



FIG. 3.

A group of the stoves, independently connected, is highly preferable to the large cumbersome water- or steam-baths generally employed.

THE ESTIMATION OF PHOSPHORUS IN STEEL.

BY R. W. MAHON.

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MANY analysts regard the titration of ammonium phosphomolybdate by standard alkali and acid with phenol phthalain as indicator as the best method for the routine determination of phosphorus in steel.

By two modifications of one of the approved processes for obtaining phosphorus in steel as ammonium phosphomolybdate, I have so shortened the operations as to enable one to execute the analysis in eight minutes, from receiving the drillings to obtaining the result. The results are accurate in the absence of arsenic. I have not experimented with the process in the presence of arsenic. No experiments have been made with steels containing silicon or carbon in considerable quantity. The process was devised for facile determination of phosphorus in steel containing scarcely any silicon, and low in carbon. It appears entirely probable that with high carbon and silicon, perfectly accurate results would be obtained. The two modifications I have to suggest are (1) effecting the partial neutralization in a different manner, and (2) precipitating at a higher temperature. Before the steel drillings are received, the following preparations for the analysis are made: Seventy cc. of water and thirty cc. of nitric acid (sp. gr. 1.4) are poured into a sixteen-ounce Erlenmeyer flask, a measured quantity of standard caustic potash solution and a little water are poured into a small beaker, the reading of the acid burette is recorded, and the filter is placed in its funnel, at the suction-pump, ready for use.

Four grams of drillings are placed in the flask containing the

acid, and this immediately placed over the gas. As soon as dissolved add three cc. of a standard solution of potassium permanganate, and boil until no longer pink. Add ten cc. of hydrochloric acid (sp. gr. 1.20), boil until clear, remove from gas, wait for a few seconds until the boiling has nearly ceased, and then add a mixture of fifty cc. of molybdate solution and ten cc. to fifteen cc. of ammonia (sp. gr. 0.90), made ready just before use. Pour into the middle of the flask, not down the side. Twirl the flask in both directions, successively, without delay. Shake the open flask for about one quarter of a minute. Filter and wash with cold water. Place the paper and precipitate in the beaker, containing an excess of caustic potash solution, add phenol phtalein, and determine the excess of caustic by the standard acid.

The two standard solutions are of the customary strength; the molybdate solution is made according to the following formula: 100 grams of molybdic acid are dissolved in a mixture of 200 cc. of ammonia (sp. gr. 0.90) and 200 cc. of water, and the solution filtered into 1,250 cc. of nitric acid (sp. gr. 1.20), blowing air through the mixture. Fifteen cc. of ammonia (sp. gr. 0.90) will neutralize fifty cc. of this solution, hence the amount of ammonia recommended in making up the mixture for use in an analysis. Ten to fifteen cc. of ammonia can be added, and if the mixture is kept cool during mixing no separation of molybdic acid will occur for some undetermined, but considerable time. This cold mixture can be satisfactorily used. It is, however, unnecessary to take the precaution of cooling during the mixing of molybdate solution and ammonia, if the mixture is used as directed. It is to be noted here that the graduated or other vessel used for mixing should invariably be rinsed before being used for a second quantity, as neglect of this sometimes causes separation of molybdic acid. The object of adding the mixture by pouring it into the middle of the solution and at once shaking the flask is to prevent separation of molybdic acid. This is the more elegant way of working, but is not indispensable, as the molybdic acid precipitated by very solid addition of the mixture, not poured accurately into the center, at once goes into solution again, when the prescribed course of manipulation is followed.

The temperature of the solution at the moment of addition of molybdate is about 105°C . The temperature of the mixture added is about 60°C . The amount of free acid before the molybdate is added is such that eighteen to twenty cc. of ammonia (sp. gr. 0.90) would, if added, produce a decided precipitate. A standard steel in which phosphorus had been determined many times with elaborate care by the molybdate-magnesia method, as 0.108, was used to make two series of estimations, titrating ammonia phosphomolybdate by standard caustic solution, one (A) with the older method of precipitating—that is adding ammonia in excess to the cooled solution of the steel, acidifying with nitric acid, and bring to 75°C . before adding molybdate solution; the other (B) with the method of precipitating described above—that is mixing molybdate solution and ammonia, in definite quantities, and precipitating a few seconds after removal from lamp.

Four estimations vary- ing from 0.107 to 0.110.	Twelve estimations vary- ing from 0.108 to 0.111.
(A).	(B).
0.108	0.108
0.107	0.111
0.110	0.109
0.107	0.111
—	0.109
Mean, 0.108	0.109
	0.110
	0.109
	0.110
	0.109
	0.109
	0.111
	—
	Mean, 0.110

In the series (B) above, the determinations were made to study the value of this method of precipitation, not to prove the time needed, a number of the estimations having been made simultaneously; consequently the filtration was only made immediately in some of the number.

In the following the time needed was the principal object of study, and each filtration was made a few seconds after adding molybdate :

Time consumed in analysis.	Phosphorus. Per cent.
Eleven minutes	0.108
Ten minutes	0.107
Eight and a half minutes.....	0.107

I have also carried out one, adding the potassium permanganate before the last few particles of steel had dissolved, and only boiling until the pink color nearly disappeared.

Time consumed in analysis.	Phosphorus. Per cent.
Seven minutes	0.109

Although successful here, this last procedure might fail with high carbon steel. All of the foregoing filtrates remained perfectly clear. The quantity of ammonia recommended is a vital point.

ESTIMATIONS BY METHOD B VARYING THE QUANTITY OF AMMONIA.

	Phosphorus. Per cent.
Five cc. of ammonia	0.096
Twenty cc. of ammonia.....	0.113

ESTIMATION BY METHOD B WITH VARIATION IN THE TEMPERATURE OF PRECIPITATION.

75° C., 0.110 per cent. phosphorus filtered in one minute.
65° C., 0.103 " " " " " " " hour.
35° C., 0.108 " " " " " " twenty-four hours.

This method of working is now in daily use, having been repeatedly checked by the older style, invariably with entire satisfaction. Used as a rapid method and with the details of manipulation described, the results are identical with those by (A). My principal use of it has been simply to lessen the number of operations needed; used in this way the drillings are placed in an empty flask, and the acid and water poured upon them; the filtration not necessarily being made after a fraction of one minute, nor with the suction-pump at all, but with the use of funnel and platinum cone, and filtering after the lapse of an indefinite time, varying from a few minutes to hours. Used thus, the results are 0.002 per cent. phosphorus higher.

The caustic solution is empirically standardized in these experiments by precipitates made in the older way (A); hence these appear as correct, and those by (B) as 0.002 per cent. phosphorus high. Were (B) precipitates used in standardization the record would make the results by (B) appear as correct, while those by (A) would average 0.106.