Organic Chemistry

Petrochemistry Properties of petrochemical products LD Chemistry Leaflets

C2.3.3.1

Boiling range distribution of petroleum ether

Aims of the experiment

- To observe and understand a boiling range distribution (distillation)
- To determine the important points on a boiling curve of petroleum ether
- To draw conclusions about the composition of petroleum ether
- To recognise the connection between structure, chemical compounds and the properties as a fuel resulting from these

Principles

Petrol is a fuel whose combustion energy is immediately converted into mechanical work in combustion engines (petrol engines).

Fuels are hydrocarbons with various and different long, straight, branched or ring-like hydrocarbon chains. Relatively short-chain molecules are better able to turn into gas and, as a consequence, are required for better cold-starting properties of the engine. Long-chain molecules, on the other hand, are richer in energy and help to reduce fuel consumption.

The composition of petroleum ether can vary greatly with regard to these hydrocarbons and therefore the boiling points can be very different. Conclusions can therefore be drawn about the composition and the performance of a fuel in an engine via the boiling point curve of the petrol. Here, the petrol fractions are divided into various subgroups, so-called special boiling point solvents. For example, petroleum ether boils between 40 and 70 °C and normal petroleum between 65 and 90 °C.

To determine the boiling point of a liquid, it is heated in a distillation apparatus. Here, the liquid initially evaporates on the surface and changes from the liquid into the gas phase. When after some time the vapour pressure of the liquid is equal to the surrounding pressure, the whole of the liquid can evaporate. The liquid begins to boil. When this situation occurs, the boiling temperature of the liquid has been reached. This is measured in the rising vapour. This is important, as the vapour at this point is at a temperature which just prevents it from condensing. It is in equilibrium with the boiling liquid. In the distillation apparatus, this vapour is converted



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again into the liquid phase via the cooler and can be channelled off.

In this experiment, the liquid being investigated is petroleum ether. Based on the recorded boiling curve, conclusions can be drawn about its composition. For this, the boiling point is measured in relation to the volume of liquid distilled off. As the sample volume is 100 mL, the quantity of distilled liquid collected can be immediately read off in volume % and the important boiling points for characterisation of the petroleum ether can be determined.

Risk assessment

Petrol is highly flammable! Therefore extinguish all flames! As superheating (bumping) is possible, be sure to use boiling stones. Perform the experiment in a fume cupboard and do not breathe in the vapours.

Petrol	
	Hazard statements
¥3	H225 Highly flammable liquid and vapour.
	H304 May be fatal if is swallowed and enters the airways.
JH.	H315 Causes skin irritation.
	H336 May cause drowsiness or dizziness.
	H411 Toxic to aquatic life with long- lasting effects.
	H361f Suspected of damaging fertility.
	H373 Causes damage to organs through prolonged or repeated exposure.
	H302 Harmful if swallowed.
Hazard	H410 Very toxic to aquatic life with long-lasting effects.
	Precautionary statements
	P101 If medical advice is needed, have product container or label at hand.
	P102 Keep out of reach of children.
	P103 Read label before use.
	P210 Keep away from heat. No smok- ing.
	P260 Do not breathe vapours/spray.
	P262 Do not get in eyes, on skin, or on clothing.
	P243 Take precautionary measures against static discharge.
	P301+ P330 + P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.
	P403+P233 Store in a well ventilated place. Keep container tightly closed.

Equipment and chemicals

1	Pocket-CASSY 2 Bluetooth
1	CASSY Lab 2
1	NiCr-Ni adapter S, type K524 0673
1	Temperature probe, NiCr-Ni, 1.5 mm, type K529 676
1	Claisen distillation bridge 250 mm
1	Round-bottom flask, 250 mL, NS 19/26664 301
1	Support ring for round-bottom flask, 250 mL667 072
1	Joint clip, plastic, NS 19/26
2	PVC tubing 7 mm diam., 1 m604 501
2	Hose clamp 812 mm604 460
1	Measuring cylinder 100 mL, with plastic base 665 754
1	Protective sleeve for temperature probes666 194
1	Heating mantle 250 mL, adjustable
1	Laborboy II (laboratory jack-stand)
2	Adhesive magnetic board 500 mm666 4659
2	Holder, magnetic, size 2, 1114 mm
2	Holder, magnetic, size 3, 1822 mm666 4663
1	Panel frame C50, two-level, for CPS666 425
1	Boiling stones 100 g661 091
1	Petroleum ether, 90110 °C, 250 mL670 8200
1	Petroleum ether, 100140 °C, 500 mL670 8210
1	Stopcock grease, 60 g661 082
41	so required:
1	PC with Windows XP/Vista/7/8
41	so necessary for wireless measurement:
1	Battery for Pocket-CASSY 2 Bluetooth524 019
1	Bluetooth dongle524 0031

Construction and preparation of the experiment

Construction of the apparatus

The distillation apparatus consisting of a Claisen distillation bridge, a 250 mL flask, a 100 mL measuring cylinder, a temperature probe with protective sleeve, a laboratory jack-stand and a heating mantle is set up on the adhesive magnetic board (see Fig. 1). Make sure that all glass connections are adequately greased with stopcock grease. Secure the glass connections additionally with a joint clip. Connect the distillation bridge to a water tap in reverse flow using tubing and hose clips. The temperature probe, which should not touch the inner wall of the distillation apparatus, is connected to a computer using the temperature adapter S and the Pocket CASSY. Install the measuring cylinder so the distillate runs down the inner wall, as this makes it easier to read off the volume.

Performing the experiment

1. Load CASSY Lab 2 settings.

2. Remove the flask, place it onto the support ring and, using the measuring cylinder, fill it with 100 mL of petroleum ether (e.g. 90 - 110 $^{\circ}$ C) and 3 - 4 boiling stones. Then locate the heating mantle under the filled flask by raising it on the laboratory jack-stand.

Note: Petrol from the filling station can also be used.

3. Open the water tap slightly to cool the distillation bridge.

4. Switch on the heating mantle to "maximum" and heat up the petroleum ether. It should boil steadily. Should a dangerous situation occur, the heat source can be quickly removed using the laboratory jack-stand.

5. When boiling starts, record the temperature at which the first drop of distillate falls into the measuring cylinder by pressing the button on the Pocket CASSY or O in the software. If necessary, adjust the speed of the distillation to a

maximum of 3 drops/second by reducing the temperature (to "minimum") or by lowering the laboratory jack-stand.

6. Then immediately record the boiling temperature each time a further 5 mL of distillate have been collected, i.e. have passed into the measuring cylinder.

7. As soon as 95 mL of distillate has been collected, stop the heating by switching off and lowering the heating mantle. The measurement is now completed and can be repeated with another petroleum ether.

Observation

By heating the petroleum ether, the liquid transfers from the liquid phase to the gas phase during distillation. When the hot vapour meets the cold glass of the flask, it condenses and small droplets of liquid form. The longer the liquid is heated, the more vapour is formed and condenses on the glass of the flask. The liquid droplets increase in size and run back into the petroleum ether. With time, the hot vapour rises even higher as the glass of the flask and the vapour become hotter.

After a while, the vapour condenses on the protective glass sleeve and heats up the temperature probe. This increase in temperature is recorded. It corresponds to the boiling point of the petroleum ether. The vapour then flows into the Claisen distillation bridge where it condenses owing to the cooling water, and is thus converted into the liquid phase and flows into the measuring cylinder.

Evaluation

The evaluation is performed in CASSY Lab 2. A diagram has been prepared for this. In the diagram "Boiling point", the temperature profile of the distillation is considered (Fig. 2). The boiling point of the petroleum ether increases steadily.



Fig. 1: Boiling curves of different petroleum ethers. Red: Petroleum ether 90 - 110 °C, black: Petroleum ether 100 - 140 °C

As the sample volume is 100 mL, the quantity of distilled liquid collected can be immediately read off in volume %. The associated boiling points can be determined by placing horizontal marks through the 10 vol %, 50 vol % and 90 vol %

values (open the context menu by right-clicking in the diagram, $+ \rightarrow$ place marks, $- \rightarrow$ horizontal line), or by reading directly from the table (Table1).

Гаb.	1:	Important	boiling	point	temperatures	of	petroleum	ether.
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Petroleum	Boiling point (°C)					
ether	10 vol %	50 vol %	90 vol %			
90 - 110 °C	86.3	91.9	98.4			
100 - 140 °C	90.6	111.9	126.8			

Results

The lower the boiling point of the 10 vol % point, i.e. the point at which 10 % of the petroleum ether has evaporated, the more volatile components the petroleum ether contains. If the proportion is too great, this can lead to vapour bubble formation in the fuel line when it is hot, or to ice formation in the carburettor when it is cold. A high boiling point of the 50 vol % fraction means that the petroleum ether has a large proportion of non-volatile components. These components can, among other things, cause incomplete combustion in the cylinders of the petrol engine and thus poor continuous power output. The average boiling points of the 50 vol % fraction and the 90 vol % fraction of the petroleum ether are an indication of its volatility (Table 2).

Tab. 2: Average boiling points of different petroleum ethers.

Petroleum ether	Average boiling point (°C)
90 - 110 °C	(91.9 °C + 98.4 °C)/2 = 95.2 °C
100 - 140 °C	(111.9 °C + 126.8 °C)/2 = 119.4 °C

The winter fuel available at the fuel station is characterised by a lower average boiling point in comparison with summer fuel due to its lower boiling range curve.

As the 10 vol % boiling point of petroleum ether 90 - 110 °C is lower compared to petroleum ether 100 - 140 °C, this indicates that more volatile components are present, i.e. more short-chain hydrocarbon molecules. Petroleum ether 100 -140 °C, on the other hand, contains a larger proportion of high boiling point molecules, which can also be observed by the higher 50 vol % boiling point. Petroleum ether 90 - 110 °C is characterised by the average boiling point 96.7 °C and petroleum ether 100 - 140 °C by the average boiling point 121.3 °C.

Other petrol ethers can be investigated in the same ways and compared on the basis of their boiling curves.

Cleaning and disposal

Under a cupboard with extraction system collect the distillate and remaining liquids in appropriately labelled brown glass reagent bottles with warning labels and ventilated stoppers in the event that further experiments are to be carried out. Alternatively, dispose of the liquids in a container for waterimmiscible, halogen-free organic liquids.