Fractional distillation with the bubble tray column with CobraSMARTsense

Students examine the fractional distillation of alcanes with a bubbly tray column.

Chemistry > Industrial Chemistry > Petrochemistry

Difficulty level	Group size	Preparation time	Execution time
medium	2	20 minutes	45+ minutes

This content can also be found online at:

http://localhost:1337/c/60f0113a6890780004f09caf

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General information

Application

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In distillation, a mixture of substances with several components is present that is to be separated.

The components are separated by heating to certain temperatures (at which a certain component evaporates), analogous to any distillation.

Fractional distillation is a separation of a mixture of substances into its constituents. Since the components are also called fractions, this type of distillation is also called fractional.

In contrast to (simple) distillation, more than two constituents are present in the mixture of substances.

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Other information (1/2)

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Prior knowledge

The students must be familiar with basics of thermodynamics (enthalpy, conservation of energy) and fundamentals of distillation.

Scientific principle

Distillation separates a mixture of liquids based on their different boiling temperatures. In fractional distillation, the bubbly tray column has an additional separating capacity.

An equilibrium between liquid and gaseous phase is established on each tray and separtes the different components.

Other information (2/2)

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Learning objective

Students learn

- the principal of a fractional distillation (seperation of a mixture with more than two components)
- every tray of a bubble tray column has a significant separtion effect during a distillation.

Tasks

Students determine:

- the mode of operation of a fractionating tower on a two-stage bubble tray column.
- Distil a mixture of three n-alcanes first with total reflux and then without any reflux.
- Subsequently, examine and compare the initial mixture and the head products

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Safety instructions

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For this experiment the general instructions for safe experimentation in science lessons apply. Wear personal protective equipment.

When handling chemicals, you should wear suitable protective gloves, safety goggles, and suitable clothing. Please refer to the appendix for detailed safety instructions.

For H- and P-phrases please consult the safety data sheet of the respective chemical.

Theory (1/3)

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Fractional distillation of crude oil

The separation of hydrocarbon mixtures is a fundamental procdure of the petrochemical industry. The more widely separated the boiling points, the better the success of the separation. The separation effect can be improved if further distillations, in which the condensate of the respective previous distillation is again vaporised and condensed, follow the first stage of distillation (vaporisation - condensation).

This process is performed continuously in industry and in the laboratory in fractionating columns.These columns can be realised as bubble tray or packed distillation columns. Bubble tray columns allow the continuous removal of condensate from arbitrary separation steps; packed columns are less complex and thus less expensive.

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Theory (2/3)

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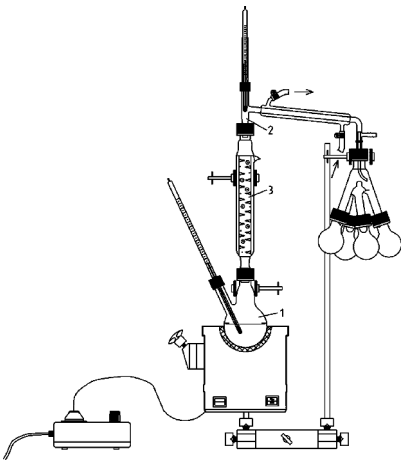

Typical distillation

During distillation the liquid first evaporates in the flask and gets from the liquid to the gas phase.

In the distillation apparatus, the vapor is then transferred back to the liquid phase via the condenser. The liquid that forms can be collected in a vessel.

The column in this experiment has two trays, which means that two fractions can be obtained. These will be mixed fractions.

The fractions can be examined via density measurement or by measuring the index of refraction with a refractometer

Theory (3/3)

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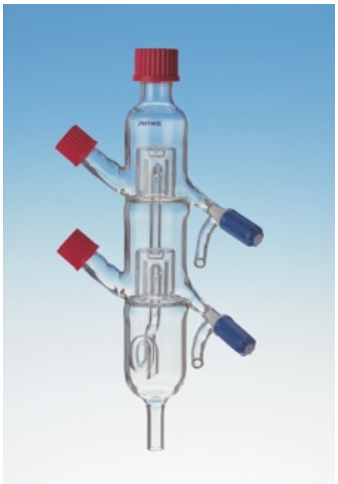

Fractional distillation

During fractional distillation, a vapor-liquid mixture is also formed in the flask. The vapor rises and enters the first bubble tray. This bubbly tray contains several passages.

The vaporized fractions rise, cool and return to the liquid state on the trays. A certain amount of a component accumulates on the bottom (equilibrium between liquid and gaseous phase).

An equilibrium between vapor and liquid phases is established on each tray.

This is the reason why bubble trays have a substantial separation effect during a distillation with total reflux.

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Equipment (1/2)

Position	Material	Item No.	Quantity
1	Cobra SMARTsense - Thermocouple, -200 ... +1200 °C (Bluetooth + USB)	12938-01	4
2	Support rod, stainless steel, 1000 mm	02034-00	2
3	measureAPP - the free measurement software for all devices and operating systems	14581-61	1
4	Immersion probe NiCr-Ni, teflon, 300 °C	13615-05	4
5	Heating mantle f. roundbottom flask, 250ml	49542-93	1
6	Clamp for heating mantle	49557-01	1
7	Power regulator, 230 V, with phase controlled modulator	32286-93	1
8	Support base DEMO	02007-55	1
9	Support rod, stainless steel, 1000 mm	02034-00	2
10	Right angle boss-head clamp	37697-00	7
11	Universal clamp	37715-01	7
12	Bubble tray column, model, with 2 trays	35914-15	1
13	Round bottom flask, 250 ml, 2-neck, GL25/12, GL18/8	35843-15	1
14	Connecting tube,IGJ 19/26-GL25/12	35800-05	1
15	Column head, with stopcock, ST 19/26	35919-01	1
16	Dimroth condenser	MAU-26525500	1
17	Cooling jacket, GL 25/8	MAU-27225000	1
18	Dropping funnel, 50 ml, Gl 18	MAU-27222000	1
19	Connecting caps,10 pcs, GL18	41230-03	1
20	Gasket for GL18, 8mm hole, 10 pcs	41240-03	1
21	Teflon sleeve IGJ 19, 10 pcs	43616-00	1
22	Clamp f.ground joint,plastic,NS19	43614-00	2
23	Rubber tubing, i.d. 6 mm	39282-00	10
24	Connecting tube,IGJ29/32-GL18/8	35678-02	1
25	Teflon sleeve IGJ 29, 10 pcs	43617-00	1
26	Funnel, glass, top dia. 50 mm	34457-00	1
27	Graduated cylinder, Borosilicate, 100 ml	36629-00	1
28	Pasteur pipettes, 250 pcs	36590-00	1
29	Rubber caps, 10 pcs	39275-03	1
30	Snap-cap vials,d=24mm,h=52mm,10p.	33621-03	1
31	Beaker, Borosilicate, tall form, 600 ml	46029-00	1
32	Glass rod, boro 3.3, l=200mm, d=5mm	40485-03	1
33	Laboratory pen, waterproof, black	38711-00	1
34	Boiling beads, 200 g	36937-20	1
35	Silicon grease Molykote, 50 g	31863-05	1
36	n-pentane 250 ml	31707-25	1
37	n-hexane,puriss. 100 ml	31369-10	1
38	n-heptane, extra pure 250 ml	31366-25	1
39	Beaker, Borosilicate, low form, 100 ml	46053-00	1

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Equipment (2/2) - main parts

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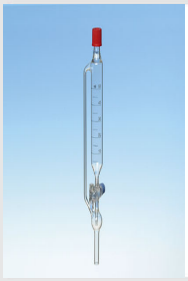
- **Bubbly tray column**: Device for the separation of a vapor-liquid mixture during distillation
- **Head column**: Device for measuring the top-temperature and the removal of the head component
- **Cooling jacket**: Device for cooling the gaseous product
- **Separation funnel:** Device for collecting the distillate after condensation in the cooling jacket

Bubble tray column

Column head

Cooling jacket



Separatin funnel

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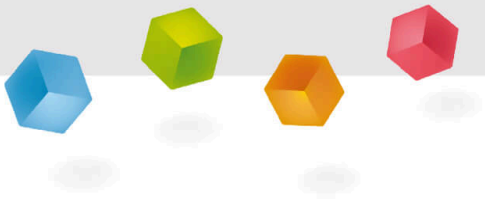

Setup and procedure

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Setup (1/5) - Download the measureAPP

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The Cobra SMARTsense and the measureAPP are required to measure the temperature value. The app can be downloaded free of charge from the App Store - see below for QR codes. Check if Bluetooth is activated on your device (tablet, smartphone).

measureAPP für Android Betriebssysteme

measureAPP für iOS Betriebssysteme

measureAPP für Tablets / PCs mit Windows 10

Setup (2/5) - Setup of the bubbly tray column

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Perform the experimental set-up according to Fig. left

- Mix each 70 ml n-pentane, n-hexane and n-heptane ("oil model") in a beaker
- Fill the solution in a 250 ml roundbottom flask with the "oil model"
- Put some boiling stones in the flask
- Put two support rods in the Support base.
- Fix the Heating mantle for roundbottom flask at the left rod
- Put the bubble tray column in the flask and fix the column on the rod.

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Setup (3/5) - Setup of the condensor & funnel

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Perform the experimental set-up according to Fig. left

- Put Column head, with stopcock, in the bubble tray column
- Fix a temperature sensor in the column head (left side in the picture)
- Put the Condenser in the column head (right side in the picture)
- Fix the column head and the condensor with a clamp on the rod.
- Combine the column head with the cooling jacket
- Connect the cooling jacket and the separating funnel
- Put the 4 Cobra SMARTsense Thermocouple sensor in the the sump, tray 1, tray 2 and the head column.

Setup (4/5) - Connecting the SMARTsense

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Cobra SMARTsense - Thermocouple

Preparation:

In this experiment you will get to know the Cobra SMARTsense temperature sensor. You can record the readings with the "measure App".

Turn on the sensor and open the "measure App". Select the temperature sensor there ("SMARTsense - thermocouple").

Go to the window with the analog display (the scale with the needle). If you now heat, you can see the temperature.

The temperature is given in the unit "Celsius", after a number simply abbreviated as "°C".

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Setup (5/5) - Connecting the SMARTsense

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- Connect the four temperature probes with measureApp on yout mobile device.
- The temperature probe, which is placed in the ”sump”, should be connected to the channel ”T1”. The probe in the first tray of the column should be defined as ”T2”
- The probe in the second tray shoud be connected to the “T3” and the probe on top of the column should be connected as "T4"

You will find information on the PHYWE Webside:

Link to information

Procedure (1/3) - with (total) Reflux

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- The stopcock of the dropping funnel is closed and the stopcock of the column head should also remain closed
- Turn on the cooling and heat the ”sump” with the heating mantle.
- Adjust the heating power with the power regulator in such a manner that the mixture boils uniformly (set the regulator to approximately setting 7 to 8).
- Monitor the temperature in the ”sump”, in the two trays and in the head of the column on the screen on your tablet.
- When the mixture is boiling and the temperatures of the two trays and in the column head are constant wait at least another 30 minutes to reach the thermal equilibrium for total reflux

Stopcock at head column

Stopcock at the dropping funnel

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Procedure (2/3) - with (total) Reflux

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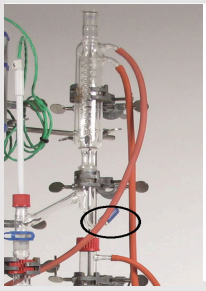
- The stopcock of the column head is to open.
- The stopcock of the funnel should remain closed.
- Wait until the funnel contains about 30 ml of liquid.
- Close now the stopcock on the column head and stop the heating.
- Put a vessel under the funnel.
- Open the stopcock on the funnel and collect about 30 ml of the liquid in the vessel.
- Use this liquid for examinations (e.g. density or index of refraction measurement)



Stopcock at head column

Stopcock at the dropping funnel

Procedure (3/3) - without Reflux

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- The stopcock of the dropping funnel is closed but the stopcock of the column head should remain open (no reflux).
- Turn on the cooling and heat the ”sump” with the heating mantle. Adjust the heating power with the power regulator in such a manner that the mixture boils uniformly (set the regulator to approximately setting 7 to 8).
- Monitor the temperature in the ”sump”, in the two trays and in the head of the column on the screen of the tablet.
- Wait until 30 ml liquid is condensed in the funnel. Close the stopcock at the column hand and stop heating.
- Use the liquid in the funnel for further examinations.

Stopcock at head column

Stopcock at the dropping funnel

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Evaluation

Evaluation (1/10) - Distillation under no reflux

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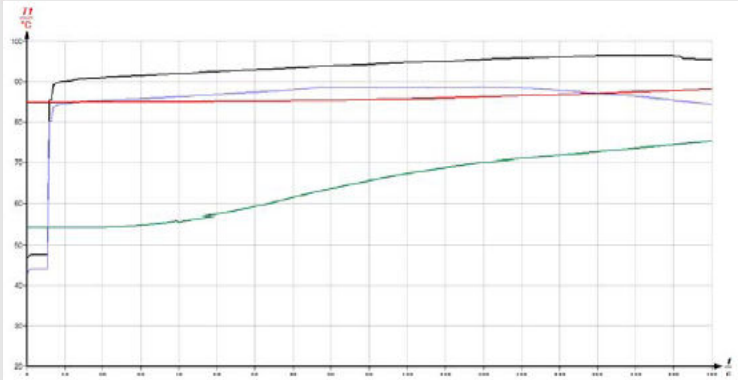

Change of the boiling points without reflux (“sump”: red line; tray 1: black line;tray 2: blue line; column head: green line).

Notes:

- During the distillation with no reflux the compositions of the mixtures are changing permanently with time respectively amount of condensate removed from the column.
- At all three places (tray 1, tray 2, column head) the amounts of the lower boiling components are decreasing and those of the higher boiling components are increasing.

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Evaluation (2/10) - Distillation under no reflux

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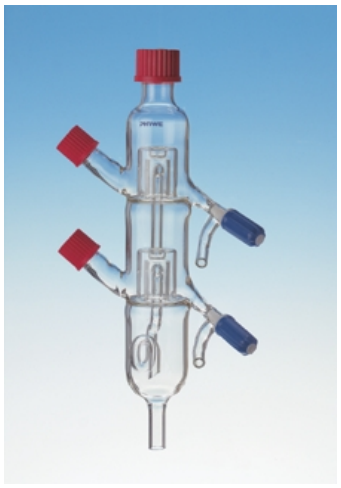
Model of a bubble tray column



- If the temperature in the distillation flask is increased, the temperature in the flask rises quickly. After a short time, the liquid begins to boil. The vapors (of the liquid) rise to the top and condense on the bottoms of the column.
- Within a few minutes of the start of boiling, vapor-liquid mixture can be seen on the first bubble tray. Some of the vapors continue to rise and thus reach the second bubble tray, where a vapor-liquid mixture also collects.
- Not all of the vapor condenses on this bubble tray either, but a portion continues to rise to the top. Here again the temperature is also determined. This temperature rises last, as the figure on the previous page shows.

Evaluation (3/10) - Distillation under no reflux

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Further evaluation methods

- You can perform a density measurement to qualitatively determine the quantity ratio of the mixtures. A hydrometer with a scale of 0.6 - 0.8 g/ml is suitable for this purpose (you need enough liquid, the hydrometer must float in the liquid. Use a large measuring cylinder for this purpose).
- For a quantitative evaluation, use a gas chromatograph. The integration of the different peaks in the chromatogram gives the quantitative ratio of the individual components

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Evaluation (4/10) - Distillation under no reflux

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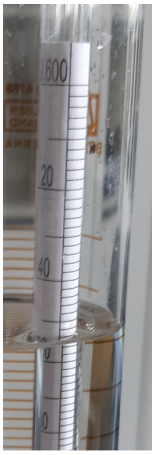
Density measurement



Density measurement with a hydrometer (with liquid in a measuring cylinder)

- Density measurment of the starting mixture: **0.655 g/ml.**
- Density calculatet: **0.656 g/ml.**

Lit. values:

C_5 of $0.626 \frac{g}{ml}$

C_6 of $0.659 \frac{g}{ml}$

C_7 of $0.684 \frac{g}{ml}$

Evaluation (5/10) - Distillation under total reflux

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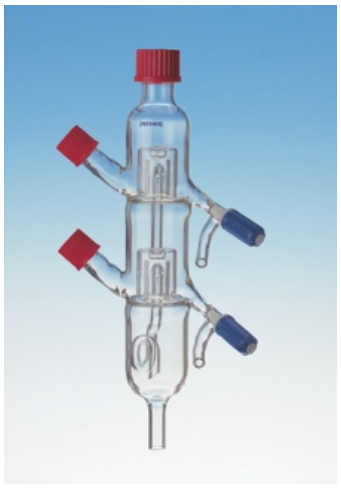
Model of a bubble tray column



- If the temperature in the distillation flask is increased, the temperature in the flask rises quickly. After a short time, the liquid begins to boil. The vapors (of the liquid) rise to the top and condense on the bottoms of the column under total reflux.
- On each tray of the column there is always an equilibrium between the vapor and liquid phases, even during the complete process of distillation. On each tray, the same amount of component passes into the gaseous state as condenses.
- Therefore, the boiling point on each tray remains constant after a certain time.

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Evaluation (6/10) - Comparison with & without reflux

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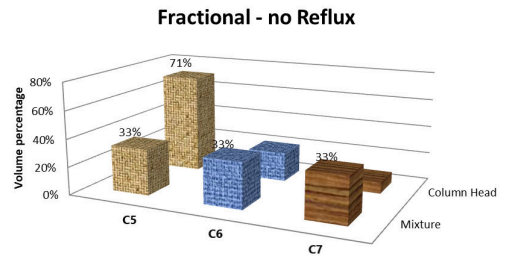
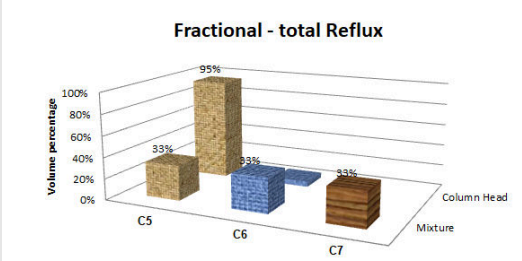
This evaluation can be performed by gaschromatography and confirms the result of the density measurement. The density measurement shows a significant increase of the C_5-fraction in the head column (in comparision with the distillation with total reflux and without reflux)



	C5	C6	C7
Mixture	33%	33%	33%
Column Head	71%	21%	8%



	C5	C6	C7
Mixture	33%	33%	33%
Column Head	95%	5%	

Evaluation (7/10) - Comparison with & without reflux

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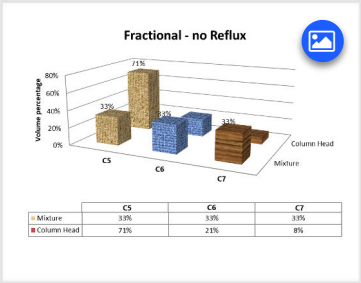
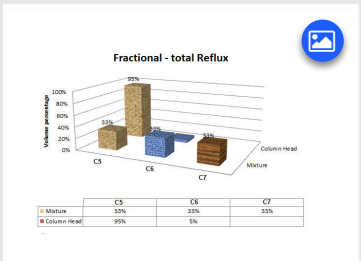


	C5	C6	C7
Mixture	33%	33%	33%
Column Head	71%	21%	8%



	C5	C6	C7
Mixture	33%	33%	33%
Column Head	95%	5%	

- **Without reflux**: n-Pentane has become the main component (around 50%) in the bottom tray (tray 1) of the column, but n-hexane and n-heptane are also present. In the column head n-pentane (around 70%) and also n-hexane and n-petane has condensed
- **With reflux**: n-Pentane has become the main component (around 50%) in the bottom tray (tray 1) of the column, but n-hexane and n-heptane are also present.
- **With reflux**: In the column head nearly pure n pentane (around 95%) with a trace of n-hexane (around 5%) has condensed
- **The evaluation shows that even with only two bubble trays a substantial separation effect can be achieved during a distillation with total reflux.**

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Evaluation (8/10) - Questions

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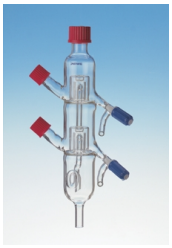

What is the advantage of a bubbly tray

- [ ] Allows to collect fractions during a distialltion
- [ ] Lowers the boilng point temperature of the components
- [ ] Significant separation effect during a distillation
- [ ] Increases the boiling point temperature of the components

Überprüfen

Evaluation (9/10) - Questions

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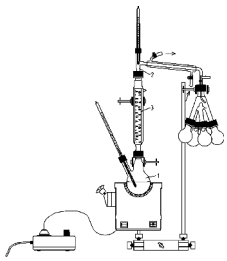

What is the component with the highest boiling point

- ○ C_5 (Pentane)
- ○ C_7 (Heptane)
- ○ C_6 (Hexane)

Überprüfen

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Evaluation (10/10) - Questions

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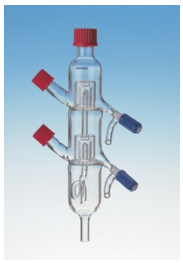

During distillation under total reflux, what is the proportion of the compontents on the bottom of tray 2 (the tray under the head colum)?

- ○ C_5 (Pentane) > C_7 (Heptane) > C_6 (Hexane)
- ○ C_5 (Pentane) < C_6 (Hexane) < C_7 (Heptane)
- ○ C_5 (Pentane) > C_6 (Hexane) > C_7 (Heptane)

Überprüfen

Slide	Score / Total
Slide 28: Advantage of a bubble tray	0/2
Slide 29: Different boiling points	0/1
Slide 30: Proportion of fractions	0/1

Total Score 0 / 4

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