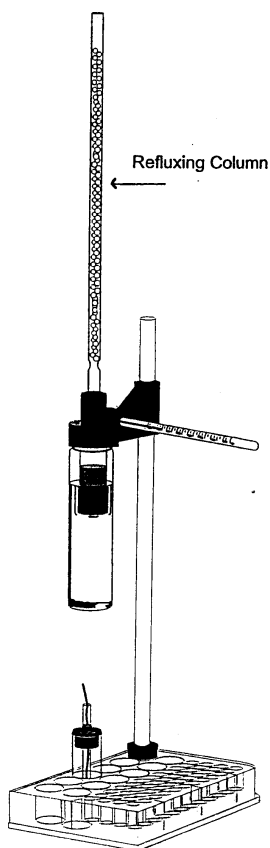


THE PREPARATION OF ETHYL ETHANOATE

Introduction

The procedure for preparing ethyl ethanoate requires refluxing the components for a period of at least five minutes.



NOTE: Ethyl ethanoate is extremely flammable. Be extra careful when refluxing or distilling not to permit the vapours or the liquid to come into contact with an open flame. Ethyl ethanoate also dissolves polystyrene; therefore, distil into a glass container or the fraction collector.

You will need

Ethanol

Ethanoic acid (glacial)

Sulfuric acid (13.5M)

Combostill

Refluxing column (filled with glass beads) Distillation column (filled with glass beads) Boiling chips

Silicone oil

Microburner

Propettes



PROCEDURE

PART I

1. Add 1.5 cm^3 of ethanol to the reaction vessel.
2. Add 1.5 cm^3 of glacial ethanoic acid to the reaction vessel.
3. Add 2-3 drops of the 13.5M sulphuric acid to this mixture. Add a few boiling chips.
4. Attach the reaction vessel to the Combostill head.

PART II

5. Fill the heating vessel with silicone oil to a level X below the top. This should require about 25 cm^3 .
6. Add a few boiling chips to the heating vessel.
7. Attach the heating vessel to the Combostill head. The silicone oil level should cover $1/2$ of the reaction vessel.
8. Attach the reflux column to the Combostill head, and place the thermometer into the sidearm of the Combostill head. (See diagram of reflux apparatus on p.127).
9. Attach this unit to the ring stand that should be placed into Well F1.
10. The microburner should now be placed into Well F4 to sit just under the Combostill heating vessel.
11. Light the burner and gently allow the burner to heat the bottom of the heating vessel.
12. As the silicone oil is heated, one should see the heat currents flow in this vessel.
13. The reaction vessel contents will begin to boil and, as this occurs, remove the direct heat from the burner by placing it into Well F5.
14. Reflux for a short period of time (about 3-5 minutes).



PART III

15. Remove the heat source completely and allow the system to cool down.
16. After cooling, remove the reflux column and replace it with the distillation column.
17. You are now ready to distil the product boiling at 76.5-77.5°C.
18. Ethanol boils at 78°C and ethanoic acid boils at 117-118°C. This means that the product has a lower boiling point than either of the reactants. However, due to the closeness of the two boiling points, some of the unreacted ethanol will certainly distil over with the product.
19. Do not distil the material to a level in the reaction vessel below X.

QUESTIONS ON THE PREPARATION

1. Give the equation for the reaction using the structural formulae of the two reactants and products
Give the skeletal formula of the product
2. The reactants are refluxed together. Explain the term refluxed
Why are they refluxed together?
3. What is the purpose of the concentrated sulphuric acid? Give 2 reasons
4. In this preparation:-
1.5 cm³ of ethanol is used (density = 0.79 gcm⁻³ and Mr = 46);
1.5 cm³ of ethanoic acid is used (density = 1.05 gcm⁻³ and Mr = 60)
The density of ethyl ethanoate is 0.90gcm⁻³
 - a) What mass of ethanol is used?
 - b) How many moles of ethanol is this? What mass of ethanoic acid is used? How many moles is this?
 - c) i) Which of the two reagents is in excess?
ii) Explain your answer
 - d) How many moles of ethyl ethanoate should be formed? This is the theoretical yield.
 - e) In an identical preparation, 1.36 g of ethyl ethanoate (Mr = 88) were obtained. How many moles of ethyl ethanoate is this?
 - f) What is the actual yield?
 - g) Explain why the yield may be less than 100%. Give 2 reasons