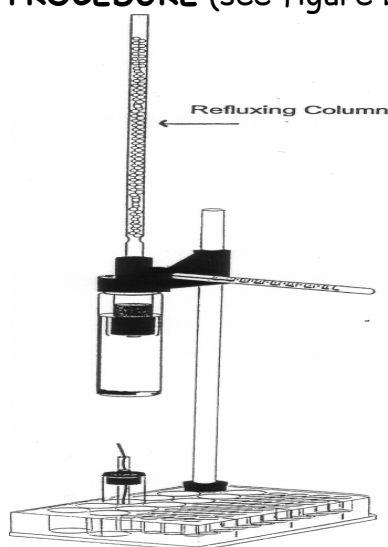


THE SYNTHESIS OF ETHANOIC ACID

You will need

Ethanol
 Potassium dichromate(VI)
 Sulphuric acid (conc.)
 Combostill
 Refluxing column (filled with glass beads)
 Distillation column (filled with glass beads)
 Boiling chips
 Silicone oil Fraction collector
 Reaction and heating vessels
 Thermometer
 Microburner + meths for heating
 Ethanol
 2 ring stands

PROCEDURE (see figure below)



PART 1

1. Add a small amount of boiling chips to the reaction vessel.
2. Weigh out between 170 and 200 mg of potassium dichromate(VI).
3. Add the dichromate to the reaction vessel.
4. Place the reaction vessel in a bath of cool water.
5. Next slowly add 5-7 drops of concentrated sulphuric acid to the reaction vessel, keeping it in the cool water bath.

6. Slowly add 1.8-2.0 ml of ethanol to the mixture in the reaction vessel.

PART II

7. Set up the Combostill apparatus with the refluxing tube in the vertical position over the Combostill.

8. Add a small amount of boiling chips to the heating vessel and fill with the silicon oil to the level of % of the volume of the vessel.

9. Place the heating vessel into position on the Combostill head.

10. Insert the thermometer into the Combostill head.

11. Place the microburner into Well F4.

12. Ensure that the base of the heating vessel and the microburner are at least 7-8 cm apart.

13. This will provide an initial amount of energy from the microburner to begin the refluxing. There may be some initial bumping of the reaction mix as the heating takes place; should this occur, move the microburner from Well F4 to Well F5.

14. Light the microburner and slowly permit the transfer of heat energy through the silicone oil to the reaction vessel.

15. As refluxing begins the mixture may turn a dirty brown to black colour.

16. Keep the refluxing going for at least 20 minutes.

THE SYNTHESIS OF ETHANOIC ACID (continued)

PART III

17. Remove the heat source from the reflux condenser and allow for a cooling period.

18. Remove the refluxing column and replace with the distillation column

19. Next set up the fraction collector

20. You are now ready to collect the distillate that has been synthesized in the reaction vessel.
21. The heating process will occur slowly as the product (ethanoic acid) has a high boiling point (108°C).
22. The temperature registering on the thermometer may not reach this point; however, you will detect the pungent odour of vinegar in the distillate, indicating the presence of ethanoic acid.

QUESTIONS ON THE PREPARATION

1. What is the function of the potassium dichromate in the preparation?
2. Write the balanced equation for the oxidation of ethanol to ethanoic acid.
3. Explain the term 'reflux'
4. Explain why the reaction mixture should be refluxed
5. In this experiment the mass of ethanol ($M_r = 46$) used was approximately 1.6g
 - a) How many moles of ethanol is this?
 - b) How many moles of ethanoic acid could theoretically be formed from this mass of ethanol?
Explain your answer
 - c) In such a preparation one student obtained 0.73 g of ethanoic acid ($M_r = 60$).
 - i) What is the percentage yield from this preparation? Show your working. Give two reasons why the yield is less than 100%.