

## ON THE ESTIMATION OF NITRIC ACID.

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IT is generally supposed that nitric acid in acid solution is only partially converted into ammonia by nascent hydrogen (Fresenius' Quantitative Analysis, 6th edition, p. 348); but I have found that it is quite possible to obtain the whole of the nitric acid present, in the form of ammonia, when proper precautions are taken. When a nitrate is dissolved in water in the presence of sulphuric acid and zinc, the  $N_2O_5$  is first converted into  $N_2O_3$ , and after prolonged action the latter is completely converted into  $NH_3$  by the nascent hydrogen.

As the reduction, when once started, requires but little attention until completed, the time occupied is of little importance, as the analysis can be left standing all night, and will,

in most cases, be complete in the morning. After the reduction the  $\text{NH}_3$  can be distilled into standard acid after making the solution strongly alkaline with caustic soda, and estimated in the usual way by titrating the residual acid with half normal alkali.

Before commencing the distillation it is necessary to test the completeness of the reduction; this is best done by adding about 1 c.c. of decinormal permanganate solution. If the permanganate is quickly destroyed, nitrous acid is still present, and the reducing action must be continued; but if, on the other hand, 1 c.c. produces a permanent tint, the action may be considered complete.

The process will be best described by giving examples: Six separate quantities of 0.5 gram. of pure nitrate of potash were taken and each dissolved in about 80 c.c. of water, 10 c.c. of  $\text{H}_2\text{SO}_4$  (one vol. acid to three of water) was added and a stick of zinc, six inches long, was placed in each flask, and the whole allowed to remain all night; in the morning 5 c.c. more acid was added to again stimulate the evolution of hydrogen. In half an hour the rods of zinc were lifted out, rinsed with distilled water, the samples were tested with permanganate, then rendered alkaline by the addition of three or four short sticks of caustic soda, and distilled into 10 c.c. of normal sulphuric acid contained in a U tube, having a bulb blown on each arm, and being immersed in cold water. After the distillation the contents of the U tube were washed into a beaker and titrated with half normal  $\text{NaHO}$  with the following results:—

No.	$\text{K}_2\text{Mn}_2\text{O}_8$ destroyed.	Standard Acid neutralized=	$\text{KNO}_3$
1	2.0 c.c.	4.9 c.c. =	.505
2	1.0 c.c.	4.9 c.c. =	.499
3	0.0 c.c.	5.0 c.c. =	.505
4	0.0 c.c.	4.9 c.c. =	.494
5	.5 c.c.	4.9 c.c. =	.496
6	2.0 c.c.	4.8 c.c. =	.494

The calculations are made, in the case of nitrate of potash, by multiplying the number of c.c. of permanganate and acid respectively by .00505 and .101, and adding the results together; but I prefer to have the whole of the nitrous acid converted into ammonia, or at least not to have more than is equal to 1 c.c. of permanganate left, as experiments made in which one-third or one-half of the whole of the nitrous acid was purposely left unconverted, and allowed for by titration with permanganate, were very unsatisfactory.

I think this method will be found more simple and manageable than Harcourt's, and the results fully as accurate; but, of course, reducible and oxidizable substances, as iron, &c., must be absent.

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