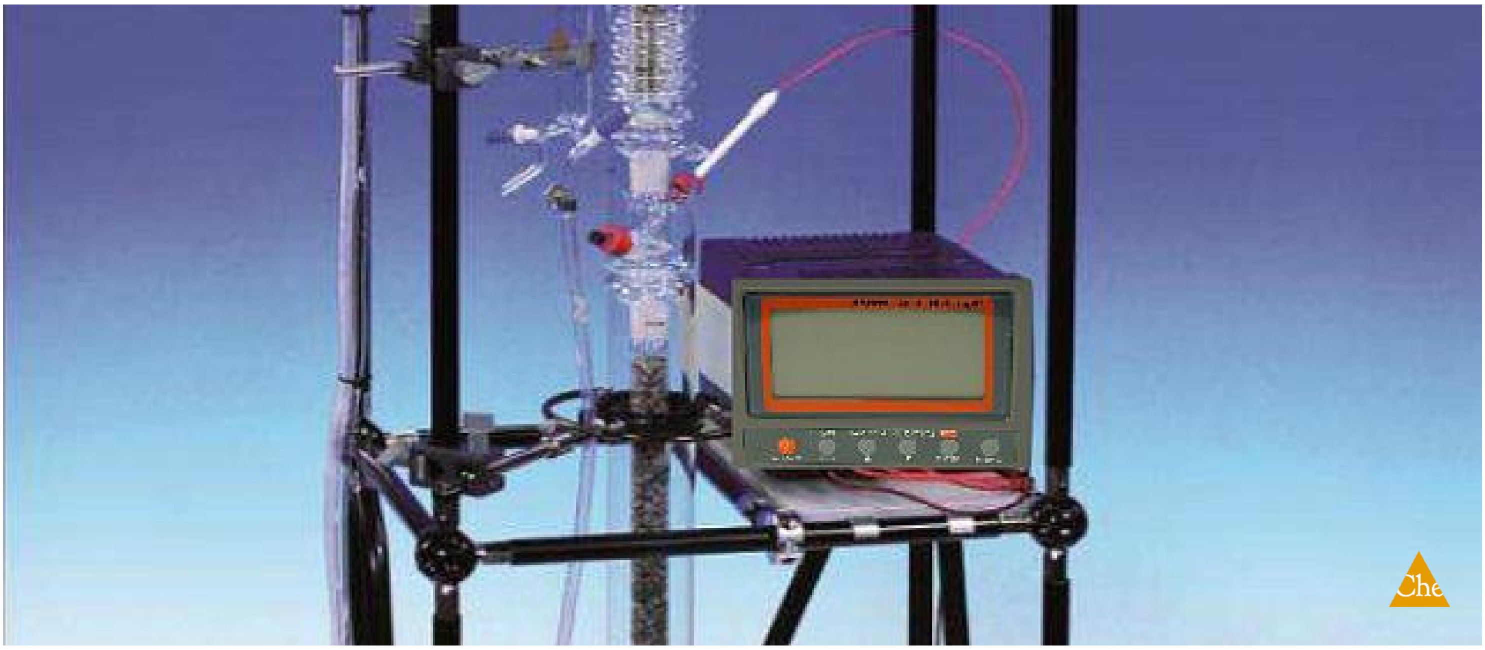
Rectification - the number of theoretical trays in a distillation column



The separation power of a rectification (fractionating) column can be determined using an appropriate binary mixture whose equilibrium composition is measured in the distillation flask and in the domed glass head of the distillation apparatus. The number of theoretical trays can be numerically or graphically obtained from the measured values.

Chemistry › Industrial Chemistry › Petrochemistry

Difficulty level	Group size	Preparation time	Execution time
medium	2	10 minutes	20 minutes

General information

Application

Refinery

Fractionating distillations with columns are widely used in the chemical industry for example in oil refinery or liquefied air separation.

This important method is visualised in this model distillation plant using transparent columns. The number of theoretical trays defines the separation power of a rectification (fractionating) column.

This value is numerically or graphically obtained from the measured equilibrium composition of a binary mixture in the distillation flask and in the domed glass head of the distillation apparatus.

Other information

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Learning objective

The number of theoretical trays, i.e. the separation power, decreases with increasing distillation rate.

If one increases the distillation rate even more, the column becomes ‘flooded’ and does not separate at all.

Tasks

- Determine the refractive indices of 10 mixtures of methyl cyclohexane and n-heptane to record a cali-bration curve.
- Determine the composition of the condensate and the number of theoretical trays in the column for a throughput of 500 and 1000 ml/h.

Safety instructions

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- For this experiment the general instructions for safe experimentation in science lessons apply.
- For H- and P-phrases please consult the safety data sheet of the respective chemical.

Theory (1/2)

Rectification is a distillation process in which, for the purpose of an improved separation, part of the condensate flows back down against the vapour and so participates in substance and heat exchange with the vapour.

The mixture to be separated is heated in an evaporating vessel until it begins to boil, increasesing the temperature continually because the composition of the mixture changesand creating a temperature gradient in the column.

This gradient ranges from a temperature below the boiling point of the substance with the highest boiling point A up to the boiling point temperature of B at the head of the column.

On evaporating the liquid, a vapour is formed which is enriched with the more volatile substance B. This vapour is then partially condensed on the the first column tray, as this is at a lower temperature.

Theory (2/2)

This liberates condensation enthalpy, which heats the condensate present there. New vapour is evolved, which is again enriched with B and flows to the second tray, on which the same procedure is repeated.

An exchange of energy (condensation enthalpy, enthalpy of vaporization) takes place at each tray and, alongside this, an exchange of substances.

We are viewing the behaviour under the assumptions that we have „ideal trays“ and „total refluxing“, whereby an ideal tray is to be understood to be one at which the backflow of liquid is in equilibrium with the rising vapour.

With total refluxing, no distillate is removed from the top of the column. All condensate returns back down the column.

Equipment

Position	Material	Item No.	Quantity
1	Abbe refractometer	35912-00	1
2	Set rectification plant, 230 V	35918-88	1
3	Immersion thermostat Alpha A, 230 V	08493-93	1
4	External circulation set for thermostat Alpha A	08493-02	1
5	Bath for thermostat, makrolon	08487-02	1
6	Rubber tubing, i.d. 6 mm	39282-00	4
7	Rubber caps, 10 pcs	39275-03	1
8	Pasteur pipettes, 250 pcs	36590-00	1
9	Snap-cap vials,d=30mm,h=50mm,10p.	33624-03	2
10	Graduated cylinder, Borosilicate, 1000 ml	36632-00	1
11	Funnel, glass, top dia.150 mm	34461-00	1
12	Stop clock, demo.; diam. 13 cm	03075-00	1
13	Boiling beads, 200 g	36937-20	1
14	HEPTANE, NORMAL 1000 ML	31366-70	1
15	Methylcyclohexane 1 l	31566-70	3
16	Water, distilled 5 l	31246-81	2
17	Temperature meter digital, 4-2	13618-00	1
18	Immersion probe NiCr-Ni, teflon, 300 °C	13615-05	4
19	Universal power supply, 600mA 3/4.5/5/6/7.5/9/12V, incl. 9 adaptors	11078-99	1

Equipment

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Rectification Plant

(incl. Data acquisitation set for set rectification plant)

35918-88
(+ 35918-50)

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Installation Manual

Equipment

1	Rack-system f.distillationsapp.	35917-00
1	Destillierkolben, 4 x NS 29, 6000 ml	35873-00
1	Heating mantle for roundbottom flask, 6000 ml	49546-93
1	Power regulator	32288-93
2	Packed column, without silvering, h = 400 mm	35917-01
2	Column intermediate piece, without silvering	35917-02
1	Top head of column with high-efficiency condenser, without silvering	35917-03
1	Plunge pipe with PTFE-valve	35917-04
1	Sampling device with 2 PTFE-valves	35917-05
1	Liebig condensor with vacuum connection, GL 25	35917-06
1	Distillation receiver with 2 PTFE-valves, 50 ml	35917-07
1	Wire mesh rings with cross-piece, steel, 185 g	35917-20
2	Column support	35917-30
1	Teflon sleeve IGJ 29, 10 pcs	43617-00
2	Connecting tube,IGJ29/32-GL18/8	35678-02
1	Connecting tube,IGJ29/32-GL25/12	35678-12
2	Stand tube	02060-00
6	Hose clamp for 5-12 mm diameter	40997-00
2	Hose clamp for 10-17 mm diameter	40998-00
1	Pipettor	36592-00
12	PVC tubing, i.d. 7mm	03985-00
1	Sheath for temp. probe, glass	11759-03
1	Syringe 10ml, Luer, 10 pcs	02590-03
1	Cannula 0.6x60 mm, Luer, 20 pcs	02599-04
4	Clamp for ground joint, plastic, IGJ29	43615-00
1	Closure caps,10, GL18	41220-03
1	Fixing bands,universal,100 pcs.	45535-00
1	Temperature meter digital, 4-2	13617-93
1	Softw. temperature meter 4-2	14405-61
1	Data cable, plug/socket, 9 pole	14602-00
1	Temp. probe, imm., Pt100, stainless steel	11759-01
3	Temp.probe, imm., PT100, -20...+300°C, teflon	11759-04

Fig. 1: 35918-88, rectification plant

Usage of rectification plant set

- The setup of the rectification apparatus is extensively discribed in the "User Manual"
- You will find this manual on the PHYWE Webside:

  Link to the manual

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Setup and procedure

Setup

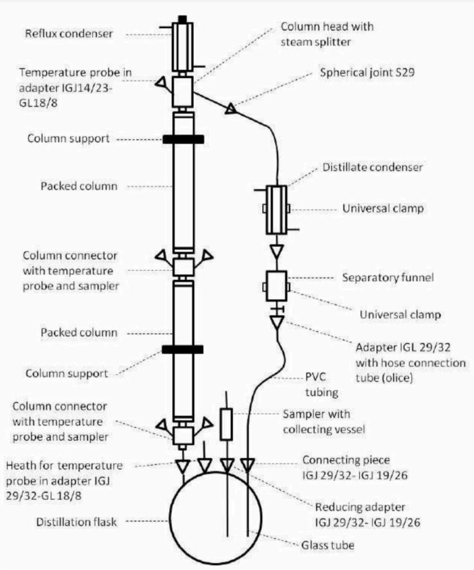
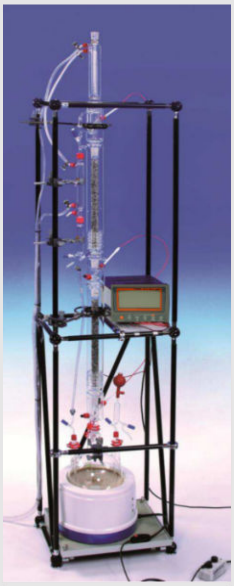


Fig. 1

Performing the measurements:

- Set up the experiment as shown in Fig. 1
- Add further quantities of methyl cyclohexane and n-heptane to the combined mixtures in the distillation flask, so that 750 ml of n-heptane and 3000 ml of methyl cyclohexane are present in the flask. Put in a few boiling beads.
- Adjust the glass tube for the recirculation of condensate from the separatory funnel in such a manner that it ends at least 5 cm below the level of the liquid in the flask, so that vapour cannot flow up through it.

Procedure (1/4)



Check the correctness and tightness of the apparatus, turn on the cooling water, and set the steam splitter to reflux.

Set the heating hood to III and the power regulator to 8. At the onset of condensation in the reflux condenser, turn the power regulator setting down to 3.5.

After two to three hours of distillation under total reflux, switch the steam splitter over to withdrawal and collect the distillate in the separatory funnel. Use the stop watch to determine the time required for 10 ml of condensate to collect and convert this to ml/h. The value should lie between 400 and 600 ml/h. If readjustment is necessary, wait for 30 minutes at total reflux before taking the next sample.

When the desired throughput has been achieved, switch back to total reflux for 30 minutes until thermal equilibrium has again established. Subsequent to this, begin taking samples of the condensate.

Procedure (2/4)

To determine the composition of the condensate you can use an refractometer.

Drop the liquid to be tested onto the surface of the prism, then cover the incident prism and lock with the knob.

Verify that the liquid layer is homogeneous, without bubbles and has been spread over the whole field of view.

Before you can examine the condensate you need to know the refractive indices of different mixtures of Methylcyclohexane and Heptane.

Procedure (3/4)

Plot the refractive indices against their mole fractions.

The respective compositions of the mixtures can be graphically or numerically determined from the slope of the regression line (Fig. 2).

Do not discard the mixtures; pour all of them into the distillation flask.

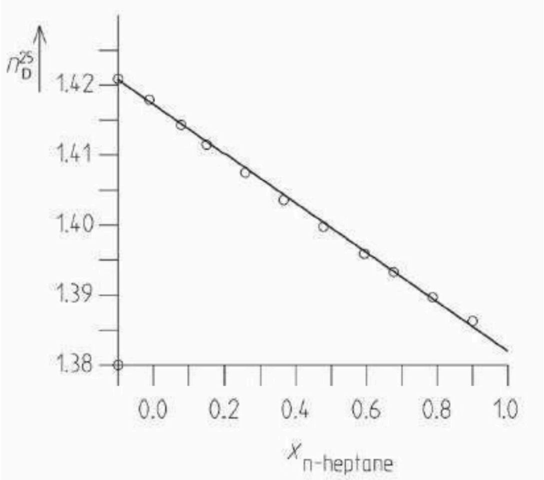
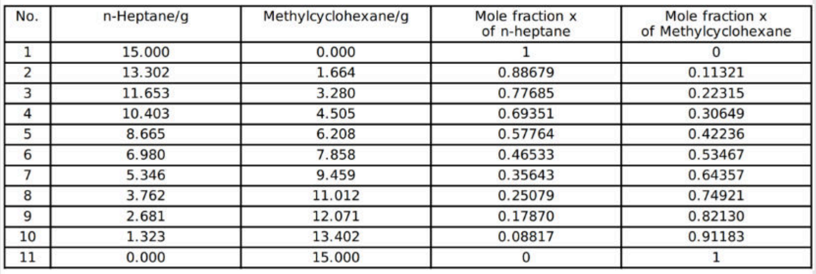


Figure 2: Calibration curve

Procedure (4/4)



No.	n-Heptane/g	Methylcyclohexane/g	Mole fraction x of n-heptane	Mole fraction x of Methylcyclohexane
1	15.000	0.000	1	0
2	13.302	1.664	0.88679	0.11321
3	11.653	3.280	0.77685	0.22315
4	10.403	4.505	0.69351	0.30649
5	8.665	6.208	0.57764	0.42236
6	6.980	7.858	0.46533	0.53467
7	5.346	9.459	0.35643	0.64357
8	3.762	11.012	0.25079	0.74921
9	2.681	12.071	0.17870	0.82130
10	1.323	13.402	0.08817	0.91183
11	0.000	15.000	0	1

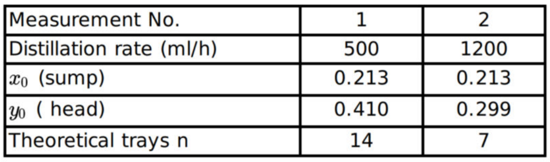
Table 1: Mixtures for the calibration curve

Recording the calibration curve:

1. Connect the refractometer to the immersion thermostat and temperature equilibrate it to 25 °C.
2. Prepare the 10 mixtures of methyl cyclohexane and n-heptane with substance ratios (mole fractions) from 0 to 1 and with step width of approximately 0.1 as listed in Table 1 and immediately measure the refractive indices.

Evaluation

Evaluation (1/10)



Measurement No.	1	2
Distillation rate (ml/h)	500	1200
x_0 (sump)	0.213	0.213
y_0 (head)	0.410	0.299
Theoretical trays n	14	7

Table 2: Measurement results

The results for an exemplary measurement are presented in Table 2.

The number of theoretical trays, i.e. the separation power, decreases with increasing distillation rate.

If one increases the distillation rate even more, the column becomes ‘flooded’ and does not separate at all.

Evaluation (2/10)

For the partial pressure p(A) and p(B) of two components A and B in the vapour space above a liquid binary mixture and with ideal behaviour, the following is valid according to Raoult’s law:

$$p_A = P_A \cdot x_A \, p_B = P_B \cdot x_B \qquad (1)$$

where p(A) and p(B) are the vapour pressures of the pure components, and x(A) and x(B) the mole fractions in the liquid phase. For a binary mixture the following is valid:

$$x_B = 1 - x_A \qquad (2)$$

and the ratio of the partial pressures in the vapour space can be formulated as:

$$\frac{p_A}{p_B} = \frac{P_A}{P_B} \cdot \frac{x_A}{1 - x_A} \qquad (3)$$

Evaluation (3/10)

According to Dalton’s law, the partial pressures p(A) and p(B) with the total pressure p are linked over the mole fractions y(a) and y(B) of the two components in the gas phase:

$$p_A = p \cdot y_A \tag{4.1}$$

$$p_B = p \cdot y_B = p\,(1 - y_A) \tag{4.2}$$

By substituting in (3) the following is obtained:

$$\frac{y_A}{1 - y_A} = \frac{P_A}{P_B} \cdot \frac{x_A}{1 - x_A} \tag{5}$$

If no indices are given, x and y refer to the more volatile components.

Evaluation (4/10)

For the relationship of the vapour pressures of the pure components, the relative volatility is used:

$$\alpha = \frac{P_A}{P_B} \tag{6}$$

Equation (5) now becomes

$$\frac{y_A}{1 - y_A} = \alpha \cdot \frac{x_A}{1 - x_A} \tag{7}$$

and after rearranging

$$\mathrm{y} = \frac{\alpha \cdot \mathrm{x}}{1 + (\alpha - 1) \cdot \mathrm{x}} \tag{8}$$

This equation can be used to construct an equilibrium diagram (mole fraction y of the more volatile component in the gas phase against the mole fraction x in the liquid phase; Fig. 4, next slide).

Evaluation (5/10)

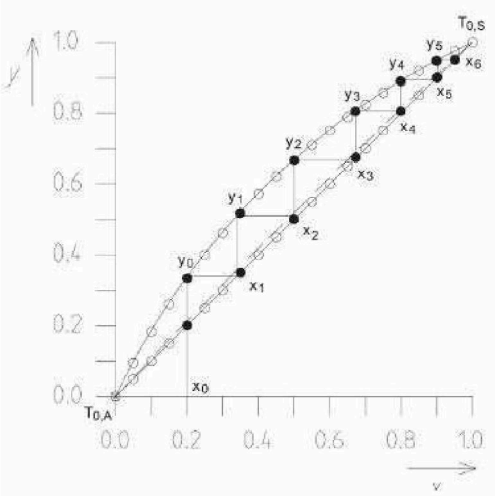


Fig. 4: Equilibrium diagram

From the isobaric equilibrium curve we can read off the exchange of substance at the individual trays.

The initial mixture contains x(0) of B. On boiling, a vapour with y(0) of B is formed. This vapour is completely condensed, and no change in the composition is caused. In the diagram we reach the bisector of the angle.

The condensate is again vaporized and the composition is now y(1). We draw in a horizontal line for each condensation, and a vertical line for each vaporization, and so obtain a series of steps.

Evaluation (6/10)

For alpha = 1, a 45° line is obtained. Under these conditions distillative separation of the mixture is not possible. If alpha > 1, curves are obtained. The upper curve in Fig. 4 corresponds to a value of alpha = 2.

If one proceeds on the assumption that the more volatile component in the liquid mixture has a mole fraction of 0.2, one can read off the composition of the vapour by moving from the 45° line perpendicularly upwards to the equilibrium curve (y(B) = 0.33). When this vapour is recondensed, move parallel to the abscissa until the 45° line is intersected. The component with the mole fraction 0.33 (complete condensation) is present in the condensate. For renewed vaporisation and condensation, a value of 0.48 is obtained, etc. Each step corresponds to a distillation procedure or a tray in an ideal bubble tray column.

The greater the reflux ratio, i.e. the greater the amount of condensate which can flow back through the column, the better the separation effect. The separation power in a technical column is expressed by its number of theoretical trays. It is defined as the number of individual distillation procedures required to achieve the same enrichment of the more volatile component in the test mixture as by rectification with the column to be investigated.

Evaluation (7/10)

The calculation of the number of theoretical trays can be performed using equation (8).

A vapour of concentration y(0) is obtained when a mixture y(0) vaporises. If the condensation is complete, the new liquid phase has the same concentration, x(1) = y(0) . After n repetitions of the vaporisation and condensation process, a mixture of concentration x(n) = y(n-1) is obtained. As the composition of the mixture in the sump remains nearly constant during total reflux, the separation effect is increasingly intensified.

$$\alpha^n = \frac{y_n\,(1 - x_0)}{x_0\,(1 - y_0)} \tag{9}$$

y(n) corresponds to the mole fraction of the more volatile component in the column head and x(0) to the mole fraction in the liquid in the distillation flask. These values can be determined from the refractive index.

Evaluation (8/10)

The relative volatility (separation ratio) alpha is 1.07 for a mixture of methyl cyclohexane and n-heptane.

The number of theoretical trays can be calculated taking the logarithm of (6). Graphically, the number of theoretical trays can be determined by plotting the curve in Fig. 4 for a relative volatility of 1.07 (broken line).

Compared with a relative volatility of 2.0, a substantially larger number of separation steps is required to reach the same final concentration for the same initial concentration.

Evaluation (9/10)

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What is Rectification?

- *Rectification* is a form of fractioning, coarser than fractional distillation
- *Rectification* is a form of Oxidation
- *Rectification* is a form of combustion
- *Rectification* is a form of electrochemical reaction

Evaluation (10/10)

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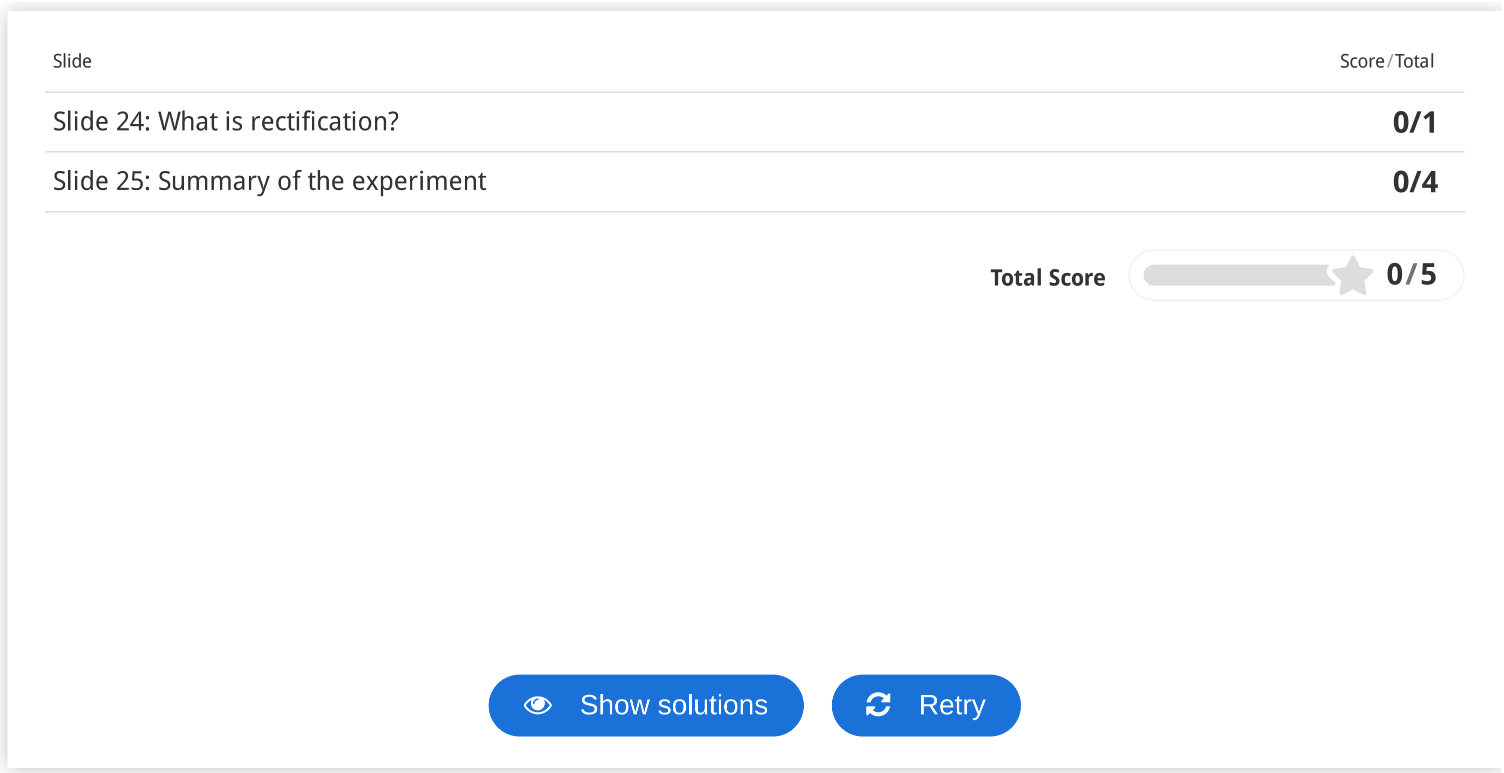
Summary of the experiment

Rectification is a ______ process in which, for the purpose of an improved separation, part of the ______ flows back down against the vapour and so participates in substance and heat exchange with the ______.
The number of theoretical trays defines the ______ power of a rectification (fractionating) column.

- separation
- distillation
- condensate
- vapour

✓ Überprüfen



Slide	Score / Total
Slide 24: What is rectification?	**0/1**
Slide 25: Summary of the experiment	**0/4**

Total Score 0/5