Flash distillation

1. Theoretical background

Flash distillation is a special operation within distillation, where a liquid mixture is heated up and fed – with constant flowrate – into a distillation equipment. The resulting vapor and liquid phases enter a phase separator – an equilibrium chamber – and are drained separately. During the operation, the total pressure and temperature of the system, as well as the compositions of the two phases in equilibrium remain constant over time.

One implementation of this operation is shown in Figure 1.

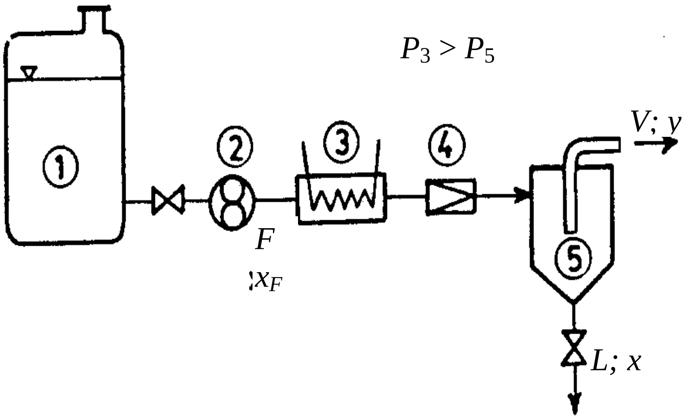

Figure 1
Flash distillation

The mixture to be separated is fed from the feed tank (1) by a pump (2) through a heat exchanger (3) at pressure P_3. Here, it is heated above the boiling point it would have at P_5, the pressure inside the phase separator (5). The pressure of the mixture is then decreased by flowing it through a valve (4), so that it partially evaporates and yields a vapor and liquid phase with equilibrium compositions.

Based on Figure 1, the following balance equations can be obtained:
mass balance

$$F = L + V \tag{Eq.1}$$

component balance:

$$Fx_F = Lx + Vy \tag{Eq.2}$$

All necessary calculation can be carried out by using balance equations (Eq.1) and (Eq.2). The balance equations are valid whether using molar flows or mass flows.

Another implementation of flash distillation is feeding a mixture of quantity F and composition x_F into a heated tank, yielding a liquid phase of quantity L and composition x, and a vapor phase of quantity V and composition y (which is the equilibrium composition for liquid phase composition x). (Eq.1) and (Eq.2) apply in this case as well, thus all results are calculated with the same methods. This second implementation requires a startup period, which will be explained in detail later.

1.1. Calculation of flash distillation

The vapor phase composition y expressed from (Eq.1) and (Eq.2) yields the following equation:

$$y = -\frac{L}{V}x + \frac{F}{V}x_F \qquad \text{(Eq.3)}$$

Figure 2 shows (Eq.3) and the equilibrium curve.

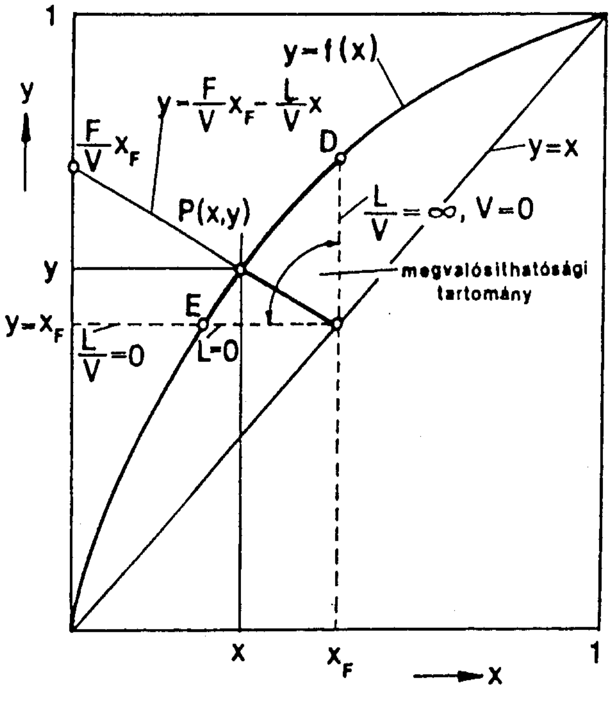

Figure 2
Equilibrium diagram for flash distillation

To calculate this operation, feed quantity and composition (F, x_F) and either the liquid and vapor quantities (L, V) or the liquid composition x (and potentially, vapor composition y) is always given. Based on (Eq.3), the intersection point between the line starting from point $x = x_F$ with gradient $-L/V$ and the equilibrium curve yields point P (x, y) which describes the vapor and liquid phase compositions leaving the equilibrium chamber.

One extreme condition that can be derived from the Figure 2 is when the entire feed is evaporated ($L = 0$; $F = V$; $L/V = 0$), in this case the vapor and feed compositions are equal ($y = x_F$). The other extreme case is when there is no vapor phase ($V = 0$; $F = L$; $L/V = \infty$), in this case the liquid and feed compositions are equal ($x = x_F$). The ratio between the liquid and vapor phases obtained with flash distillation can be changed via the degree of evaporation (heating).

Flash distillation is used in practice for components with large relative volatility, or if the separation based on the vapor–liquid equilibrium (distillation) can be combined with a physical separation based on a liquid–liquid equilibrium.

1.2. Flash distillation with startup period

The startup period of flash distillation can be implemented in two ways:

a.) The feed, with quantity L_0 and composition x_0 is fed into the distillation tank and batch distilled until the desired residue composition x_w is reached. The feed input and residue draining is then started, and heating is controlled so that the vapor and residue compositions are constant.

b.) Feed with quantity F and composition x_F is fed into the distillation tank without draining residue, with the distillate flowrate being equal to the feed flowrate $F = V$. Since the concentration of the more volatile compound is always greater in the distillate than in the tank, the concentration in the tank decreases over time. When the concentration in the tank reaches the required concentration x, the feed flowrate and heating is increased so that residue with composition x and distillate with equilibrium composition y can be drained from the tank.

1.2.1. Calculating startup time

For case b), startup time is calculated with condition $F = V$. This can be easily implemented by maintaining a constant fluid level in the tank using an overflow drain. The entire operation can be thus considered a batch distillation with refill.

In the startup period, the component balance for the operation is:

$$Fx_F - Vy_{(x)} = L_0 \frac{dx}{dt} \qquad \text{(Eq.4)}$$

where F feed flowrate, kg/h
V distillate flowrate, kg/h
$x_F = x_0$ feed concentration, mass fraction
$y_{(x)}$ distillate concentration changing over time, mass fraction

Considering the startup period condition $F = V$ rearranging and integrating the differential equation (Eq.4) yields:

$$\int_0^t dt = t = -\frac{L_0}{F}\int_{x_F}^{x} \frac{dx}{y_{(x)} - x_F} \qquad \text{(Eq.5)}$$

(Eq.5) can be integrated graphically using a $\frac{1}{y_{(x)} - x_F} - x$ diagram. (Eq.5) shows that given a constant feed stream, startup time is shorter for smaller amounts of initial mixture L_0; and for a given amount of initial mixture L_0, startup time can be reduced by increasing the feed stream flowrate (F).

2. Description of the experimental apparatus

The schematic of the apparatus is shown in Figure 3.

The apparatus consists of a feed tank (1), a flow control tap (2), a capillary flow meter (3), an internally heated flask (4), the residue cooler (5) and distillate cooler (6).

The residue and distillate are collected in flasks. The distillate forms two phases, these will be separated in a separation funnel.

3. Description of the measurement

The aim of this experiment is the recovery of ethyl acetate from an aqueous solution saturated with ethyl acetate. The solubility and vapor–liquid equilibrium data for the ethyl acetate–water system are shown in Figure 4.

Ethyl acetate and water have limited miscibility, thus the condensed vapors cooled to room temperature typically form two phases. The enrichment due to distillation is further amplified by the separation of the distillate. In this case, the industrial approach is to drain the ethyl acetate phase as the final product. Since the composition of the water phase composition usually equals the feed composition, it is recycled into the distillation tank.

Due to the phase separation, (Eq.1) and (Eq.2) must be augmented with the balance equations of the phase separator (Florence flask).

Mass balance of the phase separator:

$$V = L_1 + L_2 \qquad \text{(Eq.6)}$$

Component balance of the phase separator:

$$Vy = L_1 x_1 + L_2 x_2 \qquad \text{(Eq.7)}$$

In (Eq.6) and (Eq.7) L_1 denotes the amount of the ethyl acetate phase, L_2 denotes the amount of the water phase (both in kg/h), x_1 denotes the composition of the etyl acetate phase, x_2 denotes the composition of the water phase (both in mass fraction). Typically, $x_2 = x_0 = x_F$.

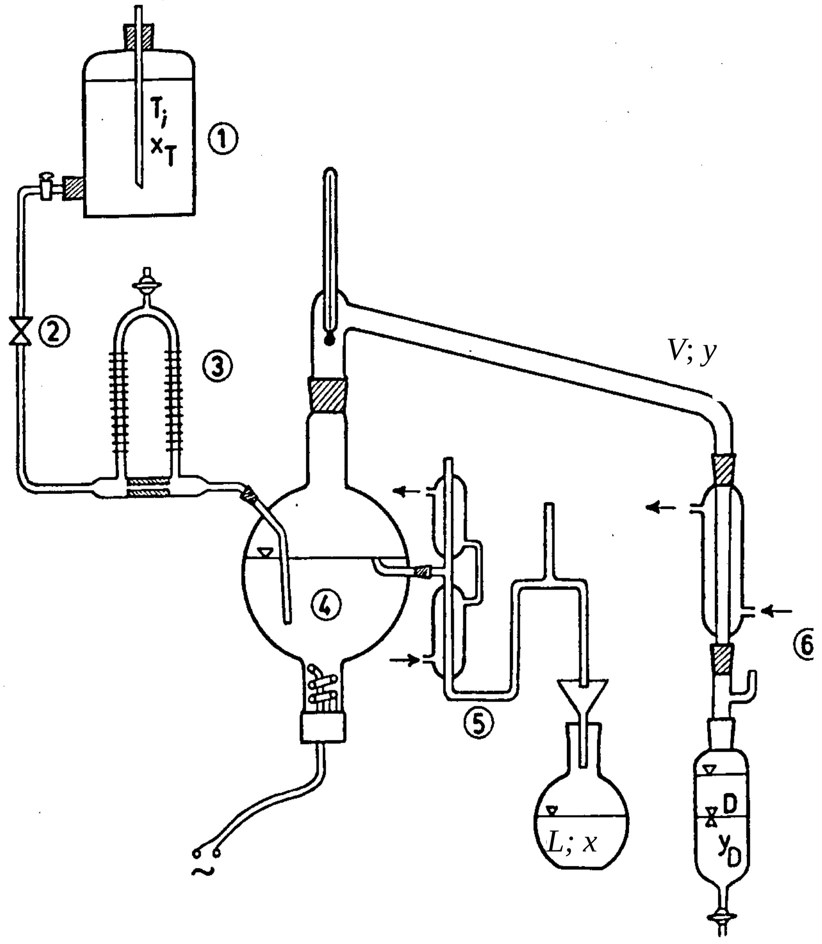

Figure 3

3.1. Measuring startup time

Flask 4 contains water saturated with ethyl acetate. First, turn on the cooling water, then start the internal heating coils with a heating voltage of 150 V. When boiling begins, adjust the heating voltage to between 100 and 120 V (the exact value will be specified by the instructor), and measure the startup time with a stopwatch starting when the first drop appears. Adjust the feed such that the level inside the boiler does not change ($F = V$), typically around 5–10 ml/min. The startup time ends when 90 °C is reached.

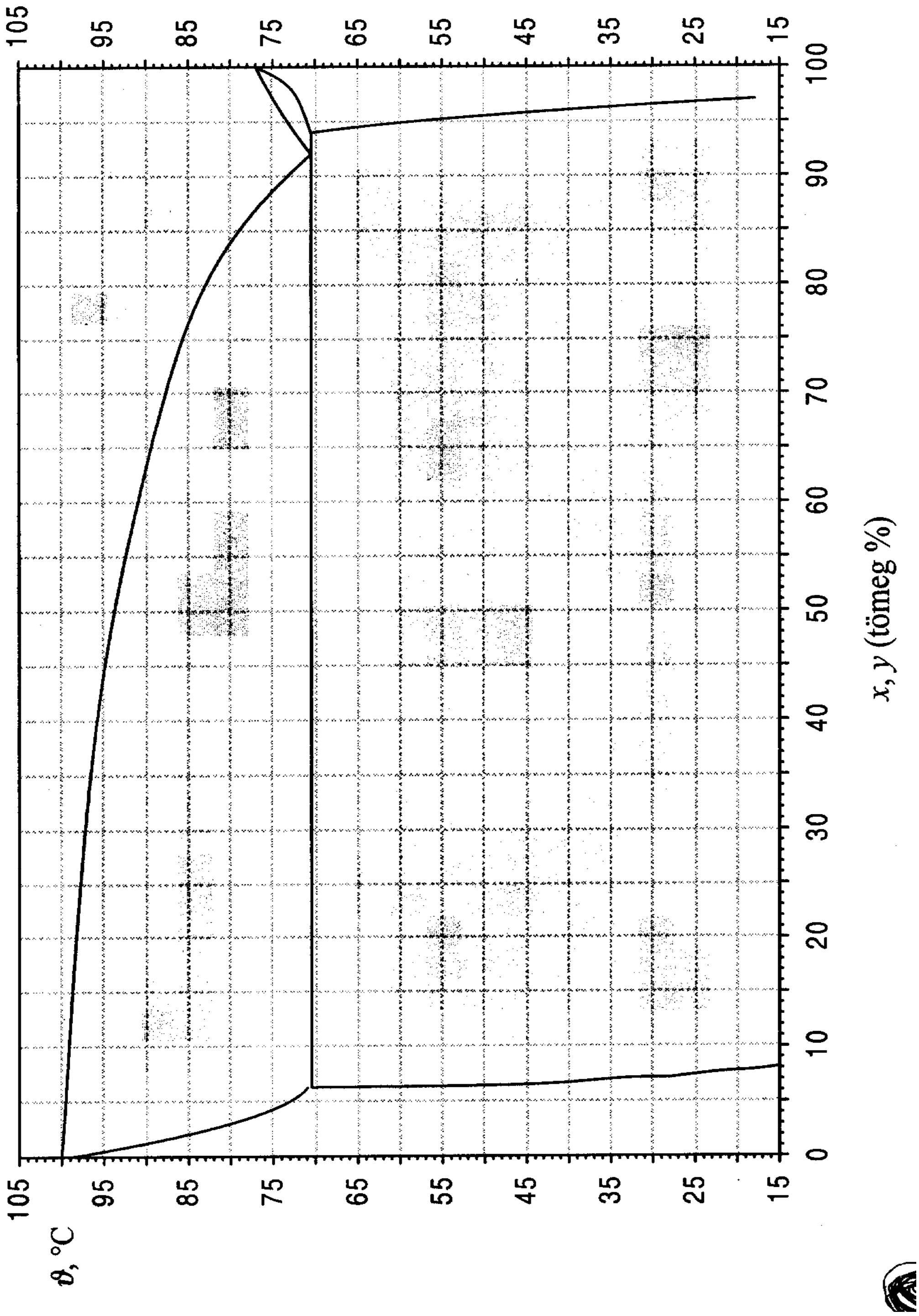

Figure 4
Equilibrium diagram of the water–ethyl acetate system

3.2. Measurement in steady state

When the boiling point reaches 90 °C, increase the feed by 8–10 times so that the boiling point stabilizes between 87 and 93 °C. A constant boiling temperature indicates that steady state has been reached. At this point, take clean collector flasks and measure in steady state for 20 minutes. Decrease the feed if the temperature dereases, increase it if the temperature increases. After 20 minutes, measure the mass of the residue and determine its concentration by refractometry. Measure the mass of the two phases of the distillate at room temperature and determine their concentration from the equilibrium diagram. The exact mass of the feed is given by the sum of the residue and distillate masses. The capillary flow meter is only used to maintain a constant feed rate.

Laboratory report:

a.) Startup period
initial mixture $L_0 =$ g
heating voltage $U =$ V
feed temperature $\vartheta =$ °C
(room temperature)
feed composition based on temperature $x_F =$ tömeg %
temperature at the end of startup $\vartheta =$ °C
measured startup time $t =$ min

b.) Steady state
measurement time $t =$ min
heating voltage $U =$ V
feed according to flow meter $F =$ g/min
boiling point $\vartheta =$ °C
distillate temperature $\vartheta =$ °C
feed refractive index $n_D^{20} =$

component balance

Distillate				Residue	
upper phase		lower phase			
L_1 g	x_1 mass%	L_2 g	x_2 mass%	L g	x mass%

To be handed in:

1.) The measure startup time, compared with that calculated from (Eq.5).

2.) Error of the component balance according to the following equation:

$$\Delta\ \% = \frac{Fx_F - (Vy + Lx)}{Fx_F} \cdot 100 = \qquad \text{(Eq.8)}$$

In (Eq.8) the average distillate composition y can be calculated from (Eq.6) and (Eq.7).