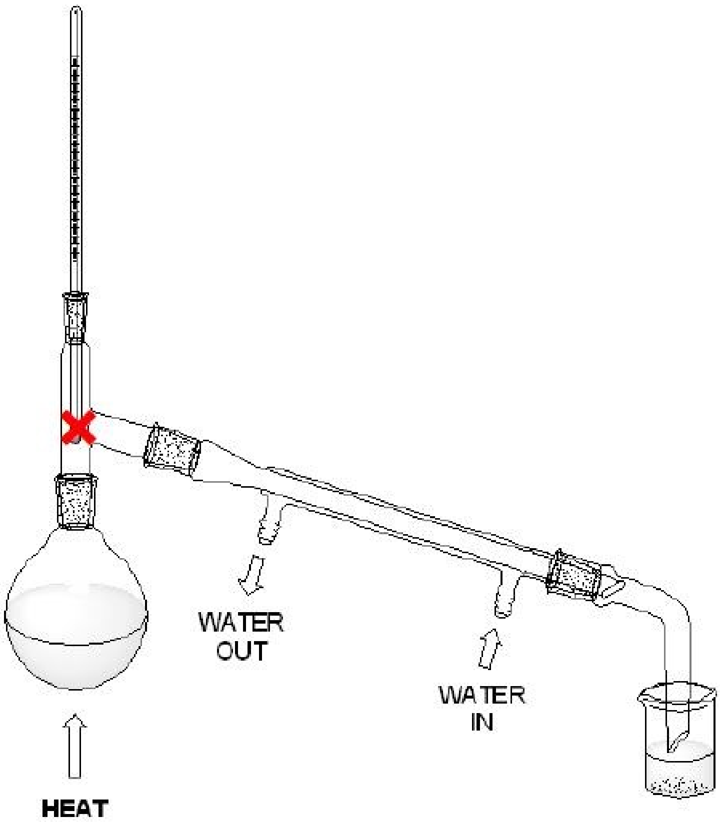
Problem 4: Alcohol detective

Teacher and Technician Pack

Pre-Lab answers

1. Key features;
- System must not be completely sealed
- System must not allow any vapour to escape before condensation
- The condenser water goes in at the bottom and out at the top
- The thermometer must be present to record the boiling point of the distillate



2.

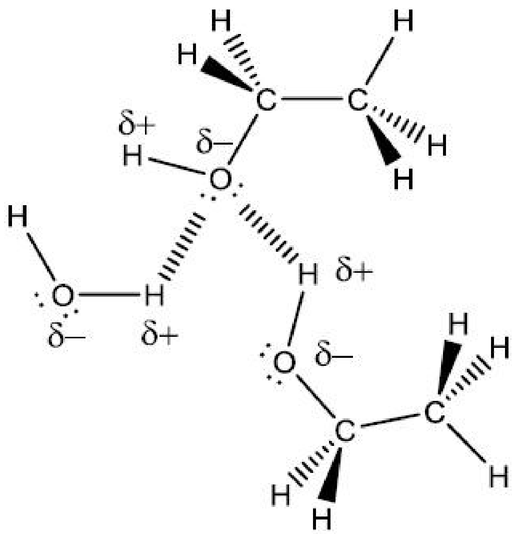
methanol	b.p. 65.1 °C
ethanol	b.p. 78.6 °C
2-methylpropan-2-ol	b.p. 82.4 °C
butan-1-ol	b.p. 117.3 °C
glycerol	b.p. 290.1 °C

The boiling points of the alcohols increase as the chain length increases. This is a result of the increased Van der Waal's forces between the chains as they get longer, meaning that more energy is needed to break them. The boiling point of 2-methylpropan-2-ol is lower than we would predict from its chain length as a result of the branching in the chain. This means the chains cannot pack together as closely as a straight chain molecule and so the Van der Waal's forces are not as effective.

The alcohols all have higher boiling points than the alkane with a similar molecular mass owing to the hydroxyl (OH) group. As a result of the hydroxyl group, hydrogen bonding occurs between the molecules. This is the strongest of the intermolecular forces and requires more energy to break.

Reference for boiling points;
Book of data, Editor R.D. Harrison, Longman Group Limited.

3. Short chain alcohols are completely miscible with water because of hydrogen bonding between the hydroxyl group and the water molecules. One possible diagram is shown below;



Key features;

- Two lone pairs shown on each oxygen atom
- δ+ and δ– shown on hydrogen and oxygen atoms respectively
- Hydrogen bonds shown between a δ+ hydrogen and a δ– oxygen atom

4. CH_3CH_2OH and $CH_3CH(OH)CH_3$

Test to distinguish between a primary alcohol and a secondary alcohol;
Heat with acidified potassium dichromate solution and distil off the product. Test the oxidation products with Tollens' reagent / Fehling's solution

- Tollens' reagent – silver mirror produced; Fehling's solution – blue to brick red
Indicates the presence of an aldehyde and so the alcohol must be primary i.e. CH3CH2OH
- Tollens' reagent – no silver mirror produced; Fehling's solution – remains blue
Indicates that the product of oxidation of the alcohol is not an aldehyde therefore the alcohol must have been secondary i.e. $CH_3CH(OH)CH_3$

$CH_3CH(OH)CH_3$ and $(CH_3)_3COH$
Test to distinguish between a secondary alcohol and a tertiary alcohol;
Heat a small quantity of each alcohol with acidified potassium dichromate solution

- $CH_3CH(OH)CH_3$ is oxidised to a ketone – solution turns from orange to green
- $(CH_3)_3COH$ as a tertiary alcohol is not oxidised – solution remains orange

CH_3OH and CH_3CH_2OH
Test to identify the presence of a $CH_3CH(OH)$ group in alcohols
The iodoform test – add a solution of iodine to the alcohol followed by a small quantity of sodium hydroxide.

- A pale yellow precipitate of iodoform is formed

Indicates the alcohol contained a $CH_3CH(OH)$ group e.g. CH_3CH_2OH

- No precipitate is formed

Indicates the alcohol does not contain a $CH_3CH(OH)$ group e.g. CH_3OH

1 mol dm^{-3} sodium hydroxide is **corrosive** and will cause severe eye damage. **Goggles** must be worn when it is in use.

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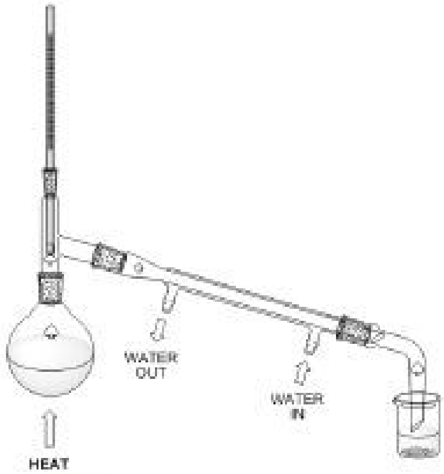
Proposed method



Using the pre-lab questions students identify that the alcohol can be extracted from both samples *via* distillation

Ethanol and *tert*-butanol are both **highly flammable**. Extreme caution should be taken to ensure that there is no escape of vapour from the distillation set-up. Anti-bumping granules must be used to ensure smooth boiling. Direct heating with a Bunsen burner should be avoided.

Students suggest an initial identity for the alcohol present based on the boiling point of the distillate

b.p. ethanol = 78 °C
b.p. *tert*-butanol = 82 °C
b.p. glycerol = 290 °C

Students confirm the presence of *tert*-butanol by adding a few drops of the alcohol to 1 cm^3 an acidified solution of potassium dichromate* [Toxic, Corrosive] and heating gently in just boiled water

ethanol – orange to green
***tert*-butanol – no change**

Students confirm the presence of ethanol using the iodoform test: 10 drops of the alcohol are placed in a test tube and 25 drops iodine solution [Low hazard] followed by 10 drops of NaOH solution [Corrosive] are added

ethanol – yellow ppt
***tert*-butanol – no reaction**

* The solution of acidified potassium dichromate can be made by placing 1 cm^3 of a 0.1 mol dm^{-3} solution of potassium dichromate(VI) **[Toxic]** in a test tube and adding a 2 mol dm^{-3} solution of sulfuric acid **[Corrosive]** until the test tube is half full.

Each group will need;

For the distillation;

- 30 cm^3 ethanol and 20 cm^3 glycerol mixed – labelled Sample A [Highly flammable]
- 30 cm^3 tert-butanol and 20 cm^3 glycerol mixed – labelled Sample B [Highly flammable, Harmful]
- Two sets of distillation apparatus; 100 cm^3 round-bottom or pear-shaped flask
- Still head
- Thermometer (0-110 ◦C)
- Thermometer adaptor
- Condenser
- Condenser tubing × 2
- Receiver adaptor
- Small beaker for collection of distillate
- Anti-bumping granules
- Clamp stand, clamp and boss x 2
- Funnel
- Heat source (either micro burner or a suitable electric heater)

For the Iodoform Test;

- Test tubes × 2
- Test tube rack
- Sodium hydroxide solution, 1 mol dm^{-3} [Corrosive – causes severe burns and eye damage]
- A solution of iodine in aqueous potassium iodide, 0.05 mol dm^{-3} [Low hazard]
- Disposable pipettes
- For the potassium dichromate test;
- Test tubes × 4
- Test tube rack
- Potassium dichromate(VI) solution, 0.1 mol dm^{-3} [Toxic]
- Sulphuric acid, 2 mol dm^{-3} [Corrosive]
- 250 cm3 beaker and access to just-boiled water
- Disposable pipettes