Batch rectification on a glass bubble plate column

1. Theoretical background

There are several reasons to perform batch rectification instead of continuous rectification:

a) the mass flow of the feed is too low
b) more than two products have to be separated in one column
c) the concentration of feed is not stable
d) a good separation is necessary on acolumn with relatively small number of theoretical plates.

If batch rectification is performed, the whole amount of feed is fed into the reboiler at once, and continuous evaporation is performed. Vapour is condensed at the top of the column int he condenser, and a part of the condensed fluid is fed back to the column as reflux, while the other part is collected as distillate. Liquid from the reflux and vapour are in counter-current flow int he column, thus rectification is performed. A schematic draw of a batch rectification unit is presented in Fig1. x_j and y_j are in equilibrium. The unit itself is basically a rectification column having only upper column part.
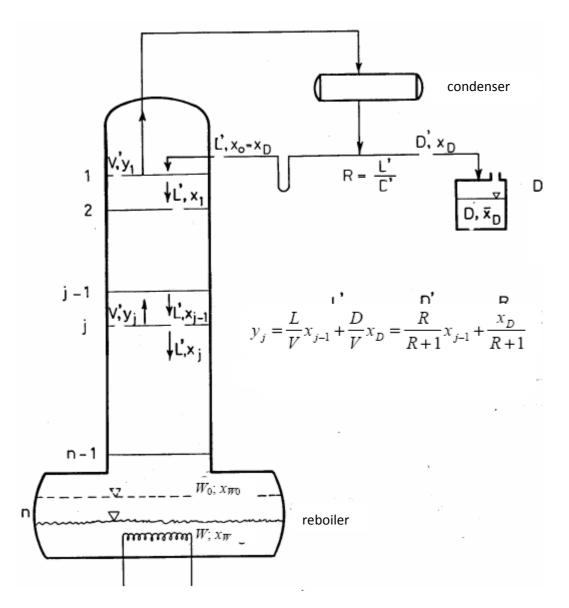
Fig 1. Schematic representation of a batch rectification unit

There are practically to ways to operate a batch rectification unit:

a) to work in constant reflux ratio (R) mode,
b) to work in constant distillate composition (x_D) mode.

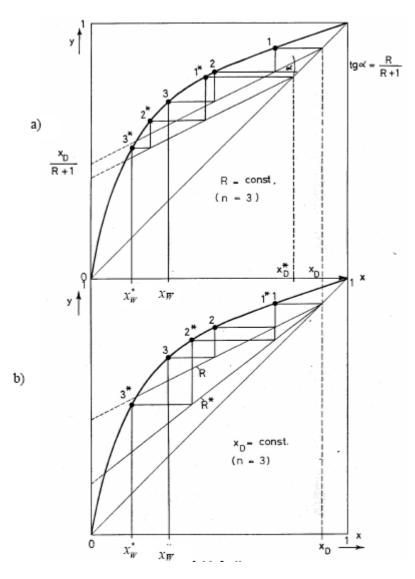These two basic modes are presented in Fig2a and Fig 2b, respectively.

Fig 2. Two basic ways of operation a batch rectification unit

It is practical to operate in constant reflux ratio mode, if the aim is to get rid of a small amount of volatile impurity. In this case, as shown in Fig2a, x_D will continuously decrease during the process, as x_W (molar ratio of the volatile compound in the reboiler) is also decreasing.

If the aim of the rectification is to get the volatile compound in high purity at the top of the column, it worths to operate it in constant x_D mode (Fig2b). In this case, since x_W is decreasing continuously, the reflux ratio have to be continuously increased to ensure constant x_D.

1.1 Batch rectification with contant reflux ratio

Mass and component balace equations if the material int he column (and the changing amount of this material) is neglected:

$dD=dW$

$Wx_W=(W-dW)(x_W-dx_W)+x_DdW$

where W is the amount of the material int he reboiler

            D is the amount of distillate

            x_D is the concentration of the more volatile compound int he distillate

            x_W is the concentration of the more volatile compound int he reboiler.

From the equations a new is easily derived, similar to Rayleigh equation:

$$\frac{dW}{W} = \frac{dx_W}{x_D - x_W}$$

After integration:

$$ln\frac{W_0}{W} = \int_{x_W}^{x_{W0}} \frac{dx_W}{x_D - x_W} = I$$

I value of the integral may be determined by graphical or numerical methods.

It is straightforward than to calculate the amount of residue $W = \frac{W_0}{e^I}$ and distillate $D=W_0-W$.

Avarage composition of distillate may be calculated from the overall component balance:

$$\overline{x_D} = \frac{W_0 x_{W0} - W x_W}{D}$$

Amount of material to be evaporised may be calculated with the reflux ratio:

$V=(R+1)D$

2. The measurement

The following basic knowledge is required beside the things detailed in this note: definition of theoretical plate, phase diagram, bubble point- cloud point diagram, reflux ratio, equation of upper working line, McCabe-Thiele method and the theoretical background required to understand and use the above phrases.

The unit may be operated in constant reflux ratio or constant distillate concentration modes. The unit is built up from a column with 10 bubble plates, an insulated coat with electrical heating, a1 l inner volume reboiler, distillation head with possibility of controlling reflux ratio.
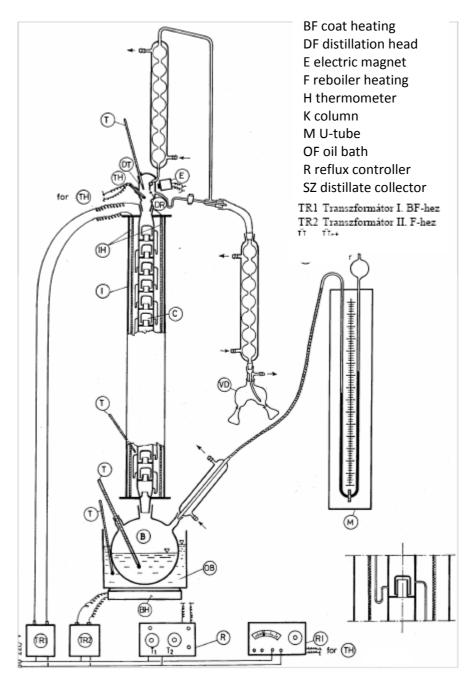
Temperatures of the reboiler and the pressure inside the reboiler have to be measured. For the measurement of pressure drop along the column (ie. pressure in the reboiler – atmospheric pressure) an U-tube manometer is installed. Liquid inside the U-tube is toluene.

The reboiler is heated by an oil bath, which is electrically heated. Read the temperature of the oil bath regularly!

Temperature of vapour is to be measured in the distillation head. COntrol of reflux is perforemd by a time-dependent electrical-magnet switch. Fractions of distillatemay be collected separately.

Electrical heating of the reboiler and that of the coat my be controlled separately. Concentrations of distillate and residue samples are to be analysed by refractometry. A schematic draw of the laboratory unit is shown in Fig 3.

Remember, that the used solvents are flammable!

BF coat heating
DF distillation head
E electric magnet
F reboiler heating
H thermometer
K column
M U-tube
OF oil bath
R reflux controller
SZ distillate collector

TR1 Transzformátor I. BF-hez
TR2 Transzformátor II. F-hez
TT

Fig 3. The laboratory batch rectification column

2.1 Starting the measurement

1. 450-500 g n-heptane- toluene mixture (heptane content between 40-55 %) is filled into the reboiler. Phase diagrams of the mixture are shown on Fig 5 and Fig 6. Refractive index data are collected in Fig 7.

2. Start the cooling

3. Start the heating at about 80 scales.

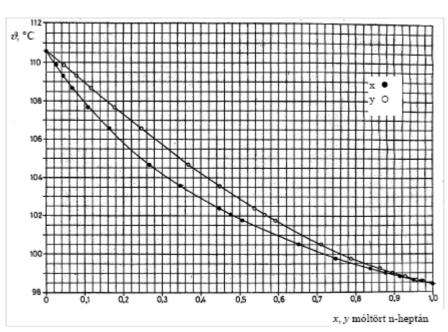4. Close the valve of distillate collection.

5. When boiling starts and the bubbleplates are already working, set the heating of coat between 10-30 scales. Take care, that the temperature of the coat should be always around 90-95°C.

6. Set the temperature of oil bath between 130-150 °C, and work in full reflux for a few minutes to determine number of theoretical plates as follows.

6a. Open the distillate collector valve to collect previously condensed sample. Fill it to the regdo vessel.

6b. Switch on the reflux controller for a short period of time to wash through all pipes and collect a few drops of sample. This should be done fast to ensure that the composition of the sample is the same as in equilibrium during full reflux mode.

6c. Read the temperature of reboiler, pressure drop of column. Calculate the concentration of the liquid based on the diagram in Fig 4., but do not forget to take into account the difference of pressures.

$$\vartheta_{reb}^{atm} = \vartheta_{reb} + 0.046 \left[760 - \left(P_b - \frac{0.862}{13.6} \Delta H_{meas} \right) \right] \ (°C)$$

Fig 4. Bubble point – cloud point diagram of n-heptane - toluene

2.2 Measurements at constant reflux ratio

1. Set the reflux ratio to 0.5-2 according to the instructions. $R = \frac{T_2 - T_1}{T_1}$, where T_2 is the total time (s) and T_1 is the distillate collection time. T1 is suggested to be set as 3-4 s.

2. Open the valve at the distillate collection pipe and start the timer.

3. Collect the samples for 30-30 s into clean vessels at the given period of the run and measure the refractive index of samples. Read in the 15th second the temperature of reboiler and all other data required in the report.

4. Stop the timer after taking the last sample and write down the duration of rectification.

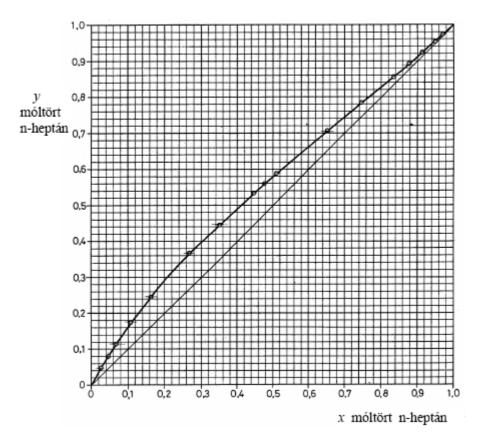5. Unify all distillate samples and measure avarage concentration.

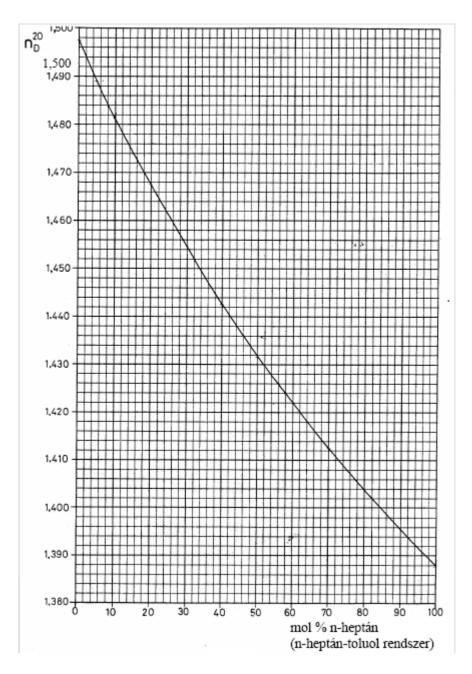Fig 5. y-x diagram of n-heptane – toluene

Fig 6. Refractive index – composition calibration diagram

Lab Report

R=constant

Starting mixture:

refractive index: $n_D^{20} =$

concentration: $x_{W0}=$ %

Atmospheric pressure: (torr)

Reflux ratio: $T_1=$ s

              $T_2=$ s

              $R=$

Heating: reboiler: coat:

Duration of rectification: (min)

time (min)	ϑ_{reb} °C	ϑ_{reb}^{atm} °C	ϑ_{oil} °C	ϑ_{coat} °C	ϑ_D °C	x_W %	ΔH mm	n_D^{20}	x_D %
4^{30}-5^{00}									
9^{30}-10^{00}									
19^{30}-20^{00}									
29^{30}-30^{00}									
39^{30}-40^{00}									
49^{30}-50^{00}									
59^{30}-60^{00}									

At full reflux: $\vartheta_{reb} =$ °C

              $\vartheta_{reb}^{atm}=$ °C

              distillate: $n_D^{20} =$ $x_D=$

     Number of theoretical plates: N_{theo}

     Avarage efficiency of plates: $\eta = \frac{N_{theo}}{N}$

     Amount of distillate: g

     Avarage concentration of distillate: $n_D^{20} =$ $\overline{x_D} =$ %

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