Preparation of an organic liquid

A video featuring this experiment is available at https://rsc.li/38LiKx6, along with teacher notes and worksheets for learners.

Equipment (per group)

- 1 x beaker 100 cm^3 (to dispense the concentrated acid)
- 1 x measuring cylinder, 50 cm^3
- 2 x conical flask, 250 cm^3
- mineral wool for plug (or use cotton wool)
- 1 x glass pipette
- 1 x separating funnel and stopper
- 3 x retort stands, bosses and clamps
- 1 x beaker, 250 cm^3 (for collection of waste aqueous layer)
- 2 x filter funnel
- 1 x measuring cylinder, 10 cm^3
- 2 x spatula
- 1 x filter paper
- 1 x pear shaped flask, 50 cm^3 (or round bottomed flask)
- 1 x deionised water in wash bottle
- 1 x full apparatus for distillation which includes:
  - 1 x still head
  - 1 x thermometer
  - 1 x Liebig condenser
  - 1 x receiver
  - 1 x sample tube
  - 1 x beaker to use as water bath (large enough to contain the pear shaped flask and water)
- 1 x kettle
- 1 x timer or digital stopwatch

Safety equipment:

- safety googles (for first part of the experiment)
- safety spectacles
- access to a fume cupboard
- gloves (to handle the mineral wool only)

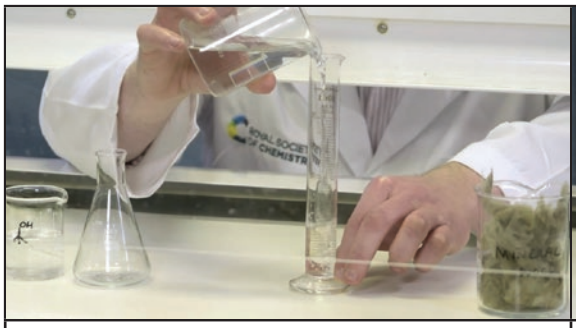
Preparation

Each group will need the following chemicals:

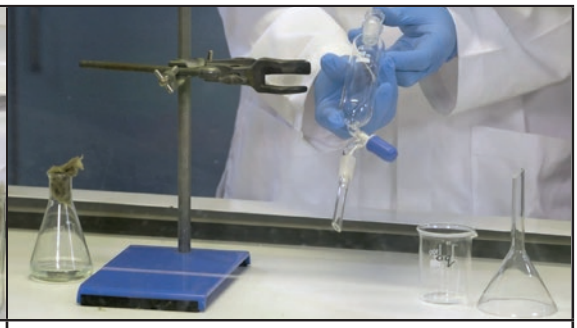
- 2-methylpropan-2-ol, 6 cm^3
- concentrated hydrochloric acid, 20 cm^3 (needs to be cold: refrigerate and supply just before use, or provide bottles kept in an ice bath)
- 5% sodium hydrogen carbonate solution, 10 cm^3
- anhydrous sodium sulfate, solid, 10 g
- anti-bumping granules, a quarter of a spatula
- 0.4 mol dm^{-3} nitric acid solution, 5 cm^3
- 0.05 mol dm^{-3} silver nitrate solution, 5 cm^3

Equipment set-up and tips

This is a complex, multi-step experiment. The video shows each stage in detail. The steps have been divided into three stages: preparation, separation and purification. Set up for each stage is shown and the time stamp for the relevant section of the video is given for easy reference.



Preparation: combining concentrated hydrochloric acid with 2-methylpropan-2-ol, see 01:08.



Separation: removing the aqueous layer using a separating funnel, see 02:07.

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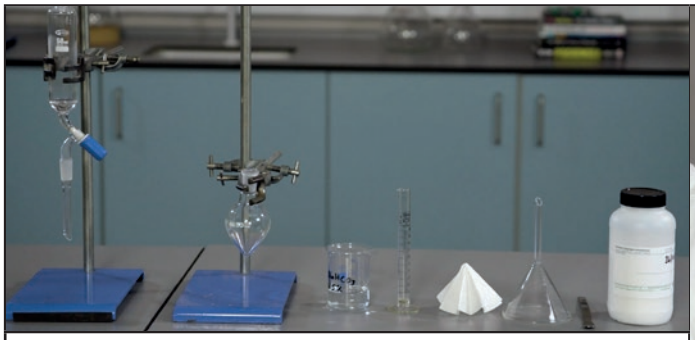

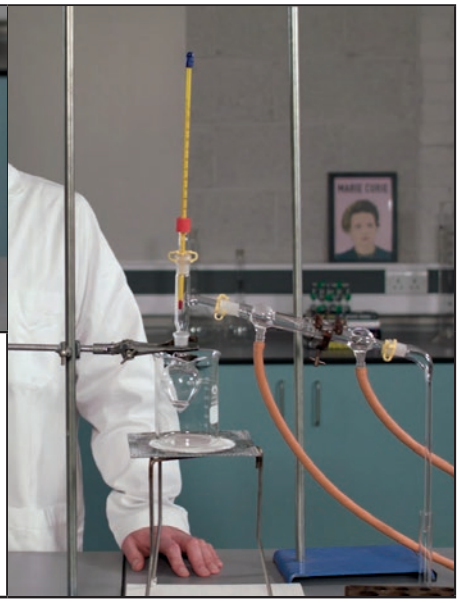

Purification: first, sodium hydrogen carbonate solution is added to remove unreacted hydrochloric acid, see 03:10. Then solid anhydrous sodium sulfate (drying agent) is added to remove any trace of water, see 04:43.

Finally, distillation separates the sample from any remaining reactant, 05:25. Use boiled water from a kettle to get to the desired temperature to collect the sample between 49–52°C.

Safety

Read our standard health & safety guidance and carry out a risk assessment before running any live practical.

Refer to SSERC/CLEAPSS Hazcards and recipe sheets.

Hazard classification may vary depending on supplier.

Chemical supplied for the practical	Preparation
2-methylpropan-2-ol $C_4H_{10}O$  **DANGER** Flammable Corrosive (eyes) Irritant (skin, respiratory) May cause drowsiness or dizziness	2-methylpropan-2-ol will be in the solid form under 25°C. To melt it, place the stock container in a plastic bag (to protect the label), loosen the lid slightly and stand in warm (not hot) water.
Hydrochloric acid Concentrated HCl (aq)  **DANGER** Corrosive (skin, eyes) Irritant (respiratory)	
Sodium hydrogen carbonate solution, 5% (w/v) $NaHCO_3$ (aq) Currently not classified as hazardous	Sodium hydrogen carbonate solid $NaHCO_3$ (s) MW= 84.01 g mol^{-1} Currently not classified as hazardous
Sodium sulfate(VI) anhydrous, solid Na_2SO_4 (s) Currently not classified as hazardous	

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Chemical produced
2-chloro-2-methylpropane C_4H_9Cl  **DANGER** Highly flammable liquid and vapour

Eye protection should be worn throughout the preparation and practical. Wear goggles/face shield when working with concentrated hydrochloric acid.

Work in a fume cupboard when using concentrated hydrochloric acid.

Keep chemicals away from fire.

Please note that CLEAPSS does not recommend wearing gloves to dispense concentrated hydrochloric acid (unless wounds or skin conditions exist) as it can be difficult to find the correct gloves and there might be a loss of dexterity. Please consult your school's health and safety advisor if you are unsure of the best measures to take to minimise any risk.

Disposal

Any unreacted concentrated hydrochloric acid would have been neutralised by the sodium hydrogen carbonate solution, but make sure equipment used to dispense it is rinsed in the fume cupboard.

The aqueous waste can be flushed away down a foul-water drain with plenty of water.

Wash separating funnels, flasks and vials with a detergent solution to emulsify traces of water-immiscible organic products. The washings from these may then also be flushed away down a foul-water drain. The organic product needs to be kept and collected by a registered waste carrier.

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