## OCR (A) Chemistry GCSE

## PAG 4 (chemistry) / PAG C2 (combined science): Distillation <br> Notes

## Extraction of Limonene

## Aim

To use distillation to extract limonene from orange peel.
To test for saturation.

## Equipment list

- Scissors
- Bunsen burner
- Heat resistant mat
- Clamp and stand
- Side arm boiling tube with delivery tube
- Bung with thermometer
- $250 \mathrm{~cm}^{3}$ beaker
- Test tube
- Dropping pipette
- 6 test tubes


## Chemicals required

- Bromine water
- Acidified potassium manganate(VII) solution
- Cyclohexane
- Cyclohexene
- Water
- Orange peel


## Method

## Distillation of orange oil from orange peel

1. Cut up the orange peel into small pieces and half fill the boiling tube with the side arm.
2. Add tap water to the boiling tube so that the orange peel is just covered.
3. Set up the apparatus as shown in figure 1.
4. For 5 minutes, gently heat the solution in the boiling tube using a half-blue flame, moving the flame up and down the tube. Avoid boiling the water or burning the orange.
5. After 5 minutes, heat the tube more strongly to distil off the water/orange oil mixture until the test tube is half full.
6. Record the temperature of the water/oil vapour.

## Testing the limonene in orange oil

1. Leave the distillate to stand for 2 minutes until two layers have separated to the top of the test tube.
2. Use a pipette to collect the top layer of the solution. Divide this top layer evenly into two test tubes.
3. Add a drop of bromine water to one test tube and a drop of acidified potassium manganate to the other. Record any observations.
4. Repeat step 3 with cyclohexene and cyclohexane. Record observations.

## Key points

- Controlled heating is required at the distillate stage. It should not be boiled vigorously as this could cause all the water to evaporate from the boiling tube into the test tube.
Excessive boiling may burn the orange peel.
- If a substance contains double bonds it is 'unsaturated'. When an unsaturated compound is added to bromine water or potassium manganate(VII), the solutions will decolourise.
- Cyclohexane and cyclohexene are used as positive and negative controls to show how the reagents react.


## Diagram



Figure 1 Experiment Setup
Chemix

## Safety Precautions

- Take care when using the Bunsen burner. Tie back long hair. When not in use, turn off the gas or leave on the orange safety flame.
- Clear up broken glassware or spillages immediately.
- Bromine water may cause respiratory irritation so do not stand over the solutions and ensure the room is well ventilated.
- Cyclohexane and cyclohexene cause skin irritation. Wear gloves and wash hands immediately if it comes into contact with the skin. Highly flammable so keep away from the Bunsen burner.

Analysis of results

| Sample | Observation with potassium manganate | Observation with bromine water |
| :--- | :--- | :--- |
| Limonene | Purple solution decolourises | Orange solution decolourises |
| Cyclohexane | Purple colour remains | Orange colour remains |
| Cyclohexene | Purple solution decolourises | Orange solution decolourises |

