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.

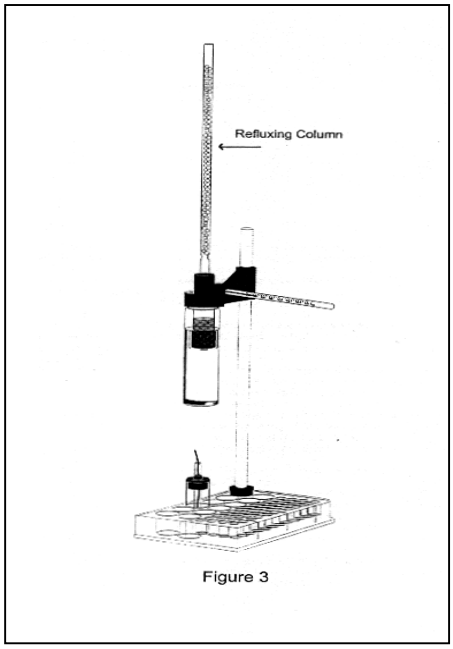

Figure 3

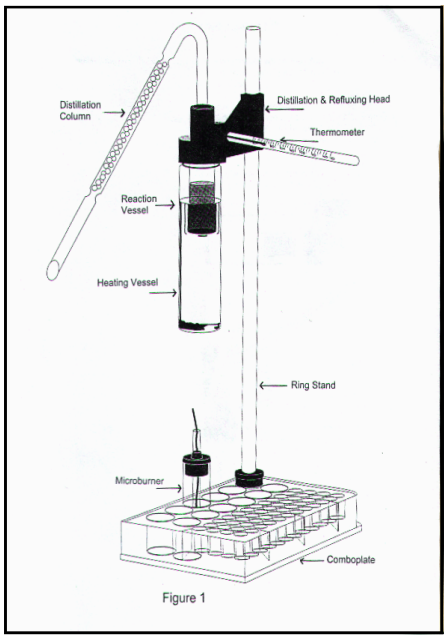

Figure 1

Apparatus per student

Combo Still including glass balls as in distillation and reflux combinations
Combo plate
Silicone oil
Salicylic acid
Methanol (CARE!)
Concentrated sulfuric acid (CARE!)
Boiling chips
Propette
Bromine water
Micro spatula
Micro burner + meths and wick
Ring stand
Propettes
Top-pan balance reading to 2 decimal places

Procedure

1 Weigh out 1.31g of salicylic acid (approximately 0.01mol)

2 Add the salicylic acid to 3.0 cm^3 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 hearting 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.