ART. XXXII.—*A Volumetric Method for the Determination of Hydrazine;* by GEORGE S. JAMIESON.

A METHOD for the determination of hydrazine has been described by Rimini[*] and recently tested further by Hale and Redfield.[†] This is based upon the oxidation of the hydrazine compound in aqueous solution by the addition of an excess of standard potassium iodate solution, according to the following equation:

$$5(N_2H_4.H_2SO_4)+4KIO_3 = 5N_2+12H_2O+2I_2+2K_2SO_4+3H_2SO_4$$

The liquid is then boiled until the iodine is expelled and the excess of potassium iodate is found after cooling by adding potassium iodide, acidifying with sulphuric acid, and titrating with sodium thiosulphate solution. The chief objection to this method, according to Hale and Redfield, is the length of time required for the analysis.

The method to be described here is based upon the titration of hydrazine by potassium iodate in a strong hydrochloric acid solution, according to the general method of L. W. Andrews.[‡] It has the advantages of being rapid, and requiring the use of only a single, very stable volumetric solution. Besides, as will be seen from the results that follow, it is very accurate.

In order to test the method, a solution containing 3·567 g. of KIO_3 in 1000^{cc} was prepared. According to the equation of the expected reaction

$$N_2H_4.H_2SO_4+KIO_3+2HCl = N_2+ICl+3H_2O+KCl+H_2SO_4$$

the equivalent of this solution is 1^{cc} = ·002169 g. $N_2H_4.H_2SO_4$ and 1^{cc} = ·000534 g. N_2H_4. Weighed portions of pure hydrazine sulphate were placed in a 250^{cc} glass stoppered bottle together with 20^{cc} of water, 30^{cc} of hydrochloric acid and 6^{cc} of chloroform. Then the potassium iodate solution was run in gradually, with shaking between the additions, until the chloroform, after increasing and then diminishing in color, was just decolorized. The following results were obtained:

	$N_2H_4.H_2SO_4$ taken	KIO_3 used	$N_2H_4.H_2SO_4$ found	Error
I	·0487^g	22·5^{cc}	·0488^g	+·0001
II	·0434	19·9	·0432	−·0002
III	·0589	27·3	·0592	+·0003
IV	·0472	21·9	·0475	+·0003
V	·0986	45·65	·0990	+·0004
VI	·1060	49·00	·1063	+·0003

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[*] Gazz. chim. ital., xxix, I, 265, 1899.

[†] Journ. Amer. Chem. Soc., xxxiii, 1362, 1911.

[‡] Journ. Amer. Chem. Soc., xxv, 756, 1903.

Determinations of hydrazine in the sparingly soluble double sulphates of zinc, cobalt, nickel, and cadmium were made in order to test the method further. The double salts were prepared by mixing hot solutions of the component sulphates in the presence of a little sulphuric acid, and after digesting for some time on the steam-bath, the crystalline products were filtered off by suction, washed with cold water and dried at 100° C. The titrations were carried out in the same way as described above in the case of the simple sulphate. It was observed that the nickel salt reacted very slowly, apparently on account of difficult solubility, while the other compounds were titrated about as readily as hydrazine sulphate alone.

The following results were obtained:

	Substance	KIO_3 used	N_2H_4 found	Calculated
$ZnSO_4(N_2H_4)_2.H_2SO_4$	·1163^{gr}	43·2^{cc}	19·83%	19·81%
"	·0880	32·6	19·77	"
$CdSO_4(N_2H_4)_2.H_2SO_4$	·1591	51·25	17·21	17·29
"	·1396	45·10	17·25	"
$NiSO_4(N_2H_4)_2.H_2SO_4$	·0692	26·30	20·29	20·23
"	·0890	33·80	20·28	"
$CoSO_4(N_2H_4)_2.H_2SO_4$	·1308	49·20	20·09	20·20
"	·1059	39·70	20·02	"

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