Ref: Vogel's
Textbook of Practical Organic Chemistry
3e edition
Longman p 372

III,89. ACETIC ANHYDRIDE

Assemble an apparatus consisting of a 100 or 125 ml. distilling flask carrying a dropping funnel, the stem of which passes below the side arm : attach the distilling flask to a condenser for downward distillation and use a 50 or 100 ml. distilling flask as receiver. Place 28 g. of finely powdered anhydrous sodium acetate (for preparation, see Section **II,50,9**) in the flask and 20 g. (18 ml.) of acetyl chloride in the dropping funnel. Disconnect the distilling flask from the condenser and immerse it in cold water or in ice water. Add about half of the acetyl chloride drop by drop ; then remove the flask from the cooling bath and mix the contents thoroughly by cautious shaking and tapping of the flask against the palm of the hand. Return the flask to the cooling bath and run in the remainder of the acetyl chloride drop by drop. Do not allow the mixture to get so hot that it boils. When all the acetyl chloride has been added, remove the separatory funnel and replace it by a solid cork ; thoroughly mix the contents of the flask as above. Attach the flask to the condenser and receiver. Clamp the flask at such a height that it can easily be heated by a Bunsen burner. Heat the flask by means of a luminous, smoky Bunsen flame, which is kept in constant motion round the base of the flask to ensure uniform heating and minimise the danger of cracking the flask. Continue the heating until no more liquid passes over. Add 2–3 g. of finely-powdered anhydrous sodium acetate to the distillate in order to convert any unchanged acetyl chloride into acetic anhydride, insert a cork carrying a thermometer into the flask, attach a condenser, and distil slowly. Collect the fraction which passes over at 135–140° as acetic anhydride. The yield is 20 g.