

CATALYSIS IN VOLUMETRIC ANALYSIS.

PART V. COLORIMETRIC ESTIMATION OF VANADIUM.

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Gopala Rao and Ramanjaneyulu (1945) have developed an iodimetric method for the volumetric estimation of vanadium in the pentavalent state, making use of the reaction between hydriodic acid and vanadate, catalysed by oxalic acid. We have now adapted the same reaction for the colorimetric estimation of minute quantities of vanadate, the colour used for comparison being the starch-iodine blue.

EXPERIMENTAL.

The reaction between vanadate and hydriodic acid is allowed to take place in an atmosphere of carbon dioxide in the presence of sodium oxalate, which catalyses the reaction. The liberated iodine gives a blue colour with the starch present. The blue colour produced in the test solution is compared with that produced in a standard solution under identical conditions.

10 ml. of vanadate solution are taken in a narrow cylindrical gas washing bottle, fitted with a ground glass stopper with fused inlet and outlet tubes, 10 ml. of 1% potassium iodide solution were then added, followed by 5 ml. of 0.1N sodium oxalate and 10 ml. of starch solution of 0.25% strength. The volume is made up to 50 ml. with distilled water and a current of carbon dioxide was passed through the solution briskly for about half an hour to ensure the complete elimination of dissolved air. The stream of carbon dioxide was somewhat slowed down, the stopper of the reaction vessel was removed for a while and 5 ml. of 4N sulphuric acid were run in quickly. The stopper was replaced and a slow stream of carbon dioxide was passed through for five minutes, after which the entrances and exits were all closed airtight. After standing thus for the requisite interval of time, the solution is transferred to a measuring flask and the volume made up to the mark (200 or 500 ml.), so that the test solution matches with the standard. The intensity of the colour produced is compared with that of a standard prepared under identical conditions. Enough starch must be added so that all the iodine present in the solution forms the blue colour. The starch used must be high grade soluble starch.

In the following table are recorded a few typical results of numerous determinations, which show that the amount of vanadate obtained by the authors' colorimetric method agrees fairly well with that taken.

Amounts of vanadate varying from 0.001 millimoles to 0.0001 millimoles produce, under conditions already mentioned, a true blue colour which is proportional in intensity to the amount of vanadate present; the colour is easily produced and matched clearly in a colorimeter. The colour remains stable for one hour or more, when compared against a standard prepared at the same time as the test solution.

TABLE I.

Amount of vanadate found. Authors' colorimetric method.	Amount of vanadate taken.
millimoles.	millimoles.
0.0100	0.0100
0.0086	0.0090
0.0079	0.0080
0.0069	0.0070
0.0059	0.0060
0.0049	0.0050
0.0039	0.0040
0.0030	0.0030
0.0010	0.0010
0.00092	0.0009
0.00071	0.0006
0.00048	0.0005
0.00040	0.0004

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