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Chemistry
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Longmans p 407

III,111.

ACETONITRILE

Attach a short Liebig condenser to a dry 250 ml. distilling flask ; use a small conical flask as a receiver. Owing to the extremely hygroscopic character of phosphoric oxide (1), the latter must be weighed out and transferred to the flask as rapidly as possible. Wrap some glazed paper around a glass tube and insert it into the flask until the lower end enters the bulb ; upon removing the glass tube, the paper roll expands and thus lines the neck of the distilling flask. Weigh out on pieces of glazed paper (using a rough balance) first 20 g. of acetamide, and then, *as rapidly as possible*, 30 g. of phosphorus pentoxide. Immediately transfer, with the aid of a spatula, the phosphoric oxide down the glazed paper cylinder into the distilling flask, then introduce the acetamide similarly, remove the paper, and at once cork the flask and mix the contents well by gentle shaking (2). Heat the flask cautiously with a small luminous flame kept in constant motion and applied uniformly over the bottom of the flask. A reaction, accompanied by much frothing, takes place. After the mixture has been heated for 4-5 minutes, distil the acetonitrile into the receiver using a somewhat larger luminous flame kept in constant motion around the flask. Add half the volume of water to the distillate, and then anhydrous potassium carbonate until the aqueous layer is saturated (about 9 g. of potassium carbonate are required for every 10 ml. of water); cool the flask in cold or ice water during the addition of the solid to prevent the loss of methyl cyanide by evaporation (3). Allow the excess of solid potassium carbonate to settle and decant the liquid to a small separatory funnel. Run off the lower carbonate layer, and transfer the upper layer through the mouth of the funnel to a small (25 ml.) distilling flask into which 2-3 g. of phosphorus pentoxide have been placed.

Fit a thermometer and small condenser to the flask, add 2-3 fragments of porous porcelain and distil slowly. Collect the fraction boiling at 79-82° as acetonitrile. The yield is 10 g.

Notes.

(1) Phosphorus pentoxide must be handled with great care since it produces painful burns if allowed to come in contact with the skin.

(2) Wet the papers thoroughly with water before throwing them away, as the residual phosphoric oxide may cause them to smoulder.

(3) The functions of the potassium carbonate are (a) to neutralise the acetic acid arising from the action of the phosphoric acid upon the acetamide, and (b) to "salt out" the otherwise soluble methyl cyanide as an upper layer.