

ethyl β -bromopropionate used). The ester is quite pure and can be used straight-away for further synthetic operations.

In many typical experiments the yield ranged from 75 to 85% of the theory. The quantity of sodium ethoxide used affects the yields considerably. Good yields are obtained only by using 2 to 2.5 g. atoms of sodium for one molecule of ethyl β -bromopropionate. When only one gram atom of sodium (which is just the theoretical amount necessary) was used, the yields were very low, 20 to 25% presumably due to the formation of ethyl acrylate as a by-product. Excess of sodium ethoxide present presumably reacts with ethyl acrylate formed converting it into ethyl β -ethoxypropionate as observed by Purdie and Marshall (*loc. cit.*).

The author's thanks are due to Prof. G. Sankaran for his interest in this work.

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Received April 17, 1946.

A NOTE ON THE IODOMETRIC ESTIMATION OF TRIVALENT ANTIMONY

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The method of Dunstan and Boole (*Pharm. J.*, 1889, 19, 385) for the iodometric estimation of trivalent antimony, in which sodium bicarbonate is used to neutralise the acid formed in the course of the reaction, suffers from the serious defect that if the titration is not finished quickly, antimonious oxide comes down as a precipitate. This is due to the neutralisation of the tartaric acid by sodium bicarbonate. It was thought that if sodium acetate was used, no neutralisation of the tartaric acid would take place and the titration could be carried smoothly; but it was found that a very slight precipitation of antimonious oxide did take place. So small amounts of acetic acid were added to the solution containing tartar emetic and sodium acetate and no precipitation took place even if traces of acetic acid were present. As expected it was found that the reverse reaction, the reduction of antimonic acid by hydriodic acid, took place if the concentration of hydrogen ion was large, but up to a concentration of $N/32$ (in the solution to be titrated) there was no sign of the above reverse reaction having taken place.

The following is the summary of experimental results. 25 C.c. of $N/10$ tartar emetic were taken and 5 g. of sodium acetate were added in every case. The strength of the acetic acid which differed from solution to solution is indicated by [HAc] and the volume of iodine used is indicated by "V" in case of all the experiments.

[HAc] " V "	N	N/2	N/4	N/8	N/10	N/32	N/64	0
	18.1	18.15	18.2	18.2	18.2	18.25	18.25	18.25

At room temperature sodium bicarbonate method and sodium acetate-acetic acid method yield the same results. Only the titration is easier in the second case and one has not to hurry up.

I am thankful to Prof. B. Prasad for suggesting the problem to me.

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Received April 12, 1946.

