

EXPERIMENT Y1
PURIFICATION OF ACETANILIDE BY CRYSTALLISATION

Introduction

This experiment introduces the technique of recrystallisation, which is one of the most important methods of purifying solid compounds, and the use of melting points as a means of identifying compounds and checking their purity. Read the section on 'Doing the Experiments' and the accompanying 'Notes on Recrystallisation' before starting. The experiment requires approximately 1 lab period. Read the section 'Writing Up and Assessment' before starting your write-up.

Procedure

Place the contaminated acetanilide (2 g) and a few boiling chips in a 25 ml B14 flask and add 40% aqueous methylated spirits¹ (10 ml). Fit a reflux condenser and clamp the flask securely over a tripod and gauze. Heat to gentle reflux with a small flame (CAUTION) until all of the solid dissolves. Remove the Bunsen, carefully add activated charcoal (ca. 0.2 g) (bumping may occur) and then reflux gently for ca. 2 minutes.

Filter the solution, while still hot, under gravity, using a fluted filter paper² and a pre-heated (use the oven) glass funnel, into a clean boiling tube. If some solid precipitates out on the side of the tube during the filtration, warm the boiling tube carefully to redissolve it (use a steam bath in the fume cupboard, not a Bunsen).

Cover the top of the boiling tube with foil and allow it to cool. When cool to the touch place it in an ice-water bath³ to ensure maximum recovery, and leave it there for 10 minutes.

Filter off the product under suction using a Hirsch funnel and a boiling tube with a side arm. Compact the crystals with a spatula and wash them with ice-cold 40% aqueous methylated spirits (2 x 2 ml) (see note 5 on page 45). Place the crystals in a labelled beaker and dry them in an oven at 70–80 °C.

Record the weight and the melting point⁴ of the recrystallised product. Calculate the percentage recovery of acetanilide. Hand in your sample in a correctly labelled sample tube.

Questions

- 1) What is the function of the activated charcoal?
- 2) What is the structure of acetanilide?
- 3) Why is it better to quote a melting point range rather than an average melting temperature?

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- 1 Prepare for yourself using tapwater. Deionised water is not required in this course.
 - 2 The supervisors will demonstrate how to prepare fluted filter paper.
 - 3 Use ice, from the ice-machine in the lab., with lots of water to make a very watery mixture.
 - 4 The use of the melting point apparatus will be demonstrated. Note that compounds melt over a temperature range, so you should record the temperature at which significant liquid was noticed and that at which melting was essentially complete, e.g. m.p. 125–126.5 °C (round the decimals to the nearest 0.5 °C). Compare your m.p. with a literature value and quote the source of the data.