DISTILLATION

1. INTRODUCTION

The unit operation distillation is a method used to separate the components of a liquid solution, which depends upon the distribution of these various components between a vapor and a liquid phase. All components are present in both phases. The vapor phase is created from the liquid phase by vaporization at the boiling point[1].

Distillation can be carried out by either of two main methods in practise. The first method of distillation involves the production of a vapor by boiling the liquid mixture to be separated in a single stage and recovering and condensing the vapors. No liquid is allowed to return to the single-stage still to contact the rising vapors. The second method of distillation involves the returning of a portion of the condensate to the still. The vapors rise through a series of stages or trays, and part of the condansate flows downward through the series of stages or trays countercurrently to the vapors. This second method is called fractional distillation, distillation with reflux, or rectification. Either of these methods may be conducted as a continuous process or as a batch process. Batch distillation is an unsteady state distillation process and it is not as widely used as continuous distillation, and the computations are more complex[2].

2. THEORY

2.1. Continuous Distillation

Continuous rectification, fractionation, is a multistage countercurrent distillation operation. For a binary solution, with certain exceptions it is ordinarily possible by this method to separate the solution into its components, recovering each in any state of purity desired[2].

The feed is introduced more or less centrally into a vertical cascade of stages. Vapor rising in the section above the feed (called the *absorbtion, enriching* or *rectification* section). The liquid returned to the top of the tower is called *reflux* and the material permanently removed is the *distillate*, which may be a vapor or a liquid, rich in more volatile component. In the section below the feed (*stripping* or *exhausting* section), the liquid is stripped of volatile component by vapor produced at the bottom by partial vaporization of the bottom liquid in the reboiler. The liquid removed, rich in less volatile component, is the *residue*, or *bottoms* . Inside the tower, the liquids and vapors are always at their points and dew points, respectively, so that the highest temperatures are at the bottom, the lowest at the top. The entire device is called a *fractionator*[3].
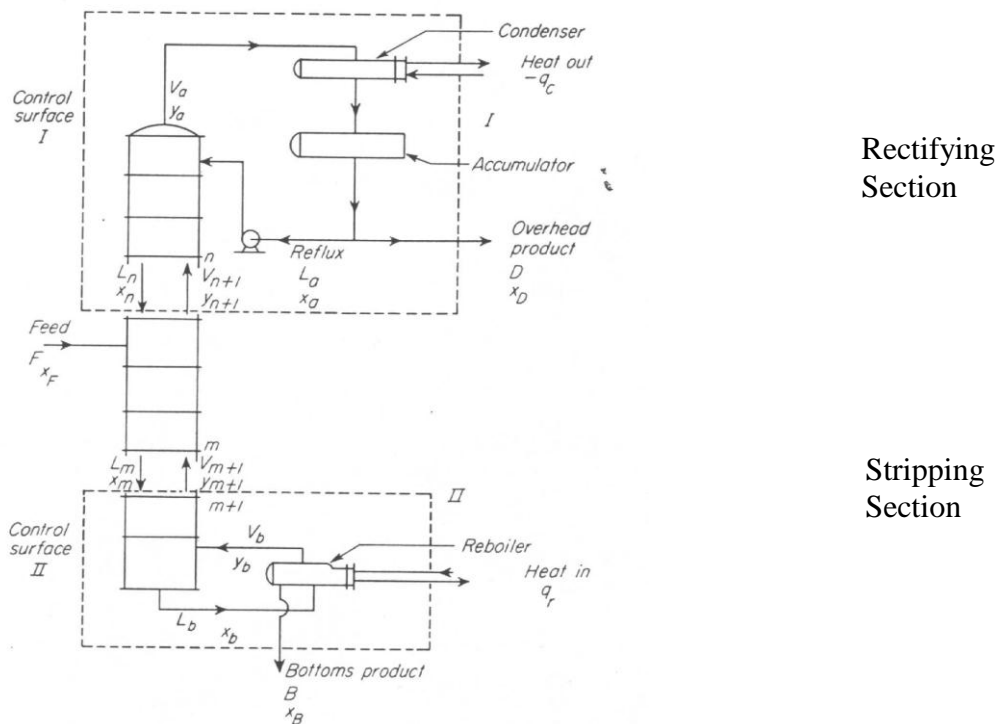

Figure 2.1 Continuous fractionating column with rectifying and stripping sections[2]

Total material balance:

$$F=D+B \tag{1}$$

Component A balance:

$$Fx_F=Dx_D+Bx_B \tag{2}$$

Eliminating B from these equations gives

$$D/F=(x_F-x_B)/(x_D-x_B) \tag{3}$$

Eliminating D gives

$$B/F=(x_D-x_F)/(x_D-x_B) \tag{4}$$

Because there are two sections in the column, there are also two operating lines, one for the rectifying section and the other for the stripping section[3].

The operating line for the rectifying section:

$$y_{n+1}= (L_n/V_{n+1})x_n+(D/V_{n+1})x_D \tag{5}$$

or

$$y_{n+1}= (L_n/(L_n+D))x_n+(D/(L_n+D))x_D \tag{6}$$

The slope of the line defined by Eq. 5 is, as usual, the ratio of the flow of liquid stream to that of the vapor stream[3].

The operating line for the stripping section:

$$y_{m+1}= (L_m/V_{m+1})x_m-(B/V_{m+1})x_B \tag{7}$$

or

$$y_{m+1}= (L_m/(L_m-B))x_m-(B/(L_m-B))x_B \tag{8}$$

The slope of the line defined by Eq. 7 is the ratio of the liquid flow to vapor flow[3].

Reflux Ratio

The analysis of fractionating is facilitated by the use of a quantity called the *reflux ratio.* Two such quantities are used. One is the ratio of the reflux to the overhead product, and the other is the ratio of the reflux to the vapor. Both ratios refer to quantities in the rectifying section[4]. The equations for these ratios are

$$R_D = L/D = (V-D)/D \quad \text{and} \quad R_V = L/V = L/(L+D) \tag{9}$$

If both numerator and denominator of the terms on the right hand side of equation 6 are divided by D, the result is, for constant molal overflow,

$$y_{n+1} = (R_D/(R_D+1))x_n + (1/(R_D+1))x_D \tag{10}$$

The slope is $R_D/(R_D+1)$ as given in Eq. 10. It intersects the y=x line (45° diagonal line) at x=x_D. The intercept of the operating line at x=0 is y=$x_D/(R_D+1)$ [4].

Equation 10 is an equation for the operating line of the rectifying section. The concentration x_D is set by the conditions of the design; and R_D, the reflux ratio, is an operating variable that can be controlled at will by adjusting the split between reflux and overhead product or by changing the amount of vapor formed in the reboiler for a given flow rate of the overhead product[4].

Feed Plate

At the plate where the feed is admitted, the liquid rate or the vapor rate, or both, may change, depending on the thermal condition of the feed. To calculate this change, it is convenient to introduce a quantity q. The q is the measure of the thermal condition of the feed and represents the moles of saturated liquid formed in the feed stage per mole of feed. It takes on following values for various possible feed thermal conditions[3].

Subcooled liquid feed: q>1

Saturated liquid feed: q=1

Partially flashed feed: 1>q>0

Saturated vapor feed: q=0

Superheated vapor feed: q<0

The q value which is defined as energy to convert 1 mole of saturated vapor/molar heat of vaporization for a particular feed can be estimated from:

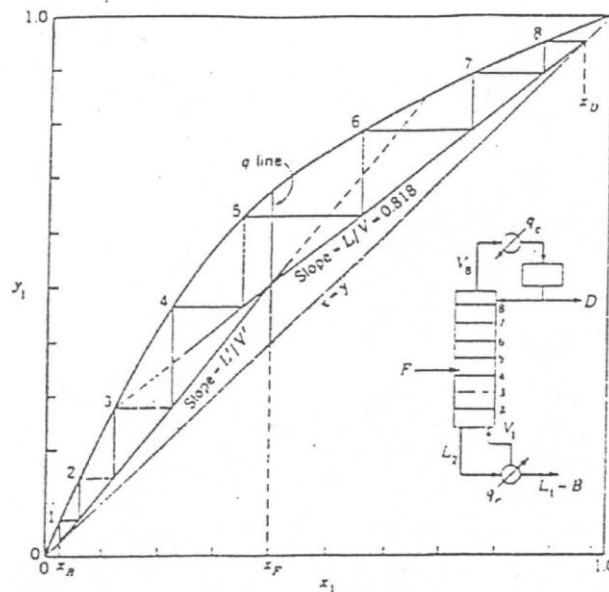The feed line equation $\qquad y=(q/(q-1))x-x_F/(q-1)$ $\qquad\qquad\qquad$ (11)

Figure 2.2 Construction for a column with a bubble-point feed, a total condenser, and a partial reboiler[3].

Minimum Stages

Enough material has been charged to the column to fill the reboiler, the trays and overhead condenser drum to their working levels. The column is then operated with no feed and with all the condensed overhead stream returned as reflux. Also all the liquid reaching the boiler is vaporized and returned to the column as vapor. Since F, D and B is all zero, $L_{n+1}=V_n$ at

all points in the column. With a slope of unity (L/V=1), the operating line must coincide with the diagonal throughout the column. Total reflux operation gives the minimum number of stages required to effect a specified seperation between x_B and x_D[4].

Fenske's equation is used to calculate Nm, which is the number of plates required to make a specified separation at total reflux, i.e., the min. value of N[4].

$$N_{min}+1=log[(x_D(1-x_B))/(x_B(1-x_D))]/log\alpha_{AB} \qquad (12)$$

If the change in the value of α_{AB} from the bottom of the column to the top is moderate, a geometric mean of the extreme values is recommended for α_{AB}[4].

2.2. Batch Distillation

The mixture is charged to a still or reboiler, and heat is supplied through a coil or through the wall of the vessel to bring the liquid to the boiling point and then vaporize part of the batch. The vapor leaving the still at any time is in equilibrium with the liquid in the still, but since vapor is richer in the more volatile component, the compositions of liquid and vapor are not constant[2].

Batch Distillation With Reflux

Batch distillation with only a simple still does not give a good seperation unless the relative volatility is very high. In many cases, a rectifying column with reflux is used to improve the performance of the batch still. If the column is not too large, it may be mounted on top of the still[1].

The operation of a batch still and column can be analyzed using a McCabe-Thiele diagram, with the same operating line operation that was used for rectifying section of a continuous distillation:

$$y_{n+1}=(R_D/(R_D+1))x_n+x_D/(R_D+1) \qquad (13)$$

The system may be operated to keep the top composition constant by increasing the reflux ratio as the composition of the liquid in the reboiler changes. An alternate method of running a batch distillation is to fix the reflux ratio and let the overhead product purity vary with time, stopping the distillation when the amount of product or the average concentration in the total product reaches a certain value[1].

2.3. Tray Towers

Tray towers are vertical cylinders in which liquid and gas are contacted in stepwise fashion on trays or plates. Each tray of the tower is a stage, since on the tray the fluids are brought into intimate contact, interface diffusion occurs, and the fluids are separated[3].

Operating characteristics of sieve trays are shown schematically in Figure 2.3. As the pressure difference is increased due to the increased rate of flow of either gas or liquid, the level in the downspout will rice further to permit the liquid to enter the lower tray. Ultimately the liquid level may reach that on the tray above. Further increase in either flow rate then aggravates the condition rapidly, and the liquid will fill the entire space between the trays. The tower is than *flooded*, the tray efficiency falls to a low value[4].

For liquid gas combinations which tend the foam excessively, high gas velocities may lead to a condition of *priming,* which is also an inoperative situation. Several other undesireable conditions may occur. If liquid rates are too low, the gas rising through the openings of the tray may push the liquid away (*coning*), and contact of the gas and the liquid is poor. If the gas rate is too low, much of the liquid may rain down through the openings of the tray (*weeping*), thus failing to obtain the benefit of complete flow over the trays; and at very low gas rates, non of the liquid reaches the downspouts (*dumping*)[4].
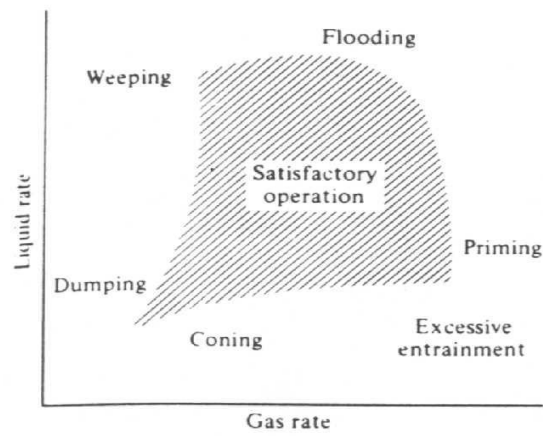
Figure 2.3 Operating characteristics of sieve trays[4]

F-Factor

Figure 2.4 shows actual test data for the common cross-flow devices. The effect of poor gas dispersion on plate efficiency at low gas rates is evident. Similarly, at high gas rates entrainment causes a loss of efficiency[1].
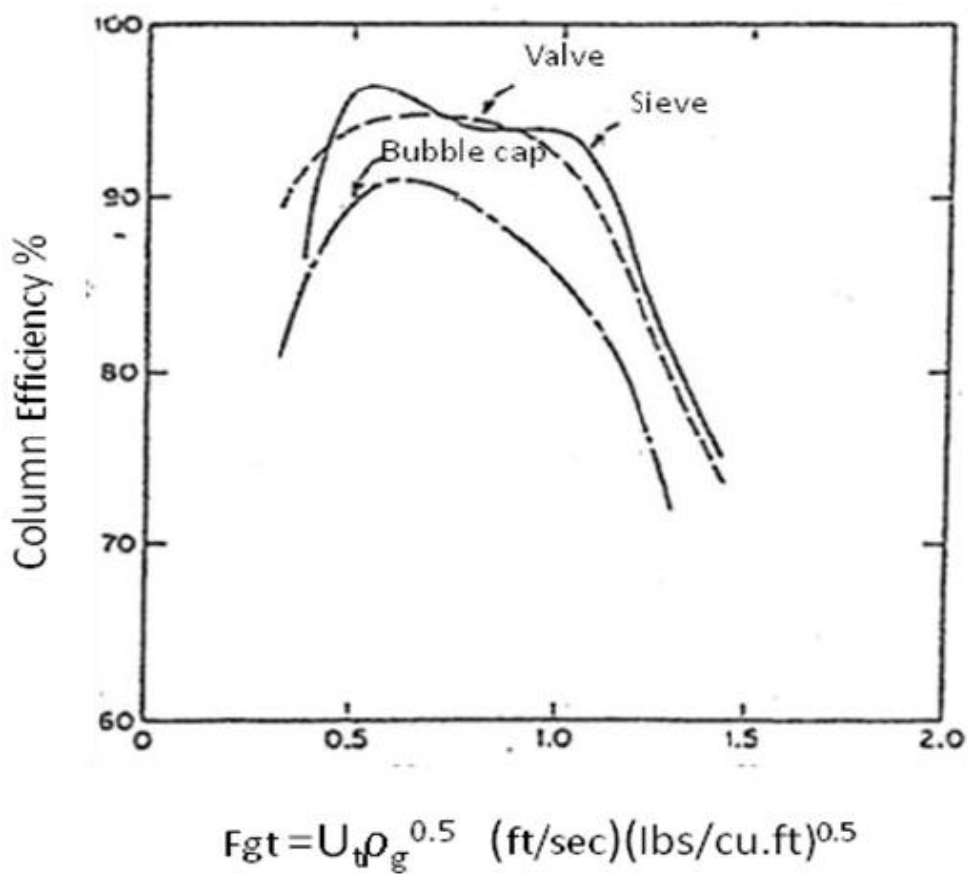
Figure 2.4 Performance profiles of plates for Methanol-water system[1]

The abscissa parameter in Figure 2.4 is a gas phase kinetic energy term, the F-factor, that is often used for correlating purposes:

$$F_{gt}=U_t\rho_g^{0.5} \qquad (14)$$

Where;

U_t: linear gas velocity based on net area, m/s

ρ_g: gas density, kg/m^3

As separation is achieved in the column, the composition of vapor changes and hence vapor density. The average composition between top and bottom is therefore taken in order to calculate F_{gt} [2].

$$F_{gt}=U_t\rho_{gav}^{0.5} \tag{15}$$

and

$$U_t=L(\rho_{lav}/\rho_{gav})(4/(\pi D^2)) \tag{16}$$

Where ;

ρ_{gav} : vapor density at average column composition, kg/m^3

ρ_{lav} : liquid density at average column composition, kg/m^3

L   : liquid rate returned to column at total reflux, m^3/sec

D   : diameter of colum

3. EXPERIMENTAL SECTION

3.1 Safety

People who work in laboratories are exposed to many kinds of hazards. However, many accidents in the laboratory can be prevented if each person in the laboratory observes a set of common precautions and uses proper experimental techniques and procedures at all times.

Please follow these instructions when you came across an accidental situation.

• If you receive a chemical splash (Ethanol solution) to your eyes, immediately flush eyes with plenty of water at least 15 minutes, lifting upper and lower eyelids occasionally[9].

• Inhalation of ethanol vapors irritates the respiratory tract. It may cause coughing, dizziness, dullness, and headache. If you come across high concentration of ethanol vapor, come out to take fresh air [9].

- Spills shall be cleaned immediately. Small spills of liquids on bench tops shall be cleaned immediately with personal protective equipment (gloves) to prevent contact with skin or clothing.

- Your hands and the buttons of the device have to be dry when you working with an electrical device.

- It is the responsibility of everyone working in the laboratory to make certain that the laboratory is left clean after work is performed.
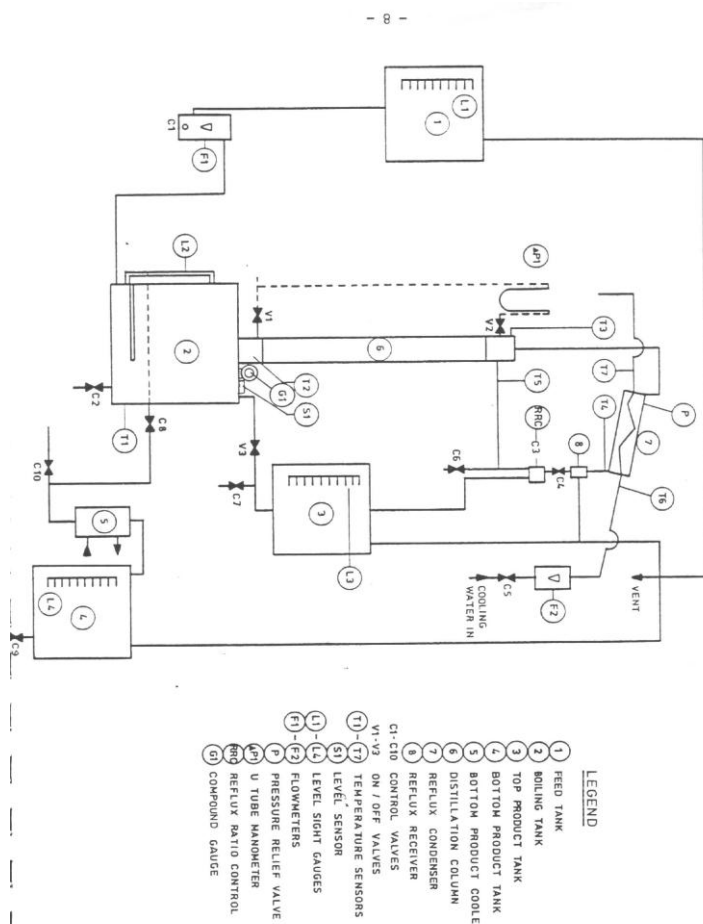
3.2. Experimental Set-Up

Figure 3.1 Distillation experiment system

3.3. Experimental Procedure

a. Continuous Operation

- Close the flowmeter control valve (C1) fully. Charge the feed tank (1) with 8 litres of mixture to be distilled. Charge the reboiler (2) 6 liters of the same mixture.

- Open the C8 valve fully

- The valves V1 and V2 must be open and V3 must be closed. C5 must be opened to allow cooling water to flow through the condenser and cooling water must be supplied to the bottom product cooller. C4 must not be closed too much or the condensate will back up into the condenser.

- Turn the heater power controller clockwise until the reading on the wattmeter is 1.5 kW. After 15-20 min. the mixture will boil and the vapour will appear in the column and rise slowly up it. After a further few minutes, when vapour has reached the condenser, liquid will begin to cascade down the trays. Leave the column to settle then turn the heater controller counterclockwise until the power consumed by the heater is only just efficient to give a gentle bubbling on the trays. Leave the column to stabilize for five minutes.

- Carefully open valve C1 on the flowmeter (F1). Allow the flow of 50 ml/min to be established, turn up the power to compansate for this cold feed. Careful adjustment of the heater power control is required to obtain a settle condition. When this is achived, adjust reflux ratio control to give ratio of, say, 2:1 initially. That is, T1 will be activated for twice as long as T2. For example, set T1 at 12 seconds and T2 at 6 seconds.

- Using valve C6 and C10, a small quantity of top and bottom product can be drawn off as samples. Do this every 10 seconds and analyze the sample using a refractometer. Then change the reflux ratio, wait for the column to settle and take further samples for analysis.

b. Batch Operation

- The still must be charged with a suitable mixture of liquids. The valves V1 and V2, isolating the manometer must be open and V3 must be closed. C5 must be opened to allow cooling water to flow through the condenser C3. The reflux ratio control must be set for total reflux. C4 must not to be closed too much or the condensate will back up into the condenser.

- Turn the heater power controller clockwise until the reading on the wattmeter is 1.5 kW. After 15-20 min. the mixture will boil and the vapour will appear in the column and rise slowly up it. After a further few minutes, when vapour has reached the condenser, liquid will begin to cascade down the trays. Leave the column to settle then turn the heater controller anticlockwise until the power consumed by the heater is only just efficient to give a gentle bubbling on the trays. Leave the column to stabilize for five minutes and then time the boil up rates.

- The heater controller should be stepped up in, say, 250 watt increments being careful to allow 10 min. between each reading for the column to stabilize. Before taking the boil up rate, the pressure drop should be read from the manometer in cms H_2O. At every increment, boil up rates, pressure drop, degree of foaming, average temperature of column and the compositions of overhead and bottom products must be taken as data. Also weep point and the flood point must be determined.

- After that, adjust the reflux ratio to 2:1 and wattmeter to 1 kW. Start the column producing distillate under these conditions and take samples of both tops and bottoms at regular intervals, say every 15 min. To obtain the concentrations, use the refractometer method. Take the samples until the readings of the bottoms from the still became constant.

4. CALCULATIONS

a. Continuous Operation Calculations

- Determine the effect of reflux ratio and the column efficiency by McCabe-Thiele method.

b. Batch Operation Calculations

- Plot the curve relating pressure drop as a function of boil up rate on log-log graph paper and determine weep and flood point.
- Calculate the number of theoretical plates for a given separation at total reflux by using Fenske's method and find the column efficiency.
- Calculate the F-factors and plot a graph of efficiency, E vs. F-factor.
- Determine the variation of top and bottom compositions with time (plot the compositions as a function of time).

SYMBOLS

B   : Flow rate of heavy or bottom product, mol/h

D   : Flow rate of light or overhead product, mol/h

F   : Feed rate, mol/h

F_{gt} : F-factor, $(m/sec)(kg/m^3)^{1/2}$

L   : Flow rate of liquid, in general or in rectifying section, mol/h

N   : Number of ideal plates; N_{min}, min. number at total reflux

R_D : Reflux ratio, L/D

T   : Temperature, ^oC

V   : Flow rate of vapor, in general or in rectifying section, mol/h; V_n,

        from plate n.

x   : Mole fraction of more volatile component in liquid phase; x_B, in bottoms; x_D, in

        overhead; x_F, in feed; x_m, x_n, on plates m and n.

y   : Mole fraction of more volatile component in vapor phase; y_m, y_n, from plates m

        and n

α_{AB} : Relative volatility, dimensionless; α_{AB}, of component A relative to component B

        in binary system.

REFERENCES

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3. R. H. Perry, D. W. Green, "Perry's Chemical Engineers' Handbook", 6th ed., McGraw-Hill, Singapore, 1984.

4. C. J. Geankoplis, "Transport Processes and Unit Operations", 3 rd ed., Printice-Hall, Inc. , New Jersey, 1993.

DATA SHEET

a) **Continuous Operation**

Feed Rate ; F=   **Power; W=**

	Sample	x_B (mol%)	x_D (mol%)	T1(^oC)	T2(^oC)	T3(^oC)
Reflux ratio :	1					
	2					
Reflux ratio :	1					
	2					
Reflux ratio :	1					
	2					

b) **Batch Operation**

Total Reflux

Power (kW)	Boil-up rate (ml/min)	Pressure drop (cm H_2O)	Degree of foaming *	T1 (^oC)	T2 (^oC)	T3 (^oC)	x_B (% mol)	x_D (% mol)

* None, Gentle localised, Violent localised, Foaming gently over whole tray, Foaming violently over whole tray, Liquid flooding in the column

Reflux ratio: **W=**

t (min)	T1 (°C)	T2 (°C)	T3 (°C)	x_B (% mol)	x_D (% mol)