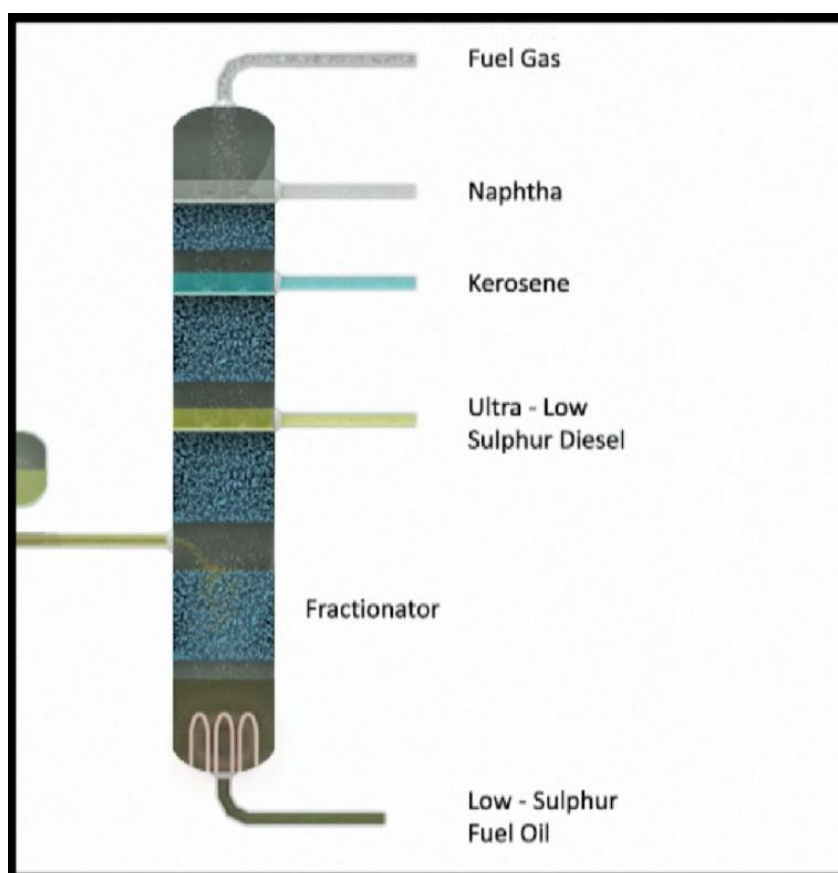


Experiment

Background

Fractional distillation is the separation of a mixture into its component parts ('fractions') by heating them to a temperature at which several components evaporate. As the temperature of crude oil increases, different hydrocarbons are separated. Those with the lowest boiling points evaporate first and those with the highest boiling points, last. A condenser is used to condense and capture each evaporated component.

In the distillation tower of the QER New Fuels Development Centre, continuous fractional distillation is performed using a large, vertical 'fractionator'. Liquid outlets at intervals up the column allow different components to be withdrawn according to their boiling point. Those with the lowest boiling points exit from the top of the column and those with the highest from the bottom.



Boiling Points of Alkanes

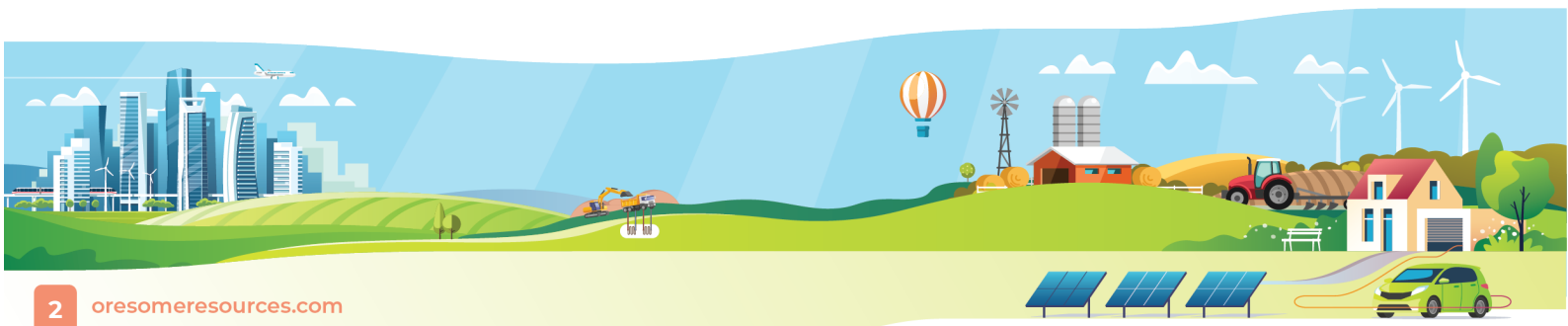
Formula	Name	Boiling Point °C
CH ₄	Methane	-161
CH ₃ CH ₃	Ethane	- 89
CH ₃ CH ₂ CH ₃	Propane	- 42
CH ₃ CH ₂ CH ₂ CH ₃	Butane	-0.5
CH ₃ CH ₂ CH ₂ CH ₂ CH ₃	Pentane	+ 36
CH ₃ (CH ₂) ₆ CH ₃	Octane	+125

Aim

In this experimental investigation you will set up and operate your own miniature petroleum refinery to perform fractional distillation on a crude oil substitute.

Equipment

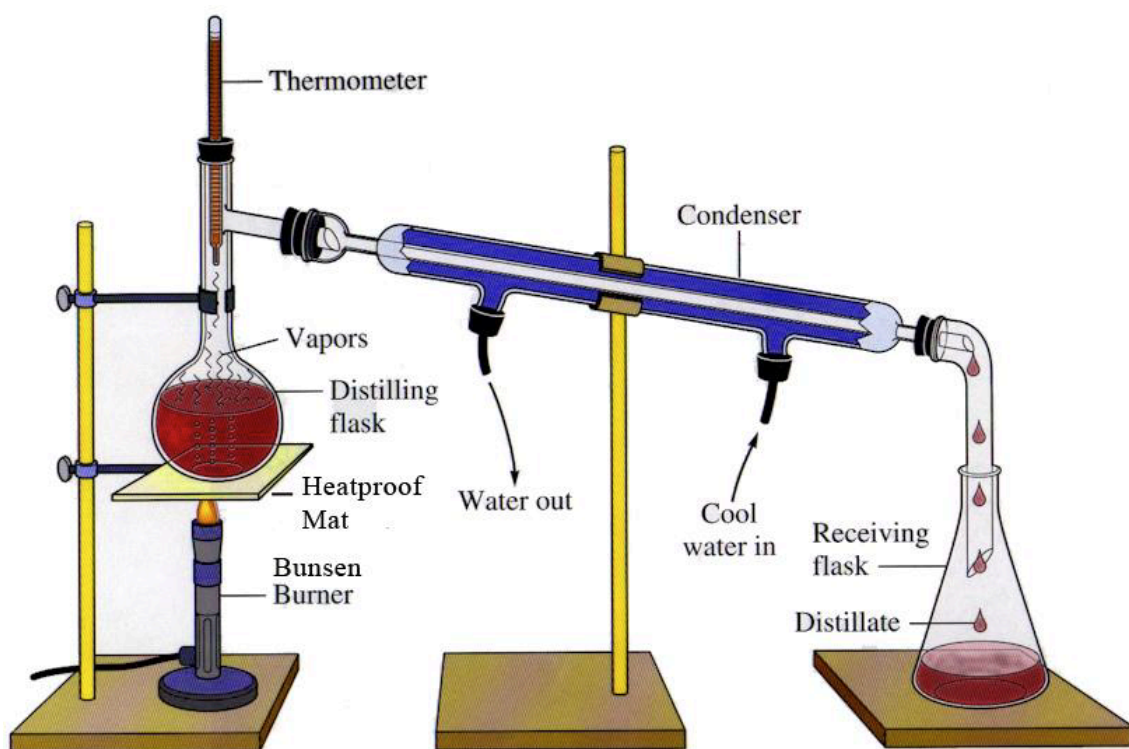
- ☐ Safety glasses and aprons
- ☐ Bunsen burner
- ☐ Heatproof mat
- ☐ Round bottom distilling flask with side arm
- ☐ Thermometer 0–360°C
- ☐ Retort stands and clamps as required to secure all apparatus
- ☐ Rubber stoppers
- ☐ Condensing tube
- ☐ Two hoses for cool water delivery to condensing tube
- ☐ Four conical receiving flasks
- ☐ Bent delivery tube
- ☐ Boiling chips
- ☐ Teat pipette for delivering crude oil substitute to distilling flask
- ☐ Four "Hard glass" (borosilicate) watch glasses
- ☐ Wooden splints



- Crude oil substitute (highly flammable and harmful), about 2cm³. It is not permitted to use real crude oil as it contains more than 0.1% benzene, which is carcinogenic. It may be desirable to add a low boiling point fraction (such as cyclohexane) to obtain a fraction below 70°C.

Note: This is a messy experiment as it can be difficult to get the apparatus clean afterwards, but it will still produce a good result if oil residues are present.

Set up



(Note: The above distillation apparatus is a common set up. This image has been adapted from http://oz.plymouth.edu/~wwf/distillation_files/image001.jpg)

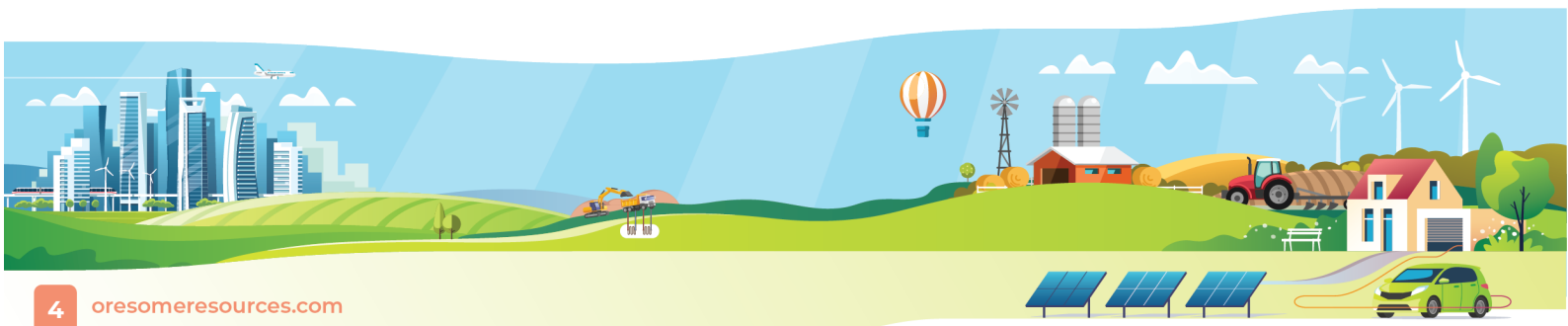


Safety

- Identify all potential hazards in this experiment, including:
- Heat – Bunsen burner and all glass components and connections.
- Glassware – Ensure all apparatus is securely supported and stable. Warm and cool all glass components gradually to avoid thermal shock.
- Stoppers – Ensure they are able to withstand the heat applied
- Hoses – Ensure all hoses are connected tightly to avoid leakage
- Use a round-bottomed rather than flat-bottomed distillation flask for smoothness of boiling
- Never more than half fill the distillation flask with the liquid to be distilled
- Use boiling chips in the distillation flask (before the heating has begun) to ensure smoothness of boiling
- Do not use a stopper to connect the condenser to the receiving flask as this will prevent vapours for escaping.

Procedure

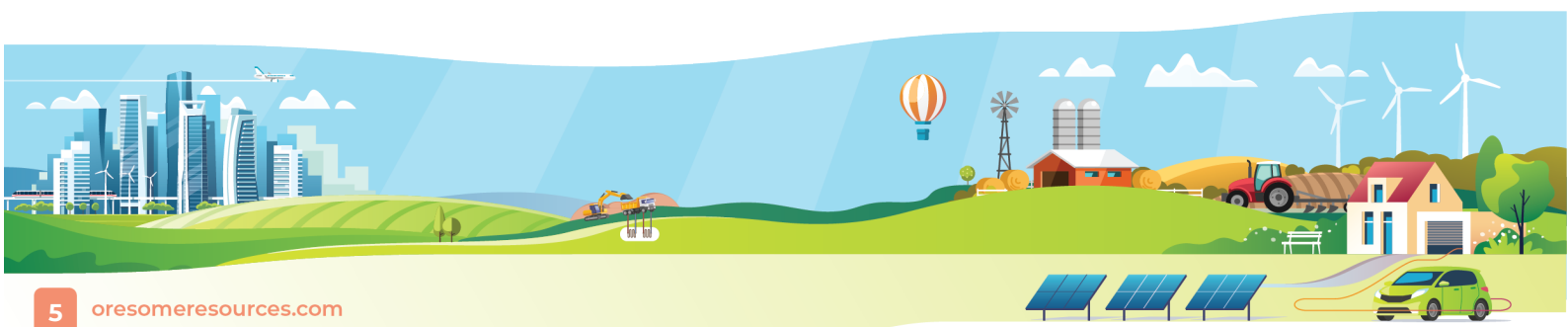
1. Familiarise yourself with the Material Safety Data Sheet for your crude oil substitute. Wear eye protection at all times.
2. Set up the apparatus as shown in the diagram. Ensure that the bulb of the thermometer is level with or just below the side arm.
3. Heat the bottom of the distilling flask gently with the lowest burner flame.
4. When the temperature reaches 100oC, remove the receiving flask. This is your first fraction. Replace the receiving flask with a clean flask.
5. Collect three further samples, to give fractions as follows:
 - a. Room temperature to 100oC
 - b. 100-150oC
 - c. 150-200oC
 - d. 200-250oC
6. Set some of each fraction aside for step 8.



7. Construct a table to record the results of each of the following tests on each of your four fractions:
 - a. Record the colour
 - b. Test for viscosity (how easily do they pour?)
 - c. Test the smell (gently waft the smell toward you with your hand and describe the smell. Liken them to familiar aromas)
 - d. Test for flammability. Pour a small sample onto a hard watch glass and light the fraction with a burning splint. Record how easily it ignites, how quickly it burns and how much smoke is produced.
8. With the samples that you set aside, combine them to see that they form a mixture very like the original sample (be sure to include some of the black residue left in the distilling flask).

Questions and Conclusions

1. In fractional distillation, what is the purpose of the condenser?
2. Why is it important that the bulb of the thermometer is level with or just below the side arm?
3. Compare crude oil fractions with higher boiling points to those with lower boiling points and answer these questions:
 - a. Which is darker in colour?
 - b. Which is more viscous?
 - c. Which is more difficult to ignite and burn (i.e. which has the higher 'flash point')?



- d. Which has more carbon atoms?
 - e. Which has higher molecular weights?
 - f. Which has more branched chain alkanes?
4. Explain why molecules with more carbon atoms, higher molecular weights and more branched chain alkanes have different boiling points to those with fewer.
5. Suggest ways in which your fractional distillation experiment could be made more efficient with regard to:
- a. Amounts of resources consumed (energy, water, etc)
 - b. Volumes of fractions captured.
6. Outline the advantages of industrial distillation columns, such as that at the QER New Fuels Development Centre, over your laboratory fractional distillation procedure.

QER is acknowledged for their assistance in the preparation of this resource.

