they do not show the minimum found with cadmium, as the insolubility of antimony sulfide makes it inconvenient to work in the less concentrated solution.

(*e*) **Arsenic.**—As L. W. Andrews explicitly states, with higher concentrations of hydrochloric acid no oxidation of arsenic takes place. We would naturally predict then that only the sulfur from the arsenious sulfide would be oxidized, thus:

$$As_2S_3 + 3KIO_3 + 12HCl = 2AsCl_3 + 2S + H_2SO_4 + 3ICl + 3KCl + 5H_2O \quad (6)$$

The results with arsenious sulfide showed, however, only $^2/_3$ of the iodate called for by this equation; no satisfactory explanation has been found for this result.

10 cc. of an 0.08 *M* solution of arsenious chloride was precipitated as sulfide and titrated with 0.05 *M* iodate with about 80% concentrated acid. The iodate required according to Equation 6 would be 24.0 cc. The iodate consumed was 16.0, 16.0, 16.3, 16.0, respectively. Whether this apparently exact ratio of 2 iodate to 1 arsenic sulfide is accidental or not we have not determined, because the insolubility of the sulfide limits the applicable concentration to practically pure acid.

This work was done under the direction of Prof. U. H. Gottschalk.

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THE DETERMINATION OF BORON IN IRON.

By J. M. Lindgren.

Received March 12, 1915.

The present work has had to deal with the determination of small percentages of boron in iron. Two procedures have been tried. First, the well-known method of Gooch,[1] which consists in the formation of methyl borate and distillation of the ester in a properly arranged apparatus, with recovery of the boron in the form of calcium borate. The complications involved in this method are accentuated by reason of the large quantity of iron oxide and the fact that distillation must be repeated at least six times.

The other method made use of is that proposed by Wherry[2] which involves titration with alkali of the boric acid in the filtrate obtained

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[1] *Am. Chem. J.*, **9**, 23 (1887).

[2] This Journal, **30**, 1687 (1908).

after precipitating the iron. The application of this method to the present problem made it necessary to determine whether it is possible to recover the relatively small amounts of boric acid in the presence of large amounts of ferric hydroxide. After considerable experimentation this was found to be entirely feasible without departing in any marked degree from the principles involved in the method as set forth by Wherry. The details of the procedure are as follows:

Two to three grams of iron are dissolved in a round-bottomed Jena flask of about 800 cc. capacity, using 10 cc. each of nitric acid (sp. gr. 1.4), hydrochloric acid (sp. gr. 1.2), and water. When the action is completed the solution is cooled to room temperature and the iron precipitated by use of C. P. calcium carbonate. If not properly carried out this precipitation may result in the formation of colloidal hydrate. It will be noted that the volume of the solution is approximately 30 cc. If to this solution there is added, all at one time, approximately double the amount of dry calcium carbonate necessary for complete neutralization, and the mixture is stirred by vigorous agitation, a pasty or solid mass results, which, upon the addition of hot water, yields ferric hydroxide in the form of a dark brown and very granular precipitate. Two hundred and fifty to three hundred cc. of carbon-dioxide-free water are added and the mixture is boiled under a reflux condenser for at least 30 minutes to assure entire absence of carbon dioxide or bicarbonate.

Before filtering, and in order to still further open up the precipitate to admit of thorough washing, there is now added a considerable quantity of asbestos fiber which has been well washed. A good method for preparing the asbestos is to digest over night on the steam bath a considerable quantity in dilute hydrochloric acid. This should be well washed and suspended in boiling water. The fiber is added wet to the precipitated iron, the amount needed in each process being approximately 50 cc. in bulk, sufficient to thoroughly impregnate the precipitated iron. After boiling the mixture for a few minutes the precipitate is filtered at once by suction through a Büchner funnel, about 15 cm. in diameter, having a double thickness of filter paper carefully fitted over the bottom. A splash trap is connected to the receiving flask. The precipitate should be washed 8 or 10 times with boiling hot water. The funnel is then removed and replaced by a solid rubber stopper. By renewal of the suction, further ebullition is produced in the flask without addition of more heat. The solution is then cooled and titrated as follows with 0.1 *N* alkali (free from carbonate). After addition of a few drops of phenolphthalein, alkali is added until a pink color appears. One gram of mannite is next added, and the titration continued until a permanent pink color is obtained.[1] Familiarity with the end point is attained by practice. A blank should

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[1] For details of the reactions see THIS JOURNAL, 30, 1699 (1908).

be carried through using pure iron and following the process exactly as described.

In testing the accuracy of the method, 0.1 *N* solution of boron was prepared by dissolving 3.5 g. of B_2O_3 in one liter of CO_2-free water. The B_2O_3 was prepared by fusing pure boric acid in a platinum dish. When titrated in the presence of mannite, 8.68 cc. of this B_2O_3 solution required 8.75 cc. 0.1 *N* KOH. Known quantities of this boric acid solution were added to a solution of iron free from boron and the operations were carried out as indicated. Contrary to the experience of Wherry, when the precipitation by use of calcium carbonate was properly carried out, it was found possible to wash the iron entirely free from boric acid and recover the full amount in the filtrate. The use of the boric acid solution made up of the pure fused material as above described also served admirably as a medium for standardizing the alkali used in the titrations.

TABLE I.—DETERMINATION OF BORON IN IRON.

% Boron taken	0.038	0.188	0.424	0.850
% Boron found	0.044	0.194	0.388	0.840

URBANA, ILL.

CHEMICAL REACTIONS AT LOW PRESSURES.[1]

BY IRVING LANGMUIR.

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In the course of investigations into the causes of the blackening of tungsten lamps,[2] the effects produced by the introduction of low pressures of various gases have been studied in considerable detail.

It had been previously known[3] that the vacuum in a lighted tungsten lamp normally improves during the life of the lamp, but it was thought that this removal of the residual gases was brought about by electrical discharges in much the same way as that commonly observed in Geissler and Roentgen ray tubes. These experiments have shown, however, that a highly heated tungsten filament will cause the disappearance or clean-up of nearly any gas introduced into the bulb at low pressure, and that this action, in the great majority of cases, is purely chemical in nature.

The experimental methods that have been employed in these investigations are relatively simple. A bulb containing one or more short filaments, usually of tungsten, was sealed to an apparatus consisting essentially of a mercury Gaede pump, a sensitive McLeod gage for reading the pressures, and an apparatus for introducing small quantities of various

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[1] Paper presented before the New York Section of the American Chemical Society, March 5, 1915.

[2] *Trans. A. I. E. E.*, **32**, 1913 (1913).

[3] THIS JOURNAL, **35**, 107 (1913).