

PREPARATION OF AN ESTER - METHYL SALICYLATE (OIL OF WINTERGREEN)

Safety

Always refer to the departmental risk assessment before carrying out any practical work. See the Part 2 Notes on practical work, found in the Additional resources section, for additional guidance and Hazcard references. Wear eye protection at all times.

Apparatus per student

Combo Still including glass balls as in distillation and reflux combinations

Combo plate

Silicone oil

Salicylic acid

Methanol \blacktriangle (CARE!)

Concentrated sulfuric acid \blacktriangle (CARE!)

Boiling chips

Propette

Bromine water

Micro spatula

Micro burner + meths and wick

Ring stand

Propettes

Top-pan balance reading to 2 decimal places

Method

1. Weigh out 1.31g of salicylic acid (approximately 0.01mol).
2. Add the salicylic acid to 3.0 cm³ of methanol in the reaction vessel.
3. Stir to ensure that the powder is completely dissolved.
4. Add 2 spatulafuls of boiling chips to the reaction vessel.
5. Using a Propette add 3 drops of the concentrated sulfuric acid to the reaction mixture.
6. Set up the reaction vessel and Combostill for refluxing.
7. Heat the reaction mixture using the Micro burner.
8. Monitor the reaction, making sure that no violent eruption occurs.
9. Continue heating for at least 5 minutes.
10. Stop heating and allow to cool.
11. Remove the reflux condenser and replace with the distillation apparatus.
12. Start heating again and distill off the unreacted methanol.
13. Do not distill the liquid below 1/3 the original volume.
14. Stop heating and allow to cool.
15. Remove the heating vessel and the reaction vessel.
16. As the reaction vessel cools, crystallisation will occur. From the smell it is clear that we have synthesized methyl salicylate.

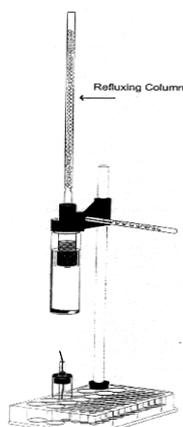


Figure 3

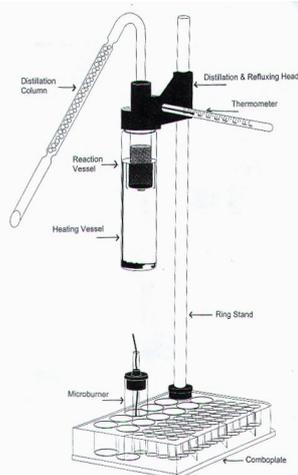


Figure 1