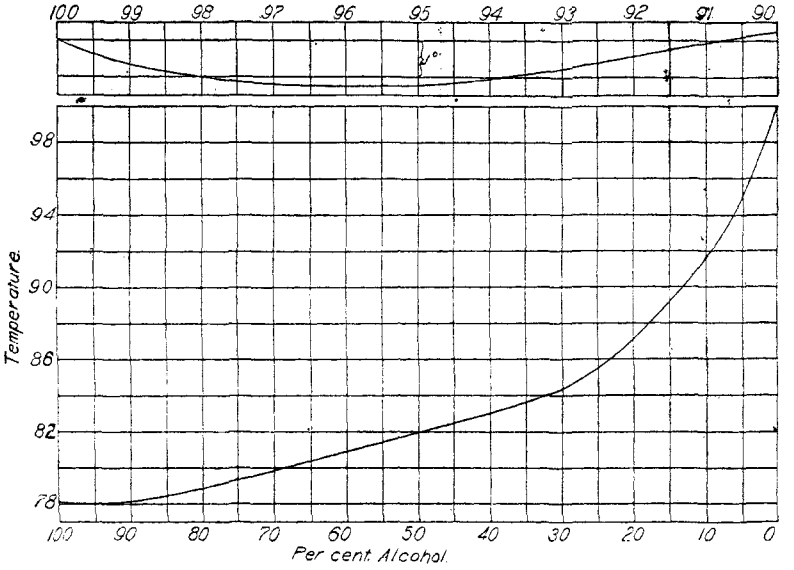

alcohol by distillation to a strength of 30 or 40 per cent. by weight, while the further concentration is relatively difficult.

ROSE POLYTECHNIC INSTITUTE,
TERRE HAUTE, June 1, 1901.

THE VOLUMETRIC DETERMINATION OF ZINC.

BY PERCY H. WALKER.

Received May 15, 1901.

THE most commonly used method for the determination of zinc is the volumetric process of titrating with standard potassium ferrocyanide, using either a uranium solution or one of cobalt nitrate or platinum chloride as indicator. The ferrocyanide titration has several disadvantages. The standard solution does not keep well, and hence must be frequently standardized. If too much ferrocyanide is added in titrating, there is nothing to do but make another determination. The method of using an indicator, by taking out drops, invariably introduces an error.

R. K. Meade[1] has given us a method based on an entirely different reaction. He precipitates the zinc as zinc ammonium arsenate, and uses this arsenate to liberate iodine, which is then titrated by thiosulphate.

The most satisfactory gravimetric process for determining zinc is the precipitation of zinc ammonium phosphate and weighing

[1] This Journal, **22**, 353 (1900).

as pyrophosphate, an analogous process being applied to manganese and magnesium.

Many years ago, Stolba[1] worked out an alkalimetric method for the determination of magnesium, by titrating the magnesium ammonium phosphate with standard acid. Handy's method[2] is a modification of Stolba's. This process may be much more easily used for the determination of zinc than for magnesium.

The process is carried out as follows: To the zinc solution, which should also contain ammonium chloride, a large excess of ammonia is added, then a large excess of sodium phosphate. The solution remains clear; but if the excess of ammonia is cautiously neutralized, a white cloud is formed as each drop of acid falls into the strong ammoniacal liquid. On stirring, this cloud dissolves until nearly all the ammonia is neutralized, when the whole solution becomes milky. It should now be heated to about 75° C. and stirred constantly, at the same time continuing the addition of dilute acid, drop by drop. In a very few minutes the precipitate becomes crystalline, and with care the liquid may be almost perfectly neutralized. It is a good plan to add a small piece of litmus paper to the liquid; this should not turn red but should remain blue or violet, while the hot liquid should have no odor, or only a very faint odor of ammonia. When the precipitation is made as above, the zinc ammonium phosphate is easily filtered, which may be safely done after five minutes' standing. The precipitate should be washed with cold water until the washings show only a faint trace of chlorides, then the paper with the precipitate returned to the beaker in which the precipitation was made, an excess of standard acid added, a few drops of methyl orange, and the exact point of neutrality determined with standard alkali.

According to the equation

$$ZnNH_4PO_4 + H_2SO_4 = ZnSO_4 + NH_4H_2PO_4,$$

we see that 1 cc. of normal acid corresponds to 32.7 mg. zinc. A solution of pure zinc oxide in hydrochloric acid was prepared for testing this method, the following results being obtained:

	Zinc taken. Gram.	Zinc found. Gram.
1	0.1490	0.1486
2	0.1490	0.1481
3	0.1490	0.1486

[1] *Chem. Centrbl.*, 1866, 727, 728; Sutton's "Volumetric Analysis," 6th ed., p. 87.

[2] This Journal, **22**, 31 (1900).

Since the zinc ammonium phosphate is not precipitated in presence of a large excess of ammonia, the process may be used in the presence of magnesium which is precipitated in the strongly alkaline liquid, and the filtrate from the precipitate neutralized to precipitate the zinc. About 1.15 grams of crystallized magnesium sulphate were added to some of the zinc solution, made strongly alkaline with ammonia, sodium phosphate added, and after standing about fifteen minutes with frequent stirring, filtered, washed, and the zinc determined in the filtrate.

Zinc taken.	Zinc found.
0.1490	0.1481

The process gives fairly good results in the presence of iron, calcium, and magnesium, as the following results will show. Where unknown but rather large quantities of solutions of iron (ferric), calcium, and magnesium salts were added to the zinc solution, the whole being strongly alkaline, sodium phosphate was added in large excess, the solution being in a graduated flask which was filled to the mark, mixed, filtered through dry paper and an aliquot part taken for determining the zinc.

Zinc taken. Gram.	Zinc found. Gram.
0.1192	0.1172
0.1192	0.1172

Manganese, however, must be previously separated, best by the nitric acid and potassium chlorate method.

UNIVERSITY OF ARKANSAS, May 8, 1901.

THE EXTRACTION OF MORPHINE WITH IMMISCIBLE SOLVENTS.

BY W. A. PUCKNER.

Received June 11, 1901.

A recent publication by F. Wirthle[1] relative to the extraction of morphine from its solution by means of a chloroform alcohol mixture leads me to publish some similar determinations. My experiments were made with a view of ascertaining whether instead of liberating the alkaloid by addition of fixed alkali there might be substituted ammonium hydroxide. This work was not completed on account of similar determinations published by Kippenberger[2]. While I did not find the substitution of ammonia ad-

[1] *Chem. Ztg.*, **25**, 291.

[2] *Ztschr. anal. Chem.*, **39**, 290.