

Fig. 3 shows the absorption curves of the salts of ethylated compounds. There is very little difference between these absorption spectra and those of the salts of the de-ethylated compounds, that is, the dyes.

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[Received, August 28th, 1915.]

CLXIV.—*The Preparation of Ethyl Bromide.*

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THE preparation of ethyl bromide has assumed great importance of late, and hence any improvements in the methods for increasing its yield are to be welcomed.

The laboratory methods—based on the method of De Vrij (*Jahresber.*, 1857, 441)—as given in the usual practical books on “organic preparations” fall short in the following respects:

(1) A large excess of alcohol is used, giving a yield of less than 50 per cent. of ethyl bromide on the alcohol used.

(2) The formation of ether, owing to (*a*) the large excess of alcohol used, and (*b*) the high temperature used in the reaction. This necessitates special treatment of the crude ethyl bromide for the removal of the ether, with concomitant loss of ethyl bromide.

(3) The use of potassium bromide instead of the less expensive sodium bromide.

By using the quantities given below, and paying attention to the rate of heating the reaction mixture, a minimum yield of pure ethyl bromide of 80 per cent. of the theoretical on either the sodium bromide or alcohol used can be obtained, and with special care yields of 90 per cent. are possible; also, it is possible to obtain these yields without the formation of even a trace of ether, thus obviating loss of time and material in purifying the crude product.

Preparation of Ethyl Bromide.

Quantities of Materials.—(1) Molecular proportions by weight of absolute alcohol, sulphuric acid (D 1.84), and sodium bromide.

(2) Water to the extent of 5–10 per cent. of the weight of alcohol taken.

(3) Excess of sulphuric acid to the extent of 5 per cent. of the theoretical weight required.

Method.—The alcohol and water are mixed in a round-bottom flask of requisite size, and the sulphuric acid is added, the mixture being well cooled during the process. To the cold mixture, the sodium bromide, in very coarsely powdered form, is added. The flask is then supported in a capacious water-bath fixed to a good condenser, the outlet of which dips into water contained in a stout, wide-mouthed bottle standing in a trough of cold water. The water-bath is heated very gently, so that the rise of temperature is slow, until drops of ethyl bromide are seen to fall from the condenser into the receiver; the temperature is only allowed to rise as the rate of formation of ethyl bromide slackens, until the water is boiling; when the rate of formation of ethyl bromide at this temperature slackens, the water-bath is removed and a sand-bath put in its place; again the temperature is carefully raised, so that the ethyl bromide distils over regularly, the heating being continued until no more ethyl bromide is obtained. By paying careful attention to the heating, no frothing is obtained, and for quantities of six gram-molecular weights of each of the reacting substances a 2-litre round-bottomed flask is of ample size. The only loss, so far as has been ascertained, is that of hydrogen bromide during the first hour of heating, and this doubtless would be brought to a minimum by increasing the quantity of water, within limits, and allowing longer time for heating. Most of the hydrogen bromide is dissolved in the water contained in the receiver, and can be recovered as sodium bromide. The crude ethyl bromide is separated from the acid water, washed with very dilute sodium carbonate solution, and then once with water, and allowed to remain over fused calcium chloride for half an hour. On distilling slowly from a water-bath, it will be found that more than 99 per cent. of the crude ethyl bromide will distil between 38.5° and 39.5°.

The following results are typical of those obtained by the method described:

Ethyl alcohol. Grams.	Water. Grams.	Sulphuric acid. Grams.	Sodium bromide. Grams.	Ethyl bromide. Grams.	Percentage yield of theoretical.
92	—	200	206	185	84.9
138	—	300	309	270	85.6
184	—	400	412	370	84.9
276	15	620	618	550	84.0
276	20	625	618	556	85.0

THE CHEMICAL DEPARTMENT,
THE POLYTECHNIC,
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[Received, July 23rd, 1915.]