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A METHOD FOR THE DETERMINATION OF HYDRIODIC AND HYDROBROMIC ACIDS.

BY W. S. HENDRIXSON.

About a year ago I presented to the Iowa Academy of Sciences a paper on a method of determining chloric acid by the reduction of the acid with metallic iron in the presence of dilute sulphuric acid, and the titration of the hydrochloric acid formed, by the method of Volhard. The same paper, somewhat extended, appeared in another periodical.* In this communication it was suggested that the method might probably be used equally well for the determination of bromic and iodic acids, and two determinations of bromic acid were included in the paper.

Early in the present academic year the study of the action of metallic iron on bromic and iodic acids, with a view to the determination of the acids, was taken up with the results that follow in this paper.

Of course there could be little doubt that iron in acid solution would completely reduce both these acids giving their equivalents in hydrobromic and hydriodic acids, but since these are both strong reducing agents, they might be expected as soon as formed to react with the iodic and bromic acids remaining, with the liberation of six equivalents of bromine or iodine, according to well known reactions. The question to be settled really was, whether the iodine and bromine thus liberated would be readily changed at low temperature to the corresponding hydriodic and hydrobromic acids, so as to avoid any loss of iodine or bromine by volatilization.

* American Chemical Journal, Vol. 32, p. 242.

Determination of Iodic Acid.—As the basis for the work on iodic acid there was used a tenth normal solution of potassium iodate, made from an excellent sample by Merk, as the following determinations indicate. Portions of 20 c.c. of the decinormal solution were digested with potassium iodide and hydrochloric acid in a well stoppered bottle in the usual way, the solutions of free iodine thus obtained were made up to known volumes and portions were titrated with standard decinormal sodium thiosulphate:

(1) 20 c.c. of iodate solution gave iodine which required 120.32 c.c. thiosulphate.

(2) 20 c.c. of iodate solution gave iodine which required 120.40 c.c. of thiosulphate.

As well known it is practically impossible to obtain commercially, potassium iodide free from iodate. In the above experiments five grams of iodide were used in each case. The same weight of the iodide digested with hydrochloric acid gave free iodine which required 0.30 c.c. of thiosulphate. Applying this correction the volumes of thiosulphate required in experiments (1) and (2) are 120.02 and 120.10, while the theory for a truly decinormal solution of potassium iodate would require 120 c.c.

In the determination of both iodic and bromic acids, the iron in the form of washed card teeth, and 25 c.c. of dilute, pure sulphuric acid and a measured volume of the solution to be determined were placed in a distilling flask with a well-ground glass stopper. The side-tube of the flask was placed in a small quantity of water in a test tube in order to condense and make apparent any iodine or bromine that might pass over. At first in the experiments with iodate, alcohol was used to absorb the iodine, but water was substituted after it was found that with proper regulation of the temperature the amounts of iodine or bromine that passed over were practically negligible. On placing the substances together in the flask iodine was at once liberated, and considerable quantities soon settled upon the bottom of the flask. At room temperature the

vapor of iodine was not observed to rise above the bulb of the flask, but the complete conversion of the iodine into hydriodic acid required at room temperature three or four hours. If, however, the bulb of the flask was immersed in water kept at 50° it was found that the reaction went on much more rapidly and was completed in about one hour. Even at this temperature very little iodine came over, and this small quantity was, near the end of the experiment, returned to the flask by removing the flask from the water so that the liquid in the test tube and the wash water sucked back into the flask. The experiment was allowed to continue till all trace of the color due to iodine had disappeared and the liquid showed only the clear, light green color due to ferrous iron.

In the five following experiments the hydriodic acid formed was determined, after oxidizing the iron in solution to the ferric condition, by titration with a twentieth normal solution of silver after the method of Volhard. The silver solution was added in excess, the silver iodide was filtered off and the excess of silver was determined in the filtrate, with sulphocyanate.

- (1) 10 c.c. iodate solution required 20.00 c.c. silver solution.
- (2) 10 c.c. iodate solution required 19.90 c.c. silver solution.
- (3) 20 c.c. iodate solution required 40.07 c.c. silver solution.
- (4) 20 c.c. iodate solution required 40.10 c.c. silver solution.
- (5) 20 c.c. iodate solution required 39.75 c.c. silver solution.

The solution obtained on oxidizing the iron was not perfectly colorless, and the color interfered somewhat with the titration with sulphocyanate. The best results were obtained when the solution was boiled to free it from nitrogen oxides and then allowed to stand till quite cold. Since it is questionable whether by Volhard's method it is permissible to determine halogens by titrating the excess

of silver without filtering off the silver halides it seems best to escape the disadvantage of titrating a colored solution by weighing the silver halide direct. In the following experiments the hydriodic acid was precipitated as silver iodide which was weighed.

(1) 20 c.c. iodate solution gave .4726 grams AgI, corresponding to .2554 grams iodine.

(2) 20 c.c. iodate solution gave .4764 grams AgI, corresponding to .2566 grams iodine.

(3) 20 c.c. iodate solution gave .4764 grams AgI, corresponding to .2574 grams iodine.

The theory requires .2539 grams iodine for 20 c.c. of the solution.

Determination of Bromic Acid.—The potassium bromate used in the following determinations was made by Merk. It contained no bromide. It was dried to constant weight at 100°. A deci-normal solution was made up and its strength was determined by digestion with hydrochloric acid and potassium iodide. 20 c.c. of this solution gave on digestion an amount of iodine which required 120.08 c.c. of a standard solution of sodium thiosulphate, deci-normal, while the theory for 20 c.c. of a normal solution of the bromate would require 120 c.c. of deci-normal thiosulphate.

The reduction with iron was carried out as already described under iodic acid, but it was found better to keep the solution at room temperature. In this case the complete reduction required about two hours. Usually no color of bromine was visible above the bulb of the flask, and care was taken to prevent the escape of any bromine by the same means as in the case of iodine. The amount of iron dissolved made it even more difficult than in the case of hydriodic acid, to use the Volhard method. Three determinations by this method gave results decidedly too low; and in fact, in three experiments in each of which 20 c.c. of bromate were used the volumes of silver used were 39.11, 39.35 and 39.07 c.c.

In the following experiments the hydrobromic acid was precipitated as silver bromide which was weighed:

(1) 20 c.c. bromate solution gave .3753 grams AgBr, corresponding to .1596 Br.

(2) 20 c.c. bromate solution gave .3764 grams AgBr, corresponding to .1603 Br.

(3) 20 c.c. bromate solution gave .3741 grams AgBr, corresponding to .1593 grams Br.

(4) 20 c.c. bromate solution gave .3758 grams AgBr, corresponding to .1599 grams Br.

The theory requires .1599 Br.

The method described seems to serve equally well for chloric, bromic and iodic acids. It is simple and direct, requires no specially constructed apparatus and in case one determines the hydrochloric, hydrobromic or hydriodic acid gravimetrically it requires no standard solutions.

Iowa College, Grinnell, Iowa, April 15, 1905.