

SIEVE TRAY DISTILLATION COLUMN

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1. GENERAL

Only continuous distillation (or stationary distillation) is considered here. Batch distillation, which is dependent of time, is not involved in this work. Distillation as a continuous and industrial unit operation occurs usually in a single device, which is called a distillation column. Sometimes several columns are used and they can be connected to each other in many ways.

Distillation as a unit operation means separating components from each other. Separation is based on different vapor pressures of components. In figure 1 there is a simplified scheme of distillation process in one column.

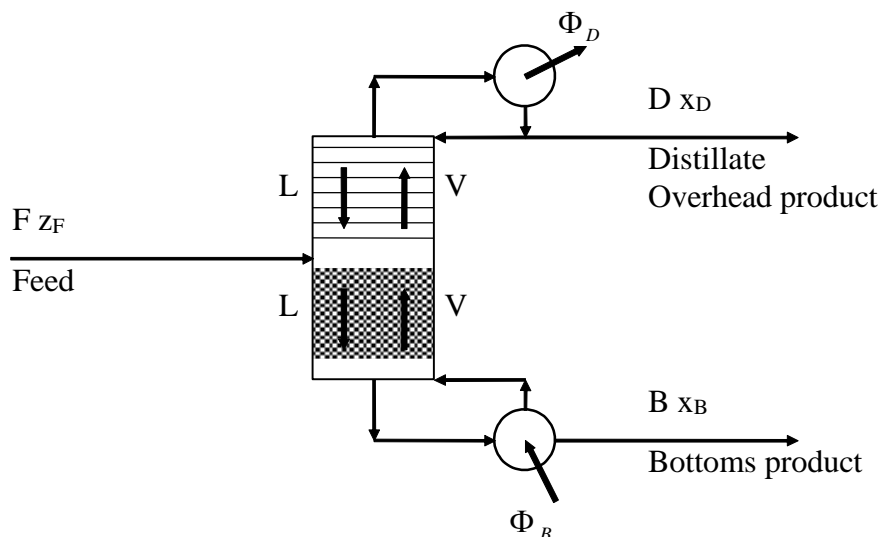


Figure 1. Simplified scheme of distillation process.

In practice continuous distillation occurs in distillation columns where vapor phase V vaporized in a reboiler by adding a heat flux Φ_B goes upwards and liquid phase L (from condenser by subtracting heat flux Φ_D) goes downwards. The column is not empty, there are usually either distillation trays or packing in order to increase the contact area between the two phases. In figure 1 there is an unlikely case where the enriching section is a tray column and stripping section is a packed column.

Phases on the same level in a column have different concentrations, which differ from the equilibrium concentration and therefore the components transfer between the two phases (actually due to their chemical potentials).

2. THEORY

The results of this work is analyzed with the simulation program Aspen Plus. The unit model used is the rigorous multicomponent distillation model Radfrac.

2.1 GENERAL

Most of the distillation processes are so called multicomponent systems i.e. the number of components is three or higher. These processes can be simulated by process simulators like the one used in this exercise, Aspen Plus. The process simulator can solve a large set of equations and unknowns evolving from the processes including many separation stages and components. Also the high non-ideality of the models describing phase equilibrium and the energy properties of the components motivates the use of simulators. It should be kept in mind that the result of the simulation is depend-

ent on the selections of adequate models for thermophysical properties, the models of the unit operations and the solution technique. If these are selected properly, the simulation is useful, otherwise not.

The modeling of a distillation stage is usually done with vertically stacked models of separation stages. The Figure beneath illustrates this. One modelling stage might represent a real tray of a column or a theoretical stage. In a packed column the stage represent a theoretical stage which corresponds to a segment height (5-150 cm) of the packing in a real column.

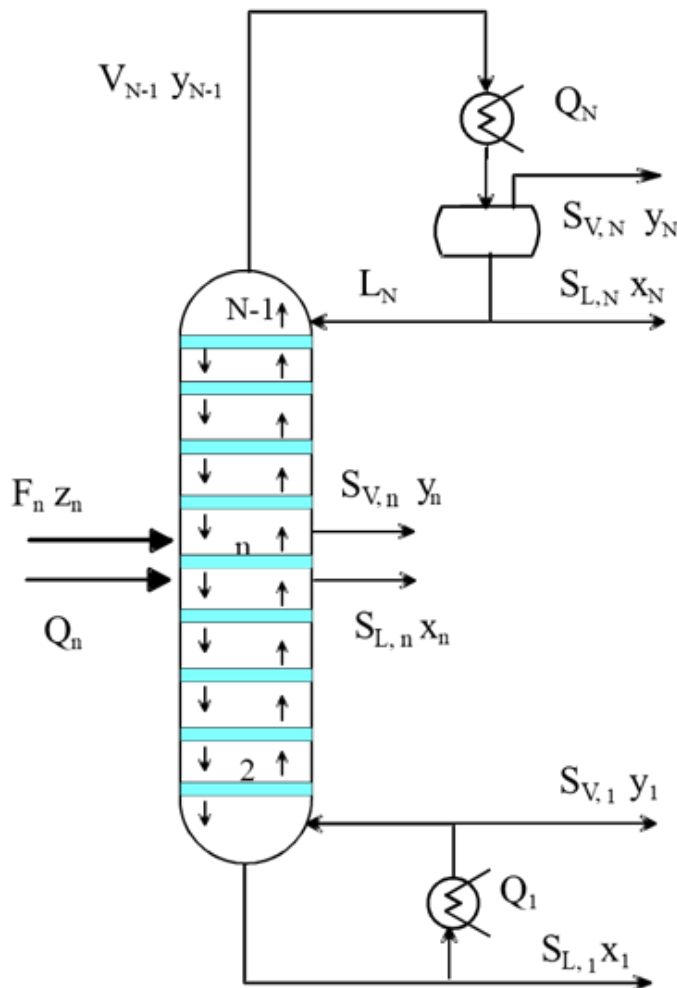


Figure 2. Schematic distillation column for the balances at the stages.

The plates are numbered either from down under or vice versa: The bottom plate is a reboiler if it exists. The top plate is a condenser, if it exists. The heat exchangers (reboiler, condenser, interboiler, intercondenser) are described only as energy flows (Q_n) on every tray. That is, the technical implementation is not considered. Q_n of a reboiler is positive, because heat is brought to the distillation column and Q_n of a condenser is negative, respectively.

A feed F (liquid, vapour or both) and energy flow Q can be introduced to all plates of the model. Also a liquid (S_L) or vapour product (S_V) (letter S from Side draw) can be collected. On most of the plates, there are no such flows, thus the respective values are zero. Often, the liquid bottoms product flow has the notation B and the liquid distillate has the notation D .

2.1.1 Multicomponent distillation, modelling a separation stage

The model of a stage is a group of balances. They are sometimes abbreviated as MESH equations, the letters evolving mass (or mole) (M), equilibrium (E), summation (S) and enthalpy (H) balances. In the next equations the subscript n refers to a plate and the subscript i to a component.

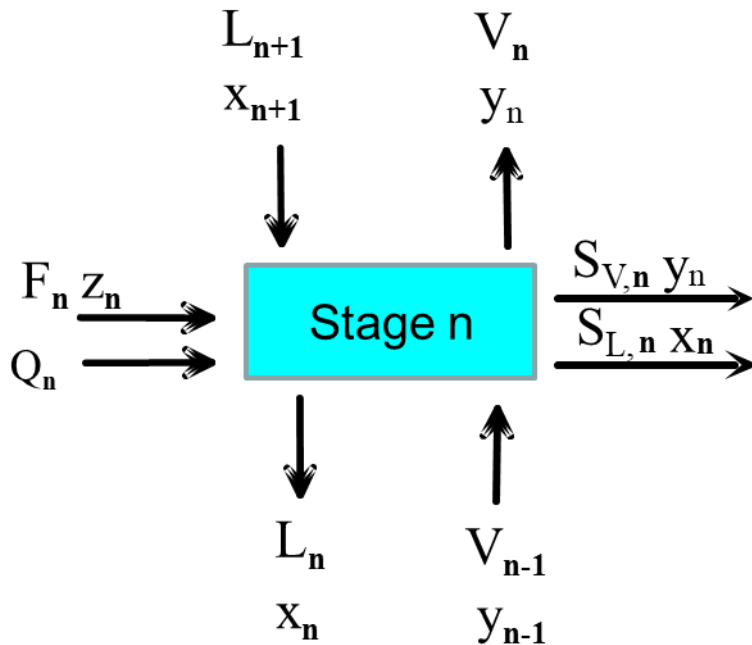


Figure 3. The variables at one ideal stage (notation n is the n'th separation stage of column of Figure 7)

Below are listed and partly developed one version of MESH -equations

A total balance (mole balance)

$$F_n + L_{n+1} + V_{n-1} - L_n - V_n - S_{L,n} - S_{V,n} = 0 \quad (1)$$

Component mass balances (as many as there are components Nc)

$$F_n z_{n,i} + L_{n+1} x_{n+1,i} + V_{n-1} y_{n-1,i} - L_n x_{n,i} - V_n y_{n,i} - S_{L,n} x_{n,i} - S_{V,n} y_{n,i} = 0 \quad (2)$$

An energy balance (enthalpy balance)

$$F_n h_n + L_{n+1} h_{n+1} + V_{n-1} H_{n-1} - L_n h_n - V_n H_n - S_{L,n} h_n - S_{V,n} H_n + Q_n = 0 \quad (3)$$

The composition and properties of the feed must be determined - that is, the quantities F_n, z_n, h_n should be known. Also product flows $S_{L,n}, S_{V,n}$ and energy flows into a plate Q_n are assumed to be known.

On every plate there are Nc+4 variables, (where Nc is the number of components)

x	concentration of liquid, number	N_C
y	concentration of vapor, number	N_C
h	enthalpy of liquid, number	1
H	enthalpy of vapor, number	1
L	liquid flow, number	1
V	vapor flow, number	1

This means, the total amount of variables is $2 \cdot N_C + 4$. However, we have only this far listed $N_C + 2$ equations so we need $N_C + 2$ independent equations more to solve the system.

The sum (summation S) of mole fractions must be one on every plate, which gives two extra equations.

$$\sum_{i=1}^{N_C} x_{n,i} = 1 \quad (4)$$

$$\sum_{i=1}^{N_C} y_{n,i} = 1 \quad (5)$$

Only one of these equations is independent and the other one is a consequence of equations (39) and (40).

$$\sum_{i=1}^{N_C} y_{n,i} - \sum_{i=1}^{N_C} x_{n,i} = 0 \quad (6)$$

Often the equations are expressed as a difference together with the equilibrium (equilibrium E) relations. If a plate is an equilibrium stage, the leaving flows are in phase equilibrium. This assumption gives us the missing N_C equations.

$$y_{n,i} = K_{n,i}(x_n, y_n, T_n, p_n) x_i \quad (7)$$

Enthalpy of liquid is dependent on following variables

$$h_n = h(x_n, T_n, p_n) \quad (8)$$

and enthalpy of vapour is dependent on following variables

$$H_n = H(y_n, T_n, p_n) \quad (9)$$

Writing these two equations keeps the degree of freedom unchangeable, because two more variables, temperature and pressure, are presented.

Either pressure (p) in the column or a pressure drop (Δp) between adjacent plates is often defined as constant. The pressure is chosen according to cooling medium (often water) and boiling medium (steam, hot oil).

$$p_{n-1} - p_n - \Delta p_n = 0 \quad (10)$$

Now we have $2 N_C + 6$ equations and variables, so the system of equations can basically be solved. As mentioned before, energy flows, the feed and product flows must be specified (even they are zero on most of the stages).

A common concept in distillation simulation is the Murphree efficiency ε . It is a measure of how close the actual tray approaches the equilibrium situation on one tray. It is the ratio between the composition differences of the vapour phase flows of a situation where the efficiency is given to the difference where the equilibrium assumption prevails

$$\varepsilon = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}} \quad (11)$$

This equation with the variable ε can be incorporated in the set of equations of a distillation column.

2.2.2 Model of the Reboiler and of the Condenser

The reboiler and the condenser can also be described as a separation stage. The reboiler can be used more or less as such the stage was described above. However, the typical configuration of the condenser needs a new concept, the reflux ratio R_D . It is the ratio between the liquid returned into the column to the product taken out of the column $R_D = L_N / S_{L,N}$.

3. EQUIPMENT

Column used in this laboratory work is an Oldershaw column; a schema is drawn in figure 4. The structure of the trays in the column is shown in figure 5.

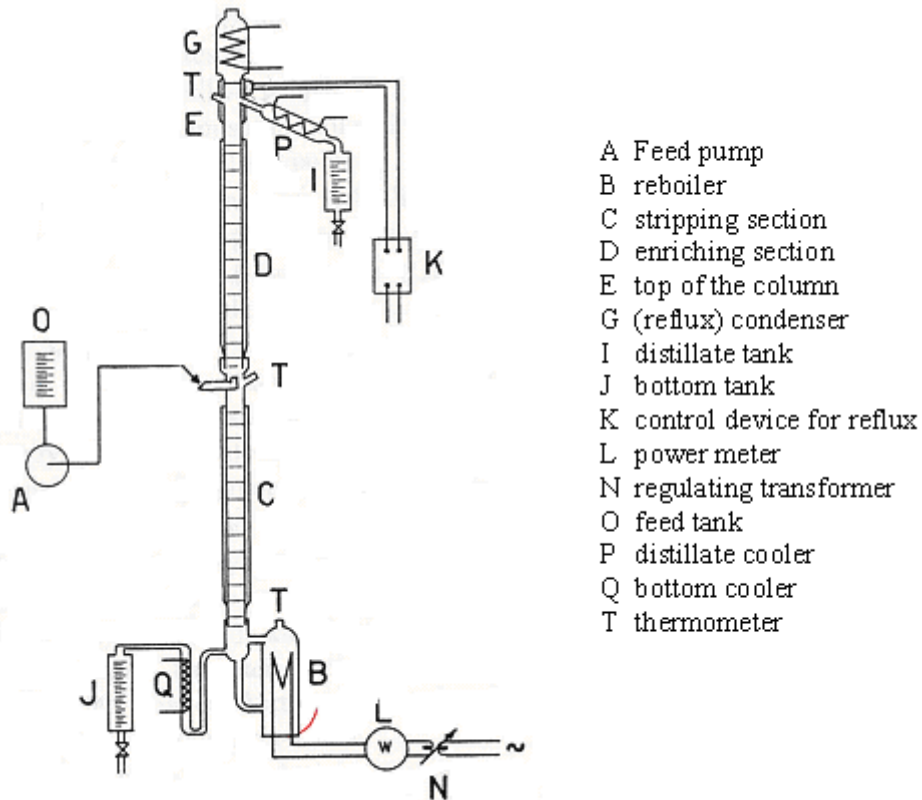


Figure 4. Schematic of Oldershaw Column

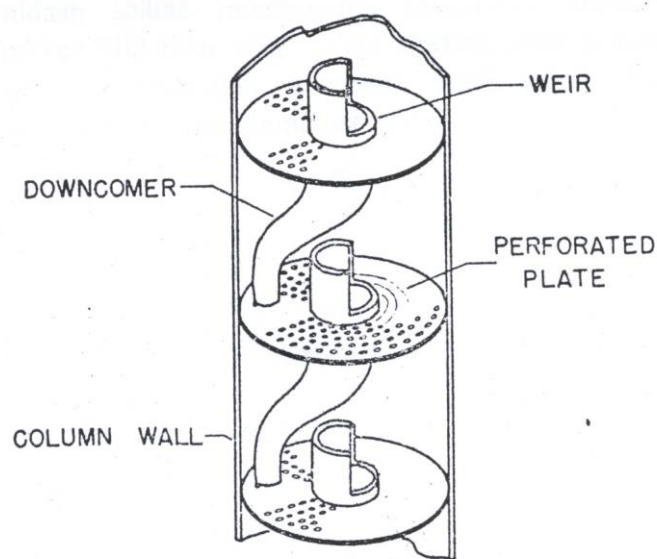


Figure 5. Structure of the column.

3.1 COLUMN

Laboratory column is an Oldershaw sieve tray column, which was first made by C.F. Oldershaw in his laboratory when working in research laboratory of Shell Development Co in California (Oldershaw (1941)). The column and its equipment were delivered by Normschliff Glasgeräte KG in 1965. The column, as the original, is made of glass.

- The height of the stripping section is 0,7 m and there are 10 actual trays.
- The height of the enriching section is 1,1 m and there 20 actual trays.
- The internal diameter of both sections is 47,6 mm and they are covered with silvered vacuum jacket in order to reduce heat losses.
- The trays are sieve trays and in every tray there are 230 holes, diameters of which are 0,95 mm. The distance between two trays is 47,6 mm.
- Reboiler is a natural-circulation - or thermosiphon reboiler. (McCabe, p. 443), and liquid volume is about 1 liter. Heating is done by two heating resistors (quartz rod). Heating power can be adjusted with the regulating transformer and the electrical power can be read from the power meter. Due to heat losses the actual vaporization efficiency is smaller than the measured power.
- Bottom product exits the column bottom as overflow.
- Feed is pumped between the two sections or in practice to the topmost plate of the stripping section, which is the feed plate. The volume flow rate of the feed can be adjusted by adjusting stroke length or frequency of the pump.
- Condenser is a coil condenser and its heat transfer area is about 0,3 m². The heat power of the condenser can be measured by measuring the flow rate and the temperature change of the cooling water.
- Between the column and the condenser is a reflux control device, which controls the reflux flow rate. There is a pendulum pipe in the control device, which can be adjusted with an electromagnet: When electromagnet is off, all distillate is refluxed and when it is on, all liquid goes to distillate.
- The reflux ratio can be adjusted by changing the impulse intervals of the electromagnet. Due to inaccuracies in this method, the actual reflux ratio is calculated from the heat balance of the condenser.

3.2 MEASURING EQUIPMENT

- stopwatch
- sample bottles
- 2 l measuring cylinder
- Scales
- Refractometer

4. OPERATING THE DISTILLATION COLUMN

4.1 ARRANGEMENTS

A mixture of n-hexane and cyclohexane is distilled in this work. The physical properties of these components are shown in appendix 1.

CAUTION 1!

The mixture of n-hexane and cyclohexane is very flammable!

CAUTION 2!

Beware of skin contact and inhaling the solvent vapors.

Cyclohexane may irritate skin and perform as a suffocating and narcotic agent. Also it may contain small amounts of benzene (at most 0,05 %).

One **must** keep watch on column when it is in operation.

4.2 STARTING THE WORK AND TOTAL REFLUX RUN MEASUREMENT

- Measure the refraction index of the feed (2 times).
- Fill the reboiler with the feed pump to the mark indicated by the assistant
- Open the cooling water valves.
- Switch the power on to the reboiler and start heating with 250 W.
- Start to monitor the temperatures of the column (5 minutes interval)
- When vapor reaches the top of the column (temperature!) wait until refluxing starts. The column operates now on total reflux (feed and product flows are zero).
- Adjust the cooling water flow of the condenser so that the change in the temperature of the cooling water is about 5 - 10 K.
- When all the temperature are in steady state take samples from the bottoms and from the reflux as the assistant indicates

4.3 FEED RUN MEASUREMENTS

- Start the feed pump.
- Switch the reflux control device on and adjust the reflux ratio.
- After starting the feed take the temperatures every 5 minutes.
- When steady state is reached, which means that the temperatures remains unchanged, make the following measurements:
 1. Take up the boiling power and reflux ratio values.
 2. Measure the feed, distillate, and bottom product mass flow rates simultaneously (at least 1000 ml).
 3. Measure refraction indexes of the bottom product and distillate.
 4. Measure the temperatures of the top, bottom, and the feed point of the column.
 5. Measure the cooling water mass flow rate as in 2.
 6. Take up the cooling water temperatures.
 7. In order to notice any gross errors, calculate the relative error of the total balance and the n-hexane balance and the heat flow removed from the condenser. Show your results to the assistant.

4.4 FINISHING THE WORK

- Show your results to the assistant and after permission:
 1. Switch the reboiler and feed pump off.
 2. Switch the reflux control device off.
 3. Pour the distillate and bottom product to feed tank.
 4. Leave the cooling water valves open. The Assistant closes them when the column is cooled enough.
 5. Clean up your working place.

5. REPORTING

The reporting is done in the format as described in general instructions of the course.

Summarize the measured results.

Change the mass flows to molar flows (all calculations are done in mole flows or in mole fraction coordinates).

Match the measured balances as in appendix 3. The MS-EXCEL spreadsheet can be found in the homepage of this course. Use the obtained values for the calculations from this on.

Set up the simulation in Aspen Plus, think about your thermodynamical model. Motivate you choice.

For the total reflux run:

- Show the stabilizing of the column graphically after starting warm up (the concentration and temperature of the top of the column as a function of time).
- Print the xy –diagramme from Aspen Plus
- Determine the amount of ideal stages graphically (total reflux run)
- Calculate the overall tray efficiency $\varepsilon = \frac{N}{N_{ACT}}$ (you get N from the graph, think how to get N_{act} . Is the total condenser an equilibrium stage what about the reboiler?)

For the feed run:

- Calculate the feed
- Show the stabilizing of the column graphically after starting the feed (the concentration and temperature of the top of the column as a function of time).
- Set up the simulation the column consists of the reboiler, actual trays and the condenser
- **Note!** Aspen Plus counts the stages starting from the condenser (stage = 1).
- Specify the feed point and the pressure of the column
- Use the measured reflux ratio and flows (B or D) as your specifications
- Specify the efficiency that you got above/previoursly as the Murphee efficiency of the actual plates
- Simulate the column
- Compare the results to your measurements (perhaps you have to calculate some things by hand), check compositions, check energy consumption (reboiler, condenser). They probably not agree perfectly.

- Now imagine that our lab column is a research column of our company. We need to know how to simulate future experiments. It is common knowledge that small equipment suffer from heat losses (especially the reboiler), the efficiencies of the plates might be a lot less than 100%. Thus, make a set of simulations where you consistently by trial and error seek a good setup for the simulation so that you can simulate a future experiment. (What are the efficiencies; we calculated an estimate from the total reflux runs, what are the heat losses; compare the product composition, flows and energy from the simulation). Do this in a consistent manner. Observe especially the interplay between energy input into the column, composition and inner flows of the column. Report your logic in the trials, the trends and your result. A dozen of simulations is expected.

- Conclusions. What can you say about the results? Why the results are like they are? Do the results fit the theory? If not, why?

Do the uncertainty estimate as in general instructions; calculate the errors of the material balances numerically, estimate gross and systematical errors qualitatively.

6. REFERENCES

- McCabe, W.L., Smith, J.C. and Harriot, P., Unit Operations of Chemical Engineering, McGraw-Hill, 5th ed., 1993.
- Oldershaw, C.F., Perforated Plate Columns for Analytical Batch Distillations, Ind. Eng. Chem. Vol 13, No 4, 1991, 265-268

7. NOMENCLATURE

B	bottom product flow, mol/s
D	distillate flow, mol/s
F	feed flow, mol/s
c_p	molar heat capacity, kJ/kmolK
h	molar enthalpy of liquid, kJ/kmol
H	molar enthalpy of gas, kJ/kmol
L	total molar flow in liquid, mol/s
N	number of ideal stages
p	pressure, Pa
r	molar latent heat, kJ/kmol
R_B	reboil ratio, dimensionless
R_D	reflux ratio, dimensionless
S	product flow, mol/s
q	state of the feed, dimensionless
T	temperature, K or °C
V	total molar flow in gas, mol/s
x	mole fraction in liquid, usually for component A, dimensionless
y	mole fraction in gas, usually for component A, dimensionless
z	feed composition, dimensionless

Greek Letters

α	relative volatility, dimensionless
Φ	heat flow, kJ/s

Subindexes

B	bottom product
D	distillate
F	feed
L	liquid
n	stage number
N	
x	liquid
y	vapor
V	vapor

Superscripts

x^*	equilibrium value for x
y^*	equilibrium value for y
\bar{L}	value of L in the bottom of the column

8. APPENDIXES

1. Physical properties of cyclohexane and n-hexane.
2. Refraction indexes for a mixture of cyclohexane and n-hexane.
3. Flow in rotameter of cooling water
4. Matching the balances of binary distillation with MS_EXCEL program.

PHYSICAL PROPERTIES OF CYCLOHEXANE AND N-HEXANE

1. Properties of pure substances

Equations for properties:

Density ρ

$$\ln(\rho/1000) = \ln A - (1 - T_r)^{2/7} \cdot \ln B, \text{ where } T_r = T/T_c, T (\text{ } \text{K}), T_c (\text{ } \text{K})$$

Heat capacity c_p :

$$c_p = A + BT + CT^2 + DT^3, (\text{ } \text{J/kmol/K}), T (\text{ } \text{K})$$

Latent heat ΔH_v :

$$\Delta H_v = \Delta H_{v,ref} \left[\frac{(T_c - T)}{(T_c - T_{ref})} \right]^n$$

Table 1. Parameters for calculating physical properties of pure substances.

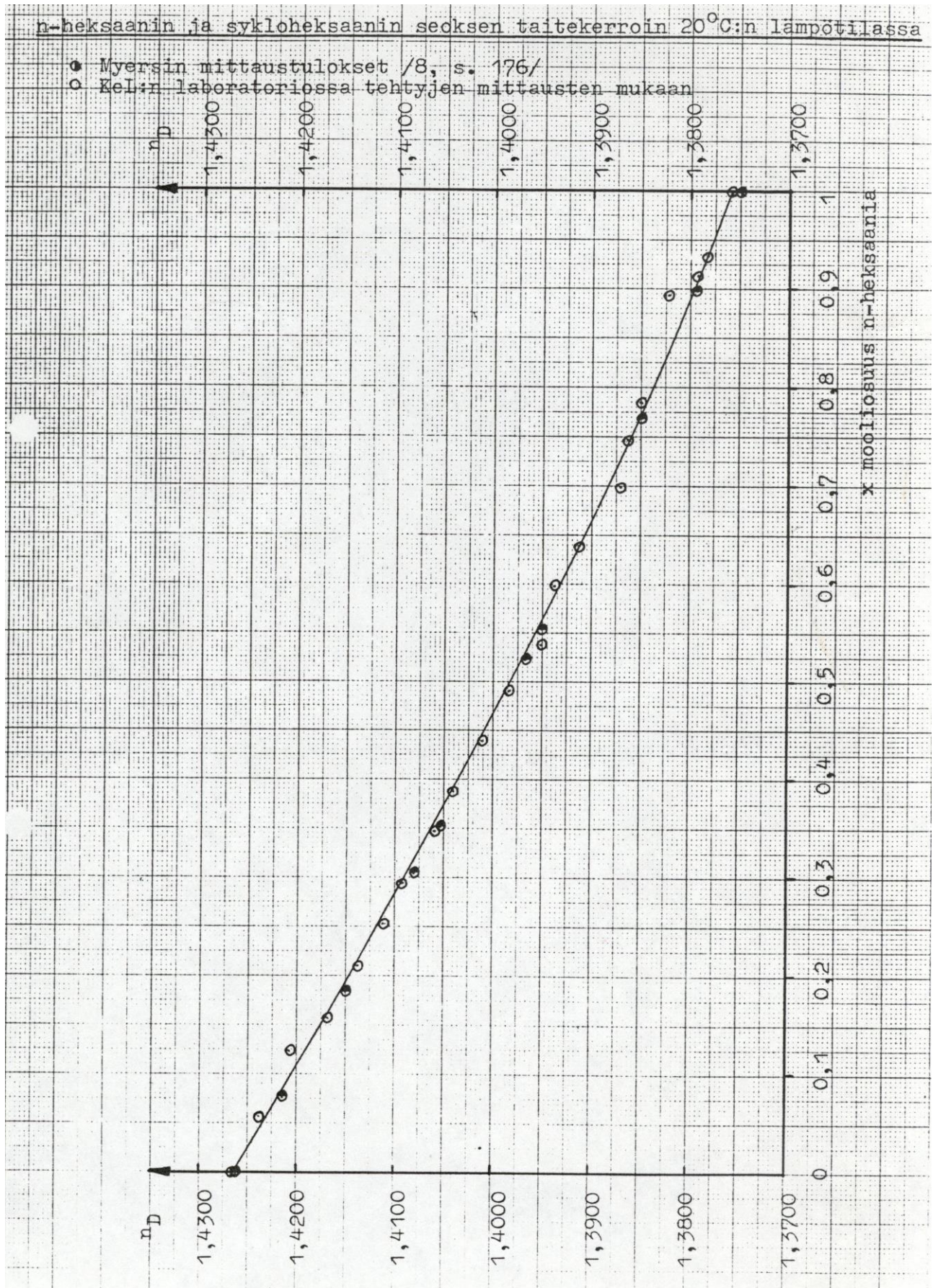
	n-hexane	cyclohexane
Molar weight M	86.178 kg/kmol	84.162 kg/kmol
Boiling point T_{kp}	68.74 °C	80.75 °C
Critical temperature T_c	234.45 °C	281 °C
Latent heat, $\Delta H_{v,ref}$ in boiling point (T_{ref})	28.66 kJ/mol	30.33 kJ/mol
exponent n	0.381	0.405
Heat capacity c_p	$A = 172120$ $B = -183,78$ $C = 0,88734$ $D = 0$	$A = -220600$ $B = 3118,3$ $C = -9,4216$ $D = 0,010687$
Density	$A = 0.2368$ $B = 0.2693$	$A = 0.2729$ $B = 0.2727$

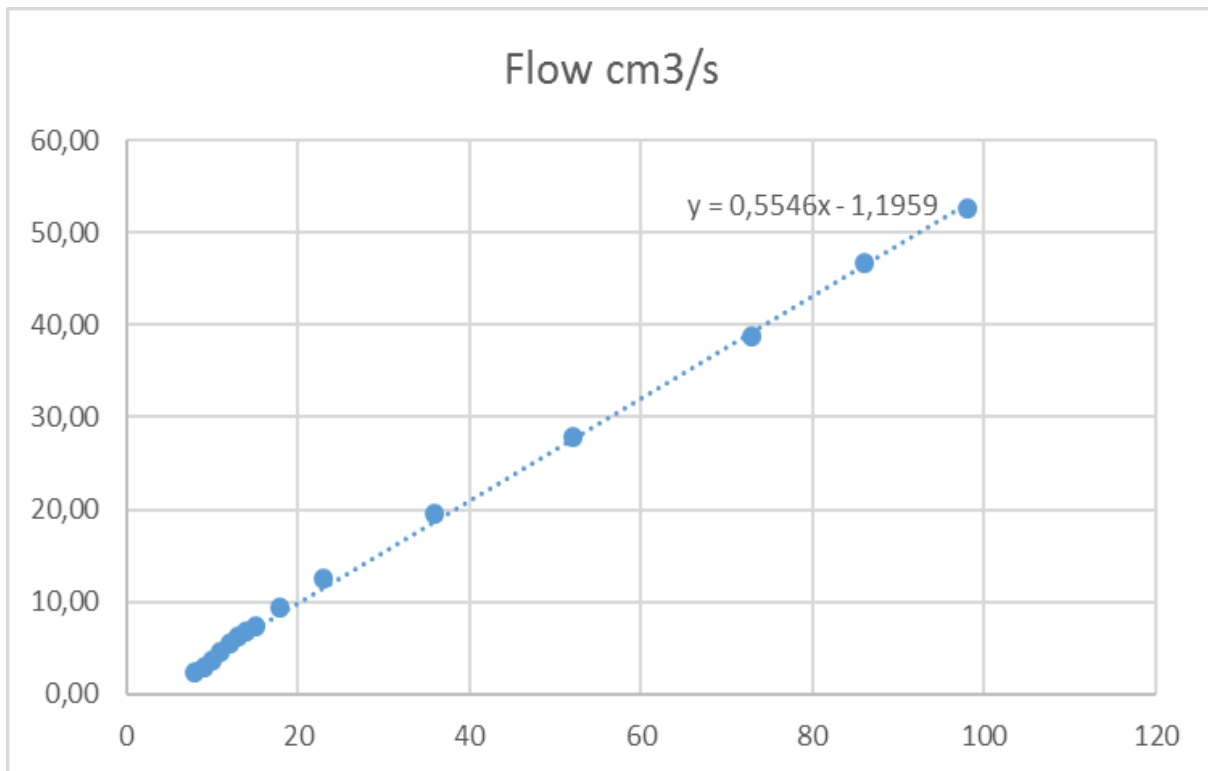
2. Properties for mixture

2.1 Equilibrium

Relative volatility α_{AB} is in the range (1.55, 1.33) when x_A is in the range (0.1,0.9).Here, a constant relative volatility is used and $\alpha_{AB}=1.4$

2.2 The refraction index of the mixture in temperature 20 °C is shown graphically in appendix 2.





Flow in Rotameter of the Cooling Water

MATCHING THE BALANCES		8.4.2005
This workbook contains the following sheets:		Color codes
MENU	This sheet.	GIVE
BASIC CASE	Protected therefore Solver does not work.	MATCHED
EXTRACTION	Protected therefore Solver does not work.	CONSTRAINTS
BINARY DISTILLATION	Protected therefore Solver does not work.	TARGET FUNCTION
MACRO	Contains the macros.	
GENERAL PRINCIPLE OF MATCHING THE BALANCES		
The sum $S = \text{SUM}(P_i * ((x_i - X_i) / x_i)^2)$ is minimized		
x_i = measured value of quantity i		
X_i = matched value of quantity i		
P_i = weighting coefficient of quantity i		
so that total and component balances hold true and the sum of mole fractions is one.		
The task at hand is a constrained optimization task, which constraints are the balances and the sum of mole fractions and the minimized function is the weighted sum of relative differences of matched and measured values.		
There is no universal algorithm to solve a problem like this, which would always lead to a solution.		
Therefore matching should be done with few different initial values.		
INSTRUCTIONS		
0 Go to sheet and use the macro CopyPage to make an unprotected sheet.		
1 Give the measured values to cells: MEASURED		
2 If needed give the weighting coefficients to cells: WEIGHT		
The greater the weighting coefficient the smaller is the difference between matched and measured values.		
Weighting coefficient of an accurate measurement should be large.		
Weighting coefficient of an inaccurate measurement should be small.		
Weighting coefficient can be in the range (0, positive infinity). Here the coefficients should be ≥ 1 .		
If there is no special need for weighting, use 1.		
3 Give initial values (e.g. measured values) to cells: INITIAL VALUES		
Leave all other cells UNTOUCHED!		
4 Choose Tools / Solver / Solve: Solver starts the optimization.		
The target cell of the optimization becomes active.		
The constraints are:		
Balances hold true, in other words the cells IN-OUT = 0		
Sum of mole fractions equals to 1, cells $\text{SUM}x = 1$		
All matched values are positive, $\text{MATCHED} \geq 0$		
5 If the result is not reasonable, try other initial values or change the weighting coefficients.		
Notice that the best result is obtained only by trial and error.		
6 If a reasonable result is not found, save the result and restart solver.		
If a reasonable result is not found after a few times, return to 5 and try other initial values.		
7 Extraction:		
Substance A (component to be extracted) = HAc		
Substance B (diluter) = MIK		
Substance S (solvent) = water		
if HAc, of which the feed is made, contains water p -weight %, feed contains $x_B = 1.0 - (1.0 + p / (100 - p)) * x_A$,		
since x_S / x_A is always $p / (100 - p)$ in feed, which is made by adding substance B		
In the solvent $y_S = 1.0$ because it is pure water.		
Raffinate and extract are saturated, so x_S is obtained from the saturation curve.		
In raffinate $x_S = 0.56 * x_A + 0.01$ and in extract $x_S = -1.17 * x_A + 0.98$.		
In both $x_B = 1.0 - x_A - x_S$		
Extra constraint: in raffinate and extract $x_A \geq 0.001$		
8 Binary distillation		
Notice that $x_B = 1.0 - x_A$		

MATCHING THE BALANCES		BINARY DISTILLATION										
		WEIGHTING COEFFICIENTS						COLOR CODES				
		IN			OUT			GIVE				
		F			B		D	MATCHED				
TOTAL		1.0			1.0		1.0	CONSTRAINTS				
x1		1.0			1.0		1.0	TARGET FUNCTION				
x2		1.0			1.0		1.0					
		MEASURED										
		IN			OUT			IN-OUT				
		F			B		D					
TOTAL		2.000			3.000		3.000	-4.000				
x1		0.200	0.400		0.300	0.900	0.300	0.900	-1.400			
x2		0.800	1.600		0.700	2.100	0.700	2.100	-2.600			
SUMx		1.000			1.000		1.000					
		MATCHED										
		IN			OUT			IN-OUT				
		F			B		D					
TOTAL		1.100			3.300		4.400	-6.600				
x1		0.110	0.121		0.330	1.089	0.440	1.936	-2.904			
x2		0.890	0.979		0.670	2.211	0.560	2.464	-3.696			
SUMx		1.000			1.000		1.000					
		RELATIVE DIFFERENCE										
		IN			OUT							
		F			B		D					
TOTAL		0.450			-0.100		-0.467					
x1		0.450			-0.100		-0.467					
x2		-0.113			0.043		0.200					
		WEIGHTING COEFFICIENT * SQUARE OF RELATIVE DIFFERENCE										
		IN			OUT							
		F			B		D					
TOTAL		0.203			0.010		0.218	SUM				
x1		0.203			0.010		0.218	0.915				
x2		0.013			0.002		0.040					