

Bismuth solution taken. cc.	Potassium perman- ganate required. cc.	Weight of bismuth found. Gram.	Weight of bismuth taken. Gram.
15	26.95	0.1207	0.12056
15	26.9	0.1205	0.12056
20	35.7	0.1599	0.16074
20	36.15	0.1619	0.16074
25	45.35	0.2031	0.20092
25	45.25	0.2027	0.20092

This shows that the results obtained by the gravimetric method are as accurate as those found by the volumetric procedure.

CONCLUSIONS.

(1) To determine bismuth by the evaporation of a nitric acid solution of bismuth nitrate the operation must be conducted in porcelain, otherwise some bismuth trioxide is reduced.

(2) In the precipitation of ammonium bismuth molybdate the use of congo red is preferable to methyl orange, and in washing the precipitate ammonium nitrate is better than ammonium sulphate.

(3) Bismuth may be determined correctly by the ignition of bismuth ammonium molybdate to $\text{Bi}_2\text{O}_3 \cdot 4\text{MoO}_3$ when the temperature of ignition is kept below a dull red heat.

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COLORIMETRIC DETERMINATION OF PHOSPHORUS.

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THE color given on passing hydrogen sulphide into an alkaline molybdate solution seemed favorable for a colorimetric determination of molybdenum, and indirectly of phosphorus. The following method has been developed on this basis:

Standard Molybdic Acid Solution.—One cc. = 0.004108 gram MoO_3 , equivalent to 0.000073 gram phosphorus.

Standard Phosphomolybdate Solution.—One cc. = 0.000006+ gram of phosphorus, 5 cc. = 0.419 cc. of the standard molybdic acid solution; prepared by dissolving ammonium phosphomolybdate in about the theoretical amount of sodium hydroxide.

In the following experiments 12-ounce bottles, 2 inches square, were used. The standard solution was placed in the bottle filled

half full with water and hydrogen sulphide was passed until the solution was saturated, then the bottle was filled to the neck.

It was found, after numerous experiments, using different quantities of molybdic acid solution and equivalent quantities of the phosphomolybdate solution, that a darker color was obtained with phosphomolybdate, thus making it necessary to use a standard comparison solution of phosphomolybdate. Equal quantities of phosphomolybdate always give the same color.

Sodium hydroxide in reasonable excess of the amount necessary to dissolve a given precipitate is not detrimental to the results. A blackish solution will be given, if too little sodium hydroxide is used.

The solution must be saturated with hydrogen sulphide, as too little will give a light colored solution. The right color, after treating with hydrogen sulphide, is yellowish red. Saturation can be obtained by passing the gas at a moderate rate for five minutes.

The solutions when treated are not affected by air, but gradually darken on standing as the reactions involved progress. By heating the sulphuretted solution in boiling water for five minutes the color assumes a shade permanent for two hours.

Using Nessler tubes of 16 mm. bore and a depth of liquid of 24.5 cc. it was possible to detect a difference of 0.00000089 gram phosphorus.

METHOD.

Weigh off 2 grams of pig iron, or steel, or the substance to be tested, dissolve and obtain the precipitate of phosphomolybdate as in any other volumetric determination of phosphorus. Collect the precipitate on a 9 cm. filter and wash with 2 per cent. nitric acid. Place the funnel with the precipitate in the neck of a 100 cc. measuring flask, churn with hot water and add N/10 NaOH until the precipitate is dissolved. Do not use stronger sodium hydroxide, as there is danger of too large an excess, since there is only a small quantity of phosphomolybdate. Add the hot water first to protect the filter. If the filter is broken, shreds will pass into the solution and interfere with the reading. Read the burette containing the N/10 NaOH and add one-half of the amount of sodium hydroxide necessary to dissolve in excess. Fill to the mark. Take an aliquot part and place in a 50 cc. Nessler tube, fill half full with water and pass hydrogen sulphide

at a moderate rate for five minutes, then place into boiling water and let stand for five minutes; remove, fill to the mark and compare with the standard, which is made by placing 10 cc. of the standard phosphomolybdate solution in a Nessler tube, filling half full with water and passing hydrogen sulphide for five minutes, heating for five minutes in boiling water and then filling to the mark. Compare the colors by looking down through the tubes, having light reflected upwards.

Standard Phosphomolybdate Solution.—The yellow precipitate was obtained in bulk by precipitating sodium phosphate with ammonium molybdate, thoroughly washing the precipitate with 2 per cent. nitric acid and drying in a steam oven. Take 0.2737 gram of yellow precipitate, place in a 500 cc. measuring flask and add just enough N/10 NaOH for solution, then add one-half of the amount necessary for solution, in excess. Make up to 500 cc. Ten cc. = 0.00009122 gram of phosphorus. If the standard phosphomolybdate solution is stronger than here given, 10 cc. will give a color too dark for comparison. N/10 NaOH is made by dissolving 4 grams of pure sodium hydroxide and making up to 1000 cc. Its strength need not be determined further.

The following are phosphorus results obtained in pig iron and steel which Mr. Camp, of Carnegie Steel Co., and Mr. Glass, of LaBelle Steel Co., kindly furnished me:

	Phosphorus by titration with standard NaOH. Per cent.	Phosphorus by above method. Per cent.
Low phosphorus metal, 1.....	0.044	0.044
“ “ “ 2.....	0.044	0.044+
“ “ “ 3.....	0.044	0.044=
“ “ “ 4.....	0.044	0.044+
“ “ “ 5.....	0.044	0.044—
Bessemer pig iron, 1.....	0.081	0.084
“ “ “ 2.....	0.081	0.086
“ “ “ 3.....	0.081	0.083
Bessemer pig iron, 1.....	0.102	0.087
“ “ “ 2.....	0.102	0.089
“ “ “ 3.....	0.102	0.087
“ “ “ 4.....	0.102	0.088
“ “ “ 5.....	0.102	0.087—
Low phosphorus metal, 1.....	0.042	0.0419
“ “ “ 2.....	0.042	0.0419
Bessemer pig iron, 1.....	0.088	0.079
“ “ “ 2.....	0.088	0.081
“ “ “ 3.....	0.088	0.083
“ “ “ 4.....	0.088	0.081—

The phosphorus given above by titration is an average of from two to five results, done by the men at the works.

Standard phosphorus pig iron of Mr. Camp's.	Phosphorus by weighing yellow precipitate. Per cent.	Phosphorus by above method. Per cent.
1.....	0.109	0.108 \mp
2.....	0.109	0.109 --
3.....	0.109	0.109 --
4.....	0.109	0.109 +

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THE DETERMINATION OF AMMONIA IN MILK.

BY W. N. BERG AND H. C. SHERMAN.

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THE object of this work was to find a method sufficiently delicate for the determination of such small quantities of ammonia as exist in fresh milk, and in which the danger of splitting off ammonia from organic matter during the determination should be reduced to a minimum. Such a method would not only be useful in the study of certain types of fermentation, but would also probably yield results of value from a sanitary standpoint. Changes which result in a breaking-down of proteid matter are more likely to render milk unwholesome than those which affect only the milk sugar, and, while it is possible that some better index of proteid decomposition may be found, the determination of ammonia naturally suggests itself in this connection.

The method which appears to have been commonly used for the determination of ammonia in milk or cheese is to boil either the aqueous infusion of the sample or the filtrate from the precipitation of proteids with magnesium oxide or barium or sodium carbonate under atmospheric pressure, the liberated ammonia being caught in standard acid. Under these conditions urea gradually breaks down with the formation of ammonia. The ammonia which could be thus split off from the amount of urea probably present would doubtless be negligible as compared with the amounts ordinarily found in the analysis of cheese, but might entirely vitiate the determination of ammonia in fresh milk. In