1592 HUNT : THE PREPARATION OF ETHYL IODIDE.

CLXXVI.—*The Preparation of Ethyl Iodide.*

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THE preparation of ethyl iodide has recently been investigated by Adams and Voorhees (*J. Amer. Chem. Soc.*, 1919, **41**, 789), who recommend the employment of Walker's method (T., 1892, **61**, 717) in a slightly modified form. An alternative method, however, appeared possible in view of the work of Beilstein and Rieth (*Annalen*, 1863, **126**, 250), and this process forms the subject of the present communication.

Experiment showed that with the use of 80 per cent alcohol in place of absolute alcohol, the vigour of the reaction, caused by the addition of iodine, was so much reduced that the time required to mix 500 grams of iodine with the requisite amount of alcohol and red phosphorus was not more than twenty minutes. Further, the yield was not affected by the omission of the customary period of keeping at the ordinary temperature, but boiling under reflux for two and a-half hours, followed by immediate distillation, was sufficient to ensure a good yield. The use of a large excess of alcohol is often suggested, but this proved unnecessary, and, indeed, rendered the process of purification more difficult, and thus reduced the yield.

In most modern text-books, the main reaction is regarded as taking place according to the equation

but Beilstein (*loc. cit.*) interprets it thus:

Estimations of the amount of unused phosphorus remaining after distillation agreed with this interpretation, which was further verified by the preparation of ethyl iodide, using the amount of phosphorus required by Beilstein's equation, when a good yield was obtained. In practice, however, it proved better to use an excess of phosphorus to ensure purity of the product.

EXPERIMENTAL.

To 500 grams of iodine in a flask of 4 litres capacity, cooled by water, about 281 grams of 80 per cent. alcohol (by weight) were added, and thereafter, during the course of about twenty minutes, 50 grams of red phosphorus, with shaking after each addition to ensure complete mixing. The first additions of phosphorus caused evolution of heat and boiling of the liquid in the flask, but no loss of vapour occurred. A reflux condenser was then attached to the flask, and the latter heated in a water-bath, so that the contents boiled gently. After about two and a-half hours, iodine vapour was no longer visible, although the mixture remained dark in colour. The ethyl iodide was, without cooling, distilled off, through a short, two-bulb condensing column, by heating, first in a water-bath and afterwards on a brine-bath. All the distillate passed over at 63—83°, and was colourless, except for a small fraction at the end, which had a yellow tinge. A few drops of sodium carbonate solution were added to ensure absence of free hydriodic acid, and a few drops of a dilute solution of sodium thiosulphate

to remove any free iodine, since ethyl iodide purified with sodium thiosulphate was proved to darken less quickly on keeping than when sodium carbonate alone was used, and the purification was accomplished more rapidly. The oil was then washed with water, dried over calcium chloride, and redistilled. The yield was 566 grams (92 per cent. of the theoretical), and the unused red phosphorus amounted to 24.45 grams.

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