Modellversuch zur fraktionierten Erdöldestillation

(Item No.: P1308600)

Curricular Relevance

Area of Expertise: Chemie

Education Level: Klasse 7-10

Topic: Organische Chemie

Subtopic: Kohlenwasserstoffe und Petrochemie

Experiment:

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Intermediate	10 Minutes	20 Minutes	1 Student

Additional Requirements:

Experiment Variations:

Keywords:

Principle and equipment

Principle

Petroleum (crude oil, order no. 31801.50) differs greatly in its composition according to its place of origin. The content of low boiling point hydrocarbons, which is low from the start, decreases further by evaporation during transport and storage. Because of this, petroleum is subjected to a conversion (for example, to cracking) in refineries prior to distillation, i.e. long chain and high boiling point components are partly split to hydrocarbons with low and medium boiling points. Crude oil is therefore usually not suitable for demonstrative experiments. It can foam up strongly when heated (the Stutzer attachment serves here as a foam breaker) and does not contain enough low boiling point components. A “model petroleum” is to be preferred. In this case, the following mixture was used (if required, a litle powdered carbon can be added as colouration):

33 % by volume *n*-pentane 31707.25 (65 mL)
33 % by volume *n*-hexane 31369.25 (65 mL)
33 % by volume *n*-heptane 31366.25 (65 mL)

The figures in brackets indicate the volumes required for a filling of the flask. Values given relate to this model petroleum. The following mixture is an alternative, and resembles petroleum more closely:

50% by volume	Crude oil	31801.50 (90 mL)
30% by volume	Benzine, boiling point 100...140°C	30037.50 (55 mL)
20 % by volume	Petroleum benzin, boiling point 50...70°C	31711.50 (35 mL)

Robert-Bosch-Breite 10
D - 37079 Göttingen
Tel: +49 551 604 - 0
Fax: +49 551 604 - 107
info@phywe.de
www.phywe.com

Student's Sheet

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Equipment

Position No.	Material	Order No.	Quantity
1	Temperature meter digital, 4-2	13617-93	1
2	Bubble tray column, model, with 2 trays	35914-15	1
3	Heating mantle f. roundbottom flask, 250ml	49542-93	1
4	Temp.probe, imm., PT100, -20...+300°C	11759-04	4
5	Frame for complete experiments	45500-00	2
6	Power regulator	32288-93	1
7	Condenser, Dimroth type GL25/12	35815-15	1
8	Shelf with hanging device	45505-00	1
9	Safety experimentation trough	39181-00	1
10	Panel for complete experimental setups	45510-00	1
11	Stutzer attachment GL25/12	35791-15	1
12	Round bottom flask, 100ml, GL25/12, GL18/8	35842-15	1
13	Round bottom flask, 250 ml, 2-neck, GL25/12, GL18/8	35843-15	1
14	Apparatus holder, variable	45526-00	1
15	Distilling bridge GL18/8	35902-15	1
16	Quartz glass wool 10 g	31773-03	1
17	Rear-cover for compl.-exp. panel	45501-00	1
18	Clamping holder,18-25mm	45520-00	4
19	Activated carbon, granular 500 g	30011-50	1
20	Clamp for heating mantle	49557-01	1
21	n-hexane 250 ml	31369-25	1
22	n-heptane, extra pure 250 ml	31366-25	1
23	n-pentane 250 ml	31707-25	1
24	Tube Coupling, d = 8 mm	47521-00	2
25	Spring plugs, 50 off	45530-00	1
26	Boiling beads, 200 g	36937-20	1
27	G-clamp	02014-00	2
28	Tweezers,straight,blunt, 200 mm	40955-00	1
29	Calcium chloride tube,str.150 mm	36941-00	1
30	Porcelain dish, 75ml, d = 80 mm	32516-00	3
31	Fixing bands,universal,100 pcs.	45535-00	1
32	Erlenmeyer flask,narrow n., 50 ml	36117-00	2
33	Graduated cylinder 100 ml	36629-00	1
34	Funnel, glass, top dia. 80 mm	34459-00	1
35	Pipette with rubber bulb	64701-00	1
36	Rubber tubing, i.d. 6 mm	39282-00	4
37	Hose clip, diam. 8-16 mm, 1 pc.	40996-02	6
38	Hose clip f.12-20 diameter tube	40995-00	1

Safety information

n-pentane, *n*-hexane und *n*-heptane are colourless, water insoluble, highly or easily imflammable, health-hazardous volatile liquids, that are lighter than water. Their vapours are heavier than air and form an explosive mixture with air. There is a danger that electrostatic charges could cause ignition. Do not inhale vapours. Avoid contact with eyes and skin. Wear protective clothing, protective gloves and protective goggles when working with them. Observe the detailed information on safety measures in the appendix.

Set-up and procedure

Set-up

Position the clamping holders on the panel for complete experiments as shown in Fig. 2. The equipment is to be subsequently assembled and fixed to the clamping holders as shown in Fig. 1. The drying tube which is to be connected to the receiver flask must first be filled with activated charcoal, then plugs of quartz glass wool be inserted into each end, so that activated charcoal cannot fall out. The activated charcoal is used to prevent teachers and students from being exposed to the smell of petroleum during the experiment. Connect up the tubings for the water flow through the cooling jacket. Mount two quick-connect hose couplings in this arrangement in order to have the possibility of quick disconnection from the water tap (Fig. 1). Use hose clips to secure all connections against slippage. Fix the tubings to the frame with fixing band.

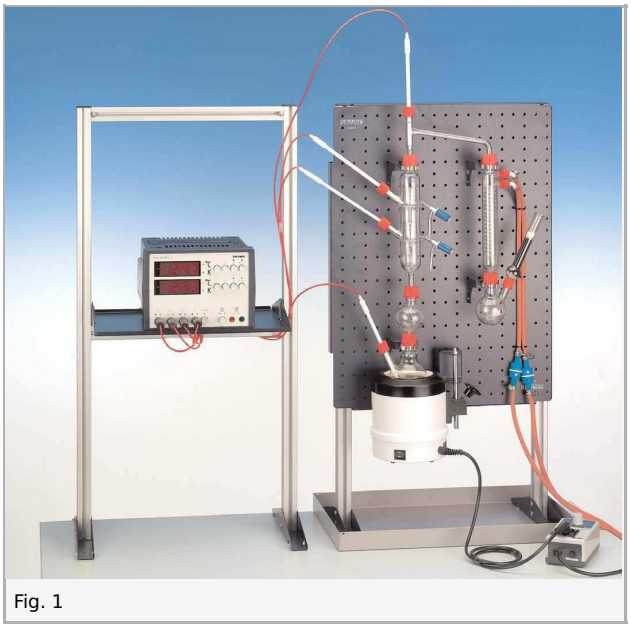

Fig. 1

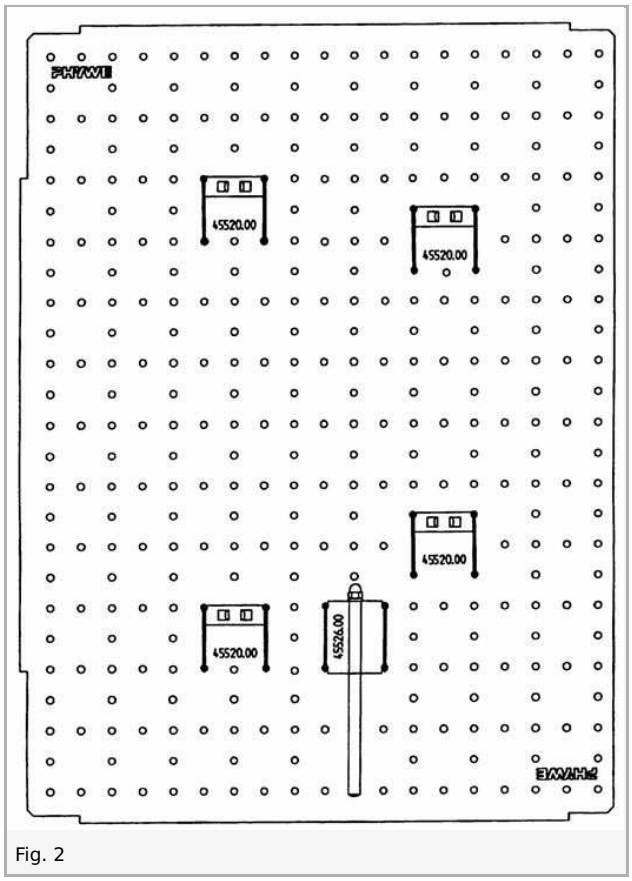

Fig. 2

Variations

1. When complete refluxing is wanted:

This variation can be selected to obtain an improved separation efficiency of the two trays. The following materials are not required in this case (and no further materials):

Clamping holder, d = 18...25 mm	45520.00 1
Temperature probe, immersion type, Teflon Pt100	11759.04 1
Round bottom flask, two-necked with side neck angled, DURAN, 100 ml	35842.15 1
Distilling bridge DURAN, 160 mm	35902.15 1
Stutzer attachment, GL 25/12	35791.15 1
Drying tube, l = 200 mm	36941.00 1
Quartz glass wool, 10 g	31773.03 1
Activated charcoal, granulated, 500 g	30011.50 1

Position the clamping holders as shown in Fig. 4. Assemble the equipment as shown in Fig. 3 and fix it to the clamping holders.

Fill the 250 ml round bottom flask and heat it as previously described, but without subsequent re-adjustment of the power. Follow the temperature the whole time and note it at suitable time intervals. Electronic recording with a computer is also

Robert-Bosch-Breite 10
D - 37079 Göttingen
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info@phywe.de
www.phywe.com

possible here. The model petroleum begins to boil within about 10 minutes. Soon afterwards, the first liquid condenses in the lower column tray.After about 20 minutes there is also condensate in the upper tray. After a further 10 minutes, equilibrium is attained. The temperature in the flask is measured to be about 62°C. Distillate is at a temperature of about 41°C in the lower tray and about 37°C in the upper tray. At the high initial temperatures (step 10 is used throughout), there are at first large differences in temperature between the plates (up to 10°C), but these relativize after a longer waiting time. The separation efficiency of the bubble tray column (number of plates) can be determined with this distillation without removal (complete refluxing).

2. With computer supported recording of temperatures:

Temperatures measured with the temperature probe and digital instrument during distillation can be automatically recorded with a computer. The additional requirements for this are:

Data cable, 2 x SUB-D, 9 pin	14602.00	1
Software, temperature measuring instrument 4-2	14405.61	1
Computer		

3. With thermometers instead of digital meter:

Temperature measurements during distillation can naturally also be made with four thermometers instead of with the temperature probe and digital instrument.The following are then additionally required:

Laboratory thermometer, -10...+150°C	38058.00	4
and the following are no longer required:		
Frame for complete experiments	45500.00	1
Shelf with hanging device	45505.00	1
G-clamp	02014.00	2
Temperature probe, immersion type, Teflon Pt100	11759.04	4
Digital thermometer 4-2	13617.93	1

Fig. 3

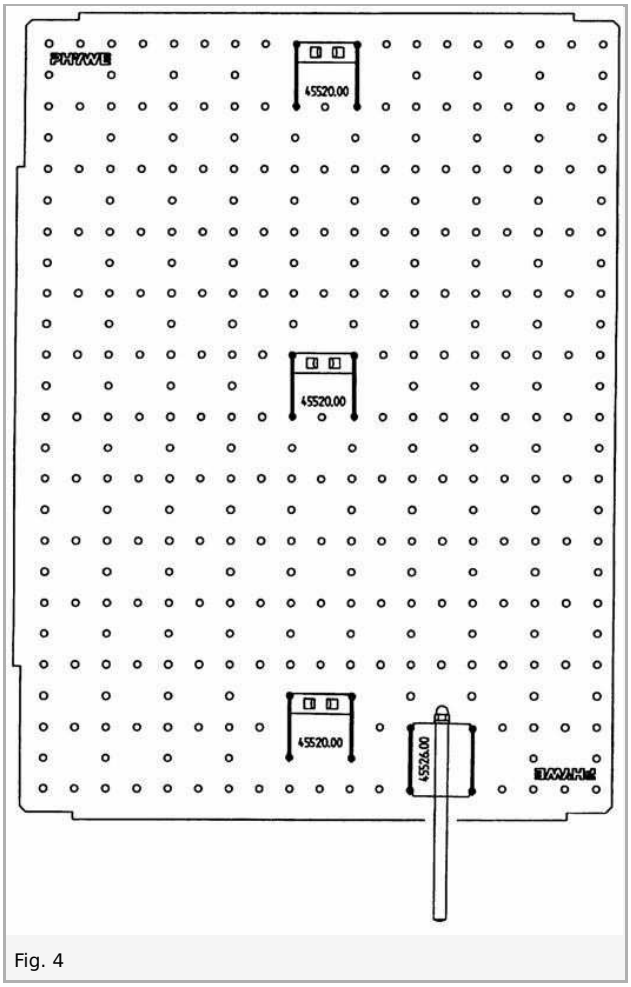

Fig. 4

Procedure

Fill the 250 ml round bottom flask about two thirds full with a mixture of n-pentane, n-hexane and n-heptane (1:1:1, “model petroleum”) and add a few boiling stones. Adjust the heating mantle to maximum heating and set the highest step (10) on the power regulator until the first bubbles in the liquid show that it is beginning to boil, then reduce the power to step 9. Reduce the power somewhat further as the amount of liquid in the round bottom flask gets less. Follow the temperature the whole time and note it at suitable time intervals. Ideally, connect the measuring instrument to a computer and so digitally record the temperature (see below).

As soon as sufficient distillate has condensed in the receiver flask – both trays should be saturated – then all three samples can be examined gas chromatographically and/or a burning test be carried out by pouring small amounts of the samples into separate evaporating dishes and setting light to them (caution!). The top fraction burns almost free of soot, whereas the two fractions from the bubble tray column have a distinct tendency to soot formation. The gas chromatographic examination of the individual fractions can be carried out analogously to the method described in experiment KV 1.21.

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Observation and evaluation

Observation

The petroleum begins to boil within about 10 minutes. Soon afterwards, the first liquid condenses in the lower column tray. After about 20 minutes there is also condensate in the upper tray. During this time, liquid is also collected in the receiver flask.

After a further 20 minutes, the temperature probes show the following values: At a temperature of 87°C in the flask, top distillation takes place at between 61°C and 62°C. Distillate is taken from the upper tray at a temperature of between 68°C and 69°C, whereas removal from the lower tray is at between 72°C and 73°C.

Evaluation

Petroleum is a mixture of hydrocarbons of various chain lengths and different boiling points. The components can be separated from each other by distillation. This succeeds more easily and better the greater the differences in the boiling points. The separating effect can be distinctly increased by repeated successive distillations of the preceding condensates. This is exactly what continuously occurs in a fractionating column. The temperature differences in the trays clearly shows the separation of the petroleum into different components.The components with the lowest boiling points collect in the receiver flask, those with somewhat higher boiling points in the upper tray, and those with even higher boiling points in the lower tray.

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