

The preparation of 2-hydroxybenzoic acid

This resource accompanies the article **Fixing the funeral footprint** in *Education in Chemistry* which can be viewed at: rsc.li/3shy8Ph

The activity was first published as a chapter in the book **Aspirin**, which can be read in full at: rsc.li/3tWoeTq

Learning objectives

- 1 Use the following practical apparatus and techniques:
 - Water bath or electric heater for heating.
 - Quickfit® apparatus for heating under reflux.
 - Filtration under reduced pressure.
 - Safe handling of hazardous chemicals.
- 2 Understand alkaline hydrolysis of esters.
- 3 Calculate percentage yield.

This activity is suitable for 16–18 year-old learners studying the chemistry of carboxylic acids and esters. The reaction, outlined in the student sheet, is an example of alkaline hydrolysis of an ester, followed by neutralisation of the salt with a strong acid to form the carboxylic acid (see equations given on the student sheet). The activity is also an opportunity for learners to develop and assess the experimental skills associated with the apparatus and techniques listed above.

Background

The alkaline hydrolysis of esters is the basis of saponification (soap making) from natural oils and water cremation – a less environmentally harmful alternative to cremation by heat.

Both oil of wintergreen (methyl 2-hydroxybenzoate) and salicylic acid (2-hydroxybenzoic acid) are widely used as pharmaceuticals. The manufacture of aspirin from salicylic acid is of major importance. Industrially, salicylic acid is manufactured at high temperature and pressure from the phenol sodium salt and carbon dioxide, with an annual worldwide production of about 50,000 tonnes.

Health, safety and technical notes

The activity typically takes 1½ to 2 hours to complete in a school laboratory and can be carried out in two sessions as long as the reaction mixture in the flask can be stoppered and safely stored between sessions. Learners will also need a few minutes in a further session to weigh the dried product.

As with all experimental work in schools, you must carry out your own risk assessment in accordance with your employer's health and safety policy. See also the RSC's standard health and safety guidance here: rsc.li/3KS5qK2.

The notes that follow give some guidance regarding the apparatus and chemicals listed on the **student sheet**. See the **technician notes** for hazards and disposal information.

Apparatus

- 10 cm³ and 25 cm³ measuring cylinders (for the oil of wintergreen and sodium hydroxide)
- Weighing balance, ± 0.01 g
- 50 cm³ pear-shaped flask fitted with a reflux condenser

Show learners how to correctly set up the Quickfit® apparatus with the water bath or electric heater and how to control the heat for steady reflux.

- Anti-bumping granules
- Water bath (eg 250 cm³ beaker) or an electric heater
- 100 cm³ beaker surrounded with ice and water in a larger container. A larger plastic beaker is suitable to contain the ice.
- Dropping pipette (to add the acid to the mixture)
- Stirring rod
- Spatula
- Buchner flask and suction apparatus

Show learners the filtration technique if they have not met it before.

- Watch glass

Chemicals

- Oil of wintergreen
- 2 mol dm⁻³ aqueous sodium hydroxide
- Concentrated hydrochloric acid
- Several pieces of litmus or universal indicator paper on a white tile
- Ice-cold water. A wash bottle containing distilled water cooled in ice is suitable.

Safety equipment

- Splashproof goggles
- Chemical resistant gloves should be available for anyone who may have a cut or graze on their hands and for all if necessary.

Answers

1. There are two layers in the flask at the start of the preparation but when the reaction is complete the mixture is homogeneous (only one layer).
2. The methanol produced remains in the filtrate because it is completely miscible (capable of mixing) with water.
3. The reaction of concentrated hydrochloric acid with the excess sodium hydroxide and the sodium salt of the salicylic acid is exothermic. The temperature also increases due to the concentrated acid dissolving. If the mixture warms up too much, more methanol evaporates, increasing the risk to health due to inhalation. Also, the salicylic acid becomes more soluble at higher temperatures, so the yield of solid will be lower.
4. Oil of wintergreen is 98% methyl 2-hydroxybenzoate (also referred to as methyl salicylate) and it shows the medicinal properties of salicylates in general. It is not usually given by mouth but is readily absorbed by the skin. It is applied as a liniment for the relief of back ache, sciatica and rheumatic conditions, as well as for minor sports injuries.
5. A good yield can be obtained in this experiment, although the product may still contain some impurities. 2.0 g of oil of wintergreen theoretically produces 1.8 g of dry 2-hydroxybenzoic acid.
6. 2-hydroxybenzoic acid has a solubility in water of 2.5 g dm^{-3} at room temperature, which increases to 77.8 g dm^{-3} at 100°C , so recrystallisation would purify it without much loss of yield. The purity could be checked by measuring it has a sharp melting point of 159°C . A less pure sample would have a lower and less sharp melting temperature.

Further investigations

- Recrystallisation of the product could be carried out as above, and the melting point of the impure and purified product compared.
- The method could be used to investigate other examples of alkaline hydrolysis – eg saponification of natural oils.
- The atom economy and environmental impact of this preparation could be compared with the industrial manufacture of salicylic acid.