Examples

Liquid- Liquid- Extraction

Lecturer: Thomas Gamse
ao.Univ.Prof.Dipl.-Ing.Dr.techn.

Department of Chemical Engineering and Environmental Technology

Graz University of Technology

Inffeldgasse 25, A-8010 Graz

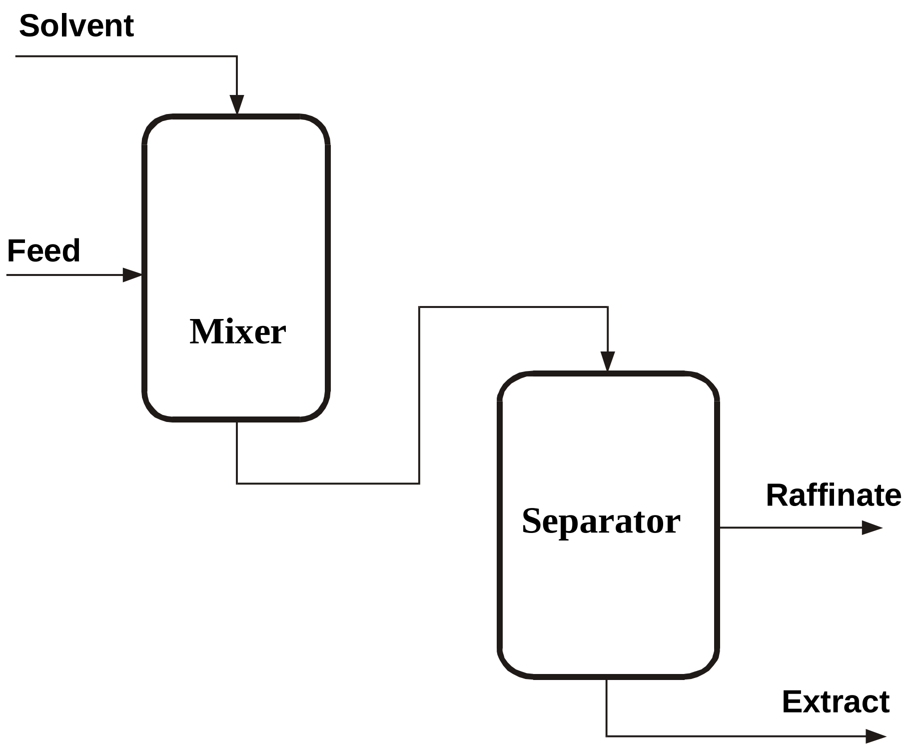
Tel.: ++43 316 873 7477

Fax: ++43 316 873 7472

email: Thomas.Gamse@TUGraz.at

Examples Liquid - Liquid Extraction

Nominating of the flows



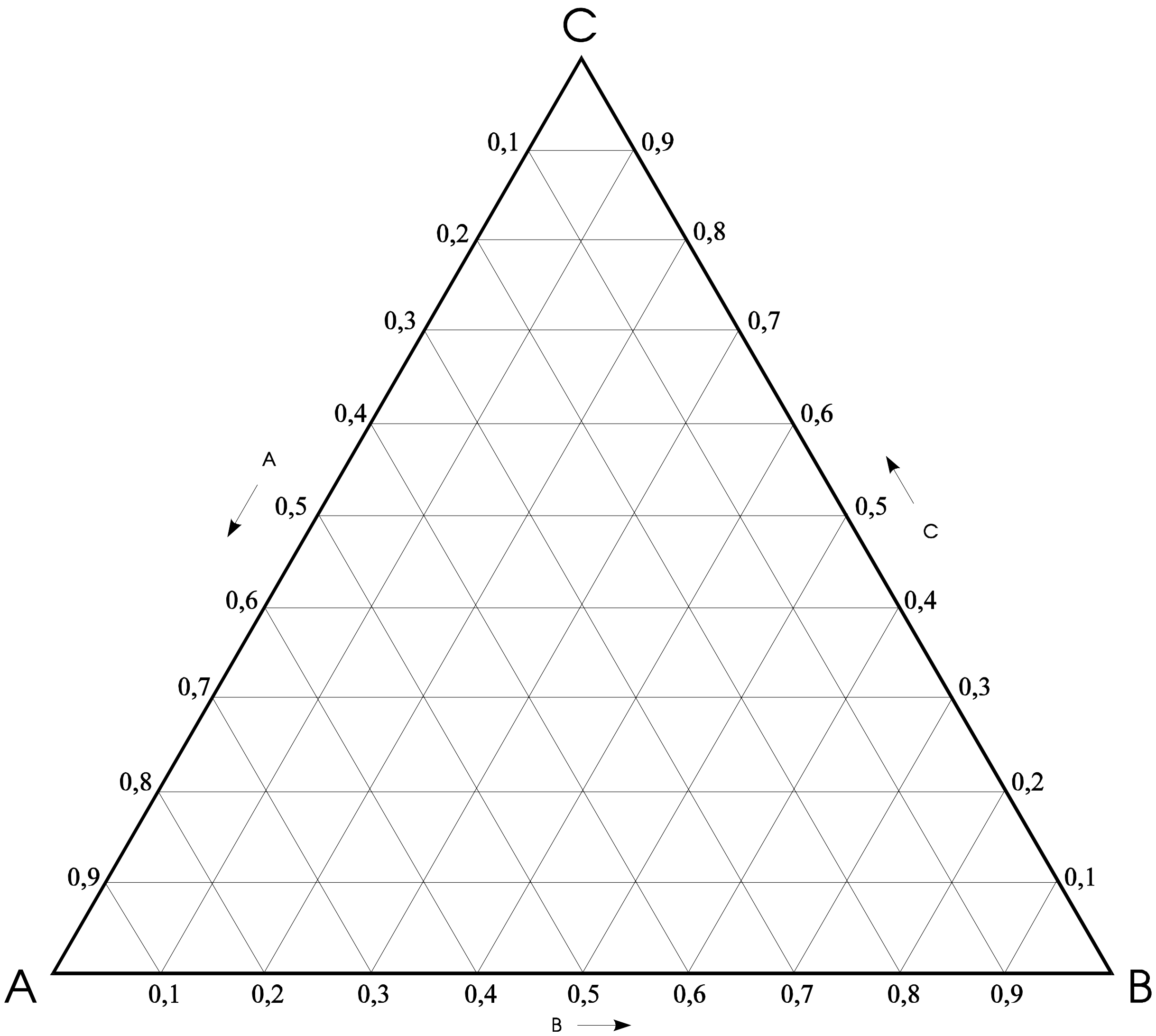
Example 1: Ternary Systems, Triangle Diagram

Two mixtures *R* and *E*, which contain both the three compounds *A*, *B*, and *C*, have to mixed in the ratio 1:2. This ternary system has no miscibility gap so that all compounds are completely soluble each other. The mixture *R* has a composition of $x_{A,R} = 0{,}7$ and $x_{B,R} = 0{,}2$; the mixture *E* exists of $x_{A,E} = 0{,}1$ and $x_{B,E} = 0{,}5$.

Please determine:

a) The points *R* and *E* in the triangle diagram and the concentration of the active agent *C* and

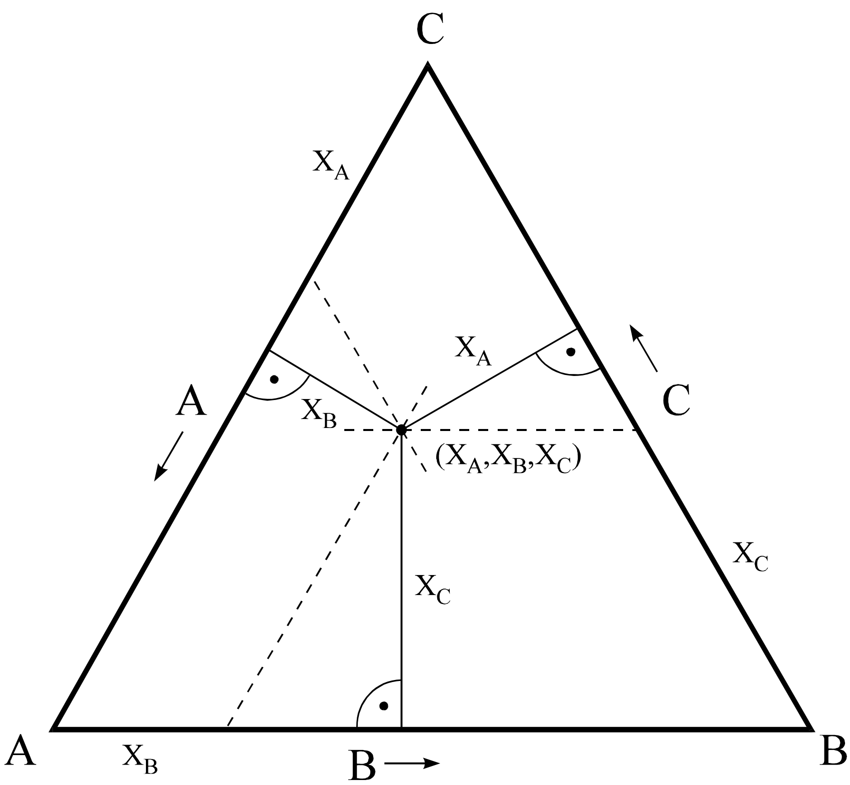
b) the mixing point (calculation and graphical determination).



a.) Mixtures in the triangle diagram

For a partial solubility of substances *A* and *B*, which is essential for extraction, all three compounds have to be taken into account for drawing of the phase equilibrium. For this reason triangle co-ordinates are used, where each of the vertexes represents the pure compounds. Points at the triangle side represent the composition of the binary system and points inside the triangle the composition of the ternary system.

The representation of a ternary point is based on the fact that the sum of the normal distances in a equal sided triangle is corresponding to the height of the triangle. If the height of the triangle is set 100% so result the concentrations of the single compounds from the normal distances (see figure).



The given points *R* and *E* can therefore be drawn in the diagram.

From these points the concentration of C can be determined.

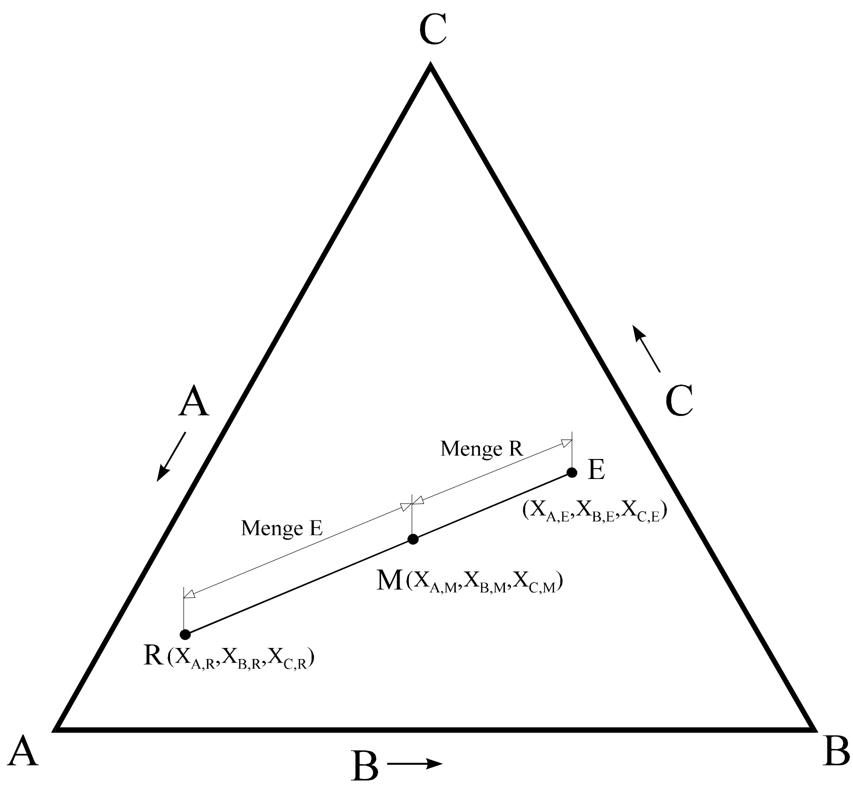
$x_{C,R} = 0{,}1$	$x_{C,E} = 0{,}4$

(Control: The sum of the components *A*, *B* and *C* must be equal 1)

The triangle diagram can also be given in weight percent *wt%*.

b) Mixing point

If two mixtures with given composition in the triangle diagram are mixed then the resulting mixing point lays on the connection line between these two points. The position of the mixing point can be calculated by a mass balance or graphically by the use of the law of balance.



Calculating:

Total balance:

$$R + E = M$$

Mass balance for compound C:

$$R \cdot x_{C,R} + E \cdot x_{C,E} = M \cdot x_{C,M} \quad \rightarrow$$

$$x_{C,M} = \frac{R \cdot x_{C,R} + E \cdot x_{C,E}}{R + E}$$

with $\frac{R}{E} = \frac{1}{2}$ the mass R and E can be eliminated, which results in

$$x_{C,M} = \frac{0{,}5 \cdot x_{C,R} + x_{C,E}}{1{,}5}$$

or $x_{C,M} = \frac{0{,}5 \cdot 0{,}1 + 0{,}4}{1{,}5}$

$$\boxed{x_{C,M} = 0{,}3}$$

Analogous results therefore

$\boxed{x_{A,M} = 0{,}3}$ and $\boxed{x_{B,M} = 0{,}4}$

Graphically:

Law of balance:

$$\frac{\overline{RM}}{\overline{ME}} = \frac{E}{R}$$

with $\frac{R}{E} = \frac{1}{2}$ follows $\frac{\overline{ME}}{\overline{RM}} = \frac{1}{2}$

From the diagram the length of the distance $\overline{RE}$ can be determined with 77 mm.

$$\overline{RE} = \overline{RM} + \overline{ME} \quad \Rightarrow \quad 0{,}5 = \frac{77 - \overline{RM}}{\overline{RM}}$$

$$\overline{RM} = 51{,}3\,\text{mm}$$

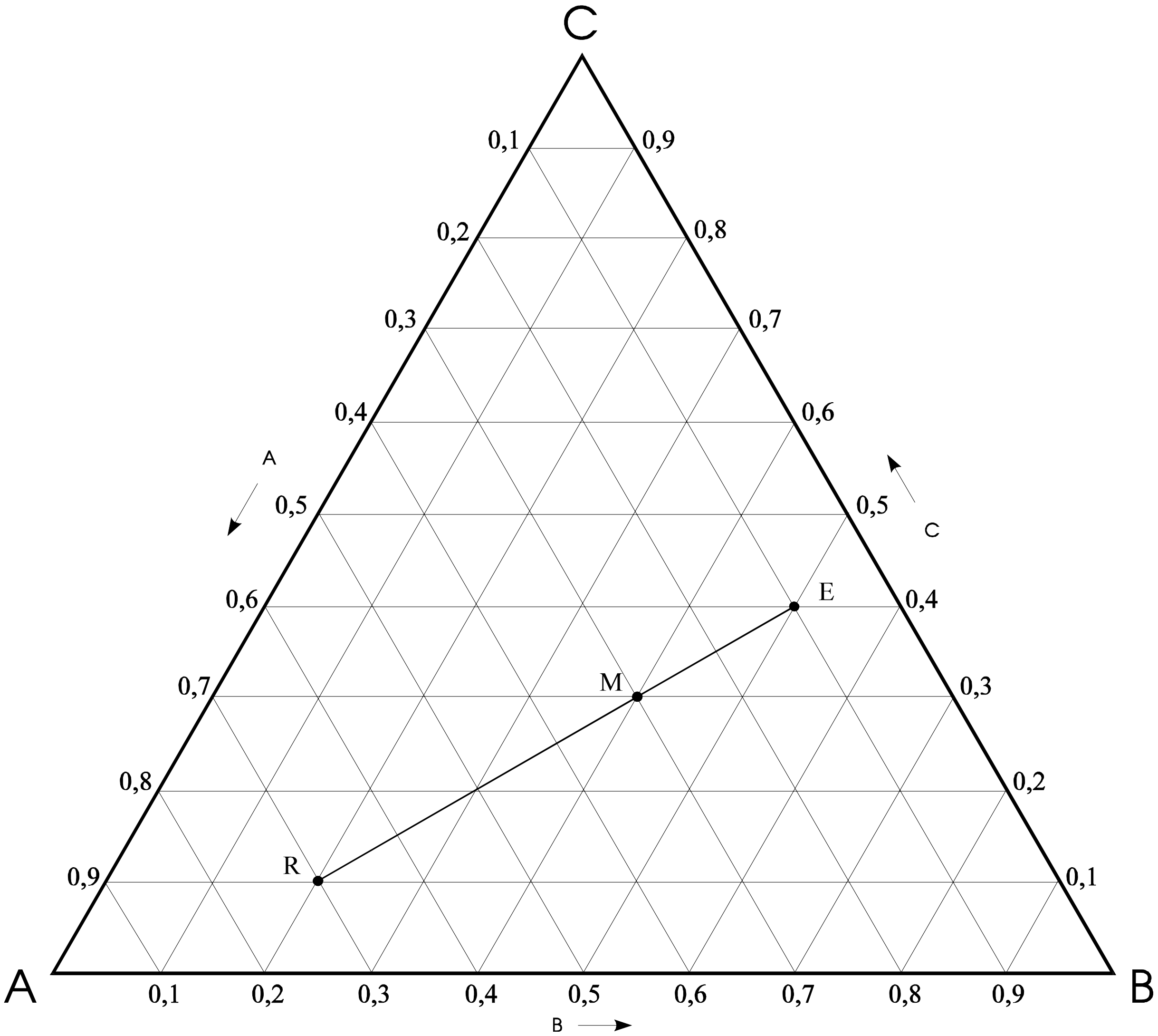
Drawing this length in the diagram results the mixing point *M* and the concentrations of the compounds can be determined.

$$\boxed{x_{A,M} = 0{,}3}$$

$$\boxed{x_{B,M} = 0{,}4}$$

$$\boxed{x_{C,M} = 0{,}3}$$

Triangle diagram



Example 2: Ternary system with mixing gap

A waste water from a process is loaded with acetone, which should be extracted with chlorobenzene.

The equilibrium data for the ternary system water / acetone / chlorobenzene are given.

composition of the coexisting phases in equilibrium in wt%					
water phase			organic phase		
water	acetone	chlorobenzene	water	acetone	chlorobenzene
99,89	0,00	0,11	0,18	0,00	99,82
89,79	10,00	0,21	0,49	10,79	88,72
79,69	20,00	0,31	0,79	22,23	76,98
69,42	30,00	0,58	1,72	37,48	60,80
58,64	40,00	1,36	3,05	49,44	47,51
46,28	50,00	3,72	7,24	59,19	33,57
27,41	60,00	12,59	22,85	61,07	15,08
25,66	60,58	13,76	25,66	60,58	13,76

You have to determine:

a.) the triangle diagram including the phase equilibrium line and connodes.

b.) The water and chlorobenzene content of the aqueous phase (raffinate) with an acetone concentration of 45 % and of the coexisting phase.

c.) Which amount of acetone has to be added to a mixture, existing of 110 g chlorobenzene and 90 g water? What is the composition of the mixing point?

d.) What is the water free composition of this mixing point?

a) Construction of the phase equilibrium in the triangle diagram

The given ternary system ha a mixing gap which separates the system in a homogeneous one phase region and a heterogeneous two phase region. The boundary is the binodal curve.

In the for the extraction interesting heterogeneous region a mixture splits in raffinate and extraction phase along a connode, which connects the two coexisting phases. The higher the amount of the active agent (extractable substance *C*) is the shorter the connodes become until they melt to one point, the critical point *K*. By this critical point *K* the binodal curve is split into two parts. Normally the part on the left side represents the raffinate phase *R*, which has a low content of solvent *B*, and the right side represents the solvent rich extract phase *E*.

According to the given table the coexisting phases (connodes) are given which can now be drawn in the triangle diagram. One line in the table corresponds to one connode.

1.connode point of the raffinate phase:

$w_{A,R} = 0{,}9989$ $w_{B,R} = 0{,}0011$ $w_{C,R} = 0{,}0$

1.connode point of the extract phase:

$w_{A,E} = 0{,}0018$ $w_{B,E} = 0{,}9982$ $w_{C,E} = 0{,}0$

Connecting these two points gives the first connode and analogous for the other given data. The last row corresponds to the critical point *K*. By connecting all raffinate and all extract points the result is the binodal curve.

b) Raffinate phase / Extract phase

Point in the raffinate phase

Drawing the acetone concentration of $w_{C,R} = 0{,}45$ on the right side of the triangle for the active agent *C* and crossing this with the binodal curve at the left side gives the point of the aqueous phase, so that the concentrations of water and chlorobenzene can be determined.

$w_{A,R} = 0{,}535$ $w_{B,R} = 0{,}015$

Point of the extract phase

The point of the extract phase has to be on a connode going through the already determined point on the raffinate side. But this connode is not given and has to be constructed.

Possibility 1:

By interpolation between the two connodes next to the point the connode through the given point can be constructed, but in a very inaccurate way.

Possibility 2:

With the help of the conjugation line the connode can be determined better and with higher accuracy. For this purpose the right and left triangle side has to be shifted parallel through the points of the connodes and the crossing of these two lines represents one point of the conjugation line. All these by this way constructed point and the critical point have to be connected to the conjugation line.

The searched coexisting phase can be constructed analogous: parallel shifting of the right triangle side through *R*, crossing with the conjugation line and crossing of the parallel shifted left triangle side through the point on the conjugation line with the right side of the binodal curve.

The by this way determined concentrations are:

$w_{A,E} = 0{,}04$ $w_{B,E} = 0{,}41$ $w_{C,E} = 0{,}55$

c) mixture near the phase boundary

First the binary mixture has to drawn at the basic side of the triangle diagram.

	mass [g]	weight-%
chlorobenzene	110	55
water	90	45
Σ	200	100

This point of the binary mixture has to be connected with the point *C*, pure acetone, and somewhere on this line the mixing point must be. The boundary between one and two phase region is the binodal curve. Therefore the searched mixing point *M* can be determined by crossing the line *GC* with the binodal curve. The necessary amount of acetone can be determine by the law of balance.

$$\overline{CM} = 40\,\text{mm} \qquad \overline{MG} = 69\,\text{mm} \qquad \overline{CG} = 109\,\text{mm}$$

$$\frac{G}{C} = \frac{\overline{CM}}{\overline{MG}} \rightarrow \frac{200}{C} = \frac{40}{69}$$

$$C = 345g$$

Composition of the mixing point *M*:

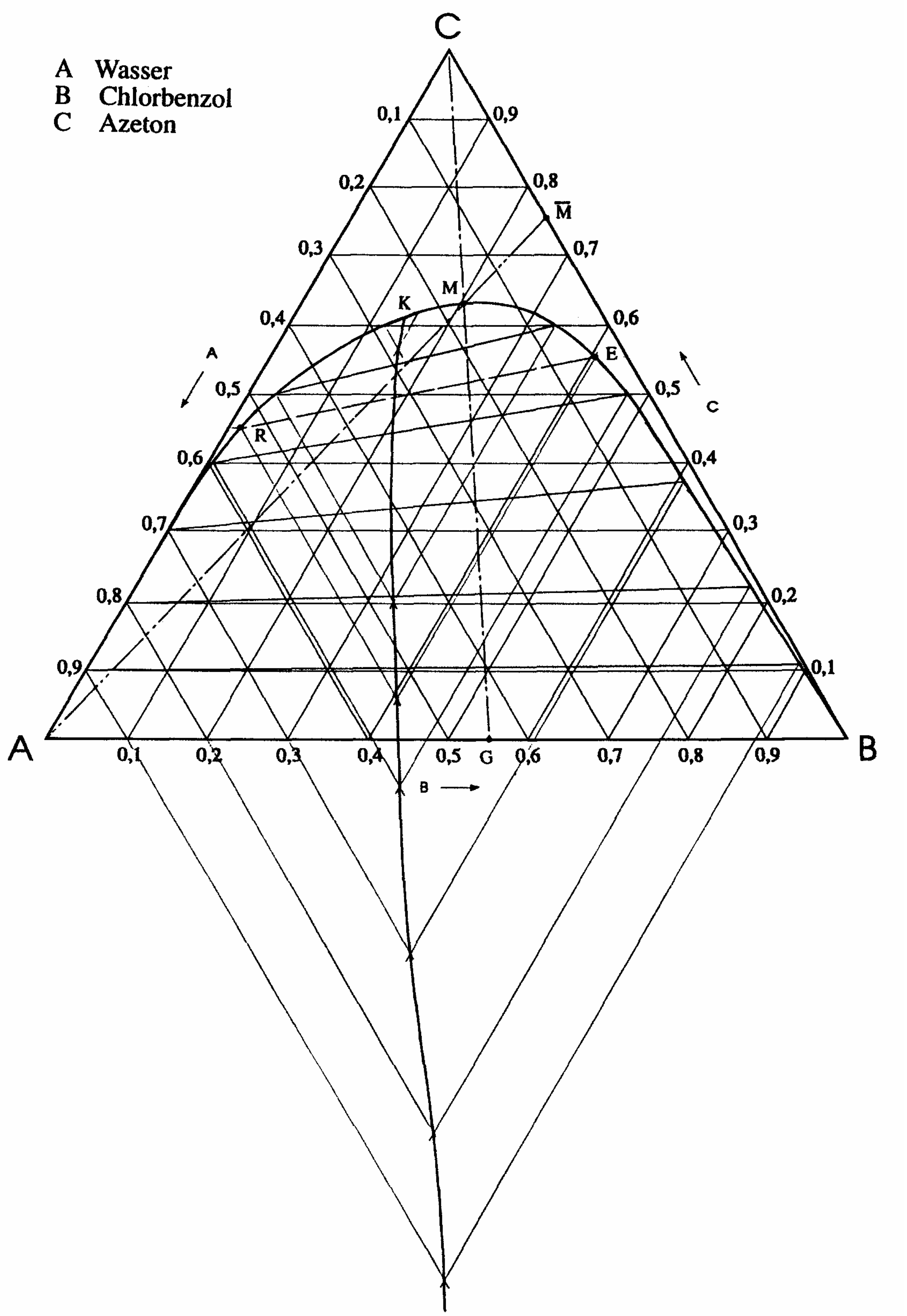
$$w_{A,M} = 0{,}16 \qquad w_{B,M} = 0{,}21 \qquad w_{C,M} = 0{,}63$$

d) water free mixing point:

To get the water free mixing point, the edge point *A* and the mixing point *M* hove to be connected and this line has to be prolonged to the right side of the triangle diagram, the water free side. The composition of the binary, water free mixture of acetone and chlorobenzene is:

$$\boxed{w_{B,\overline{M}} = 0{,}25} \qquad \boxed{w_{C,\overline{M}} = 0{,}75}$$

Triangle diagram



Example 3: Single Step Extraction

The basic mixture of 100 kg exists of 40 mole% acetone and 60 mole% water and has to be extracted with trichloroethane, which is preloaded with 15 mole% acetone. Your have to determine:

a) the phase diagram of the system acetone / water / trichloroethane in the triangle diagram.
b) the minimum and maximum amount of solvent,
c) the necessary amount of solvent, if the raffinate contains 4,82 mole% acetone,
d) the amount and composition of the produced raffinate and extract,
e) the extraction process in the triangle diagram

Phase equilibria data for the system water / acetone / trichloroethane

phase equilibria data for the coexisting phases in mole%					
extract phase			raffinate phase		
trichloroethane	acetone	water	trichloroethane	acetone	water
80,18	17,7	2,	0,07	1,9	97,9
59,01	35,9	5,	0,10	4,8	95,0
49,17	44,0	6,	0,12	6,8	93,0
35,99	53,7	10,2	0,17	10,3	89,4
25,04	58,3	16,6	0,29	14,9	84,7
14,56	56,9	28,4	0,78	21,9	77,2
9,94	52,4	37,5	1,50	27,3	71,1

a) Phase equilibrium in the triangle diagram

Drawing of the single connode points analogous to example 2 and constructing the connodes and combining the single point to the binodal curve.
The critical point was not drawn because the composition is not given and therefore the exact position is not defined.

b) minimal / maximal amount of solvent

Drawing of feed and solvent

feed:

$$x_{C,F} = 0{,}4 \quad x_{A,F} = 0{,}6$$

The point F is on the left triangle side (binary mixture).

solvent:

$$x_{C,L} = 0{,}15 \quad x_{B,L} = 0{,}85$$

The point L is on the right triangle side (binary mixture).

The mixing point M has to be on the line between these two points F and L and M has to be in the two phase region, because for extraction the mixture has to separate in two phases. The minimal and maximal amount of solvent (M_{min} and M_{max}) are the two crossings of the connection line $\overline{FL}$ with the binodal curve. By the length, which can be determined from the diagram, the searched amounts can be calculated.

Minimal amount of solvent:

$$\overline{FL} = 91{,}5\,\mathrm{mm}$$

and

$$\overline{FM_{\mathrm{min}}} = 4\,\mathrm{mm}$$

law of balance:

$$\frac{\overline{FM_{\mathrm{min}}}}{\overline{M_{\mathrm{min}}L}} = \frac{M_{\mathrm{min}}}{F} \rightarrow M_{\mathrm{min}} = F \cdot \frac{\overline{FM_{\mathrm{min}}}}{\overline{M_{\mathrm{min}}L}} = 100 \cdot \frac{4}{91{,}5 - 4}$$

$$\boxed{M_{\mathrm{min}} = 4{,}57\,\mathrm{kg}}$$

Maximal amount of solvent:

$$\overline{M_{max}L} = 2\,\text{mm}$$

law of balance:

$$\frac{\overline{FM_{max}}}{\overline{M_{max}L}} = \frac{M_{max}}{F}$$

$$M_{max} = F \cdot \frac{\overline{FM_{max}}}{\overline{M_{max}L}} = 100 \cdot \frac{91{,}5 - 2}{2}$$

$$\boxed{M_{max} = 4.475\,\text{kg}}$$

c) effective amount of solvent

The acetone concentration of the produced raffinate *R*, which has to be on he binodal curve, must be 4,82 %. With the connode going through this point *R* the extract *E* is fixed.

The mixing point *M* of feed *F* and solvent *L* is the crossing of the connode *RE* with the connection line $\overline{FL}$. With the law of balance the necessary amount of solvent *L* can be calculated.

$$\overline{ML} = 45{,}5\,\text{mm}$$

law of balance:

$$\frac{\overline{FM}}{\overline{ML}} = \frac{L}{F}$$

$$L = F \cdot \frac{\overline{FM}}{\overline{ML}} = 100 \cdot \frac{91{,}5 - 45{,}5}{45{,}5}$$

$$\boxed{L = 101{,}1\,\text{kg}}$$

d) composition and amount of raffinate and extract

Raffinate R:

$\boxed{x_{A,R} = 0{,}9508}$ $\boxed{x_{B,R} = 0{,}001}$ $\boxed{x_{C,R} = 0{,}0482}$

Extract E:

$\boxed{x_{A,E} = 0{,}0503}$ $\boxed{x_{B,E} = 0{,}5901}$ $\boxed{x_{C,E} = 0{,}3596}$

amount of raffinate:

total balance: $E + R = F + L = 100 + 101{,}1 = 201{,}1\,\text{kg}$

law of balance: $\frac{\overline{RM}}{\overline{ME}} = \frac{E}{R}$

$\overline{RE} = 95\,\text{mm}$ $\overline{ME} = 26\,\text{mm}$

$$R = \frac{201{,}1}{\frac{95 - 26}{26} + 1}$$

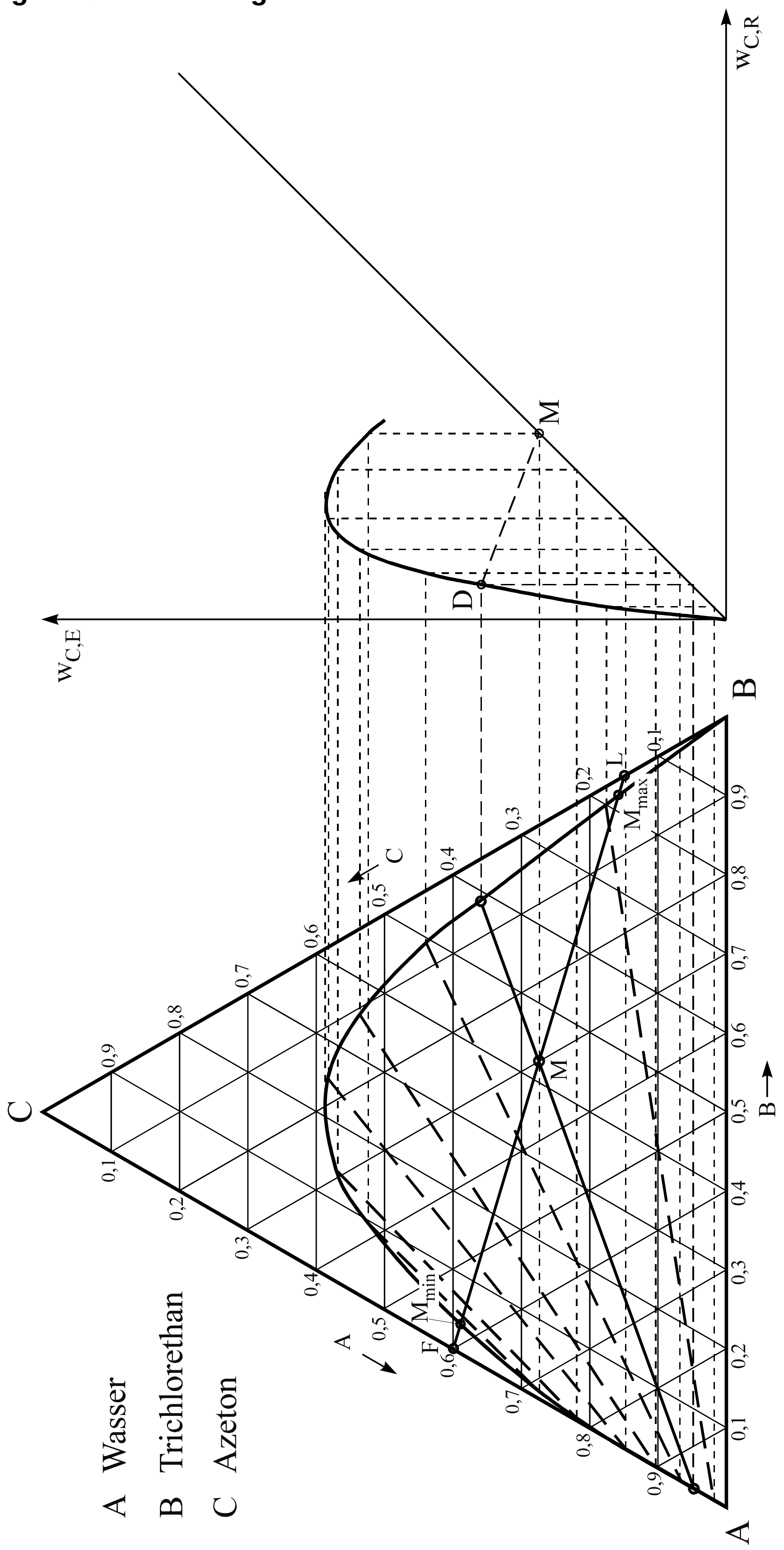
$$\boxed{R = 55\,\text{kg}}$$

amount of extract:

$$E = F + L - R$$

$$\boxed{E = 146{,}1\,\text{kg}}$$

Triangle Diagram / Nernst Diagram



Example 4: Multi Step Cross Flow Extraction

From 2.000 kg/h of an acetic acid / water mixture with 45 wt% acetic acid the acetic acid has to be extracted by a multi step cross flow extraction at an operation temperature of 20°C. The residual concentration of acetic acid has to be 10 wt% and the used solvent isopropyl ether is free of acetic acid.

You have to determine:

a) the minimum amount of solvent for the first extraction step

b) the necessary number of theoretical steps in the triangle diagram for the case that a solvent ration $\dot{L}/\dot{F}_S$ of 1 is chosen and in every step the same amount of solvent is added.

Phase equilibria data

extract phase			raffinate phase		
acetic acid	**water**	**isopropyl ether**	**acetic acid**	**water**	**isopropyl ether**
0,002	0,005	0,993	0,007	0,981	0,012
0,004	0,007	0,989	0,014	0,971	0,015
0,008	0,008	0,984	0,029	0,955	0,016
0,019	0,010	0,971	0,064	0,917	0,019
0,048	0,019	0,933	0,133	0,844	0,023
0,114	0,039	0,847	0,255	0,711	0,034
0,216	0,069	0,715	0,367	0,589	0,044
0,311	0,108	0,581	0,443	0,451	0,106
0,362	0,151	0,487	0,464	0,371	0,165

a) minimum amount of solvent

For the liquid - liquid extraction at cross flow method the feed $\dot{F}$ enters the first extraction step, where it is contacted with solvent $\dot{L}$. The extraction results in a raffinate $\dot{R}$ and a extract $\dot{E}$. The extract is withdrawn while the raffinate enters the next step where it is contacted with fresh solvent again and so on.

In the single steps equilibrium between raffinate and extract is reached so that the compositions can be determined in the triangle diagram.

First the equilibrium data have to be drawn and the binodal curve with the given connodes has to be constructed.

Then the point of the feed *F* and of the solvent *L* is drawn. The mixing point M_1 has to be on the connection line $\overline{FL}$. For the minimum amount of solvent the crossing point $M_{\min}$ at the raffinate side of the binodal curve is significant. From the law of balance results :

$$\frac{\dot{L}_{\min}}{\dot{F}} = \frac{\overline{FM_{\min}}}{\overline{M_{\min}L}} = \frac{w_{C,F} - w_{C,\min}}{w_{C,\min} - w_{C,L}} = \frac{0{,}45 - 0{,}415}{0{,}415 - 0} = 0{,}0843$$

With the amount of feed results the *minimum amount of solvent*:

$$\dot{L}_{\min} = \dot{F} \cdot 0{,}0843 = 2.000 \cdot 0{,}0843 = 168{,}6\,\mathrm{kg/h}$$

b) number of steps in the triangle diagram

The ratio of solvent of feed is given with $\frac{\dot{L}}{\dot{F}_S} = 1$

1. step

$$\dot{F}_S = \dot{F} \cdot \left(1 - w_{C,F}\right) = 2.000 \cdot (1 - 0{,}45) = 1.100\,\mathrm{kg/h}$$

With this follows:

$$\frac{\dot{L}}{\dot{F}} = \frac{1.100}{2.000} = 0{,}55$$

The mixing point M_1 of the first step can be determined by calculation or graphically.

<u>Calculation:</u>

$$\frac{\dot{L}}{\dot{F}} = \frac{\overline{FM_1}}{\overline{M_1L}} = \frac{w_{C,F} - w_{C,M_1}}{w_{C,M_1} - w_{C,L}} = \frac{0{,}45 - w_{C,M_1}}{w_{C,M_1} - 0} = 0{,}55 \qquad \Rightarrow w_{C,M_1} = 0{,}29$$

<u>Graphically:</u>

The length of $\overline{FL}$ is 173 mm and ha to divided according the ratio $\dot{L}/\dot{F} = 0{,}55$.

$$\overline{FM_1} + \overline{M_1L} = 173\,\text{mm}$$

$$0{,}55 \cdot \overline{M_1L} + \overline{M_1L} = 173\,\text{mm}$$

$$\overline{M_1L} = \frac{173}{1{,}55} = 111{,}6\,\text{mm}$$

Amount of the mixing point:

$$\dot{M}_1 = \dot{F} + \dot{L} = 2.000 + 1.100 = 3.100\,\text{kg/h}$$

The connode through the mixing point M_1 gives the extract E_1 and the raffinate R_1.

The according compositions can be taken from the diagram.

$$w_{C,E_1} = 0{,}21 \qquad\qquad w_{C,R_1} = 0{,}355$$

raffinate flow:

$$\frac{\dot{R}_1}{\dot{M}_1} = \frac{w_{C,M_1} - w_{C,E_1}}{w_{C,R_1} - w_{C,E_1}}$$

$$\dot{R}_1 = \frac{\dot{M}_1 \cdot \left(w_{C,M_1} - w_{C,E_1}\right)}{w_{C,R_1} - w_{C,E_1}} = \frac{3.100 \cdot (0{,}29 - 0{,}21)}{0{,}355 - 0{,}21} = 1.710{,}3\,\text{kg/h}$$

extract flow:

$$\dot{E}_1 = \dot{M}_1 - \dot{R}_1 = 3.100 - 1.710{,}3 = 1.389{,}7\,\text{kg/h}$$

2. step

For the second step the flow rate of the solvent isopropyl ether is also 1.100 kg/h.

$$\frac{\dot{L}}{\dot{R}_1} = \frac{\overline{R_1M_2}}{\overline{M_2L}} = \frac{1.100}{1.710{,}3} = 0{,}643$$

$$\overline{R_1M_2} + \overline{M_2L} = 167\,\text{mm}$$

$$0{,}643 \cdot \overline{M_2L} + \overline{M_2L} = 167\,\text{mm}$$

$$\overline{M_2L} = \frac{167}{1{,}643} = 101{,}6\,\text{mm}$$

amount of mixing point:

$$\dot{M}_2 = \dot{R}_1 + \dot{L} = 1.710{,}3 + 1.100 = 2.810{,}3\,\text{kg/h}$$

concentrations:

$$w_{C,E_2} = 0{,}14$$
$$w_{C,M_2} = 0{,}215$$
$$w_{C,R_2} = 0{,}29$$

raffinate flow:

$$\dot{R}_2 = \frac{\dot{M}_2 \cdot \left(w_{C,M_2} - w_{C,E_2}\right)}{w_{C,R_2} - w_{C,E_2}} = \frac{2.810{,}3 \cdot (0{,}215 - 0{,}14)}{0{,}29 - 0{,}14} = 1.405{,}15\,\text{kg/h}$$

extract flow:

$$\dot{E}_2 = \dot{M}_2 - \dot{R}_2 = 2.810{,}3 - 1.405{,}15 = 1.405{,}15\,\text{kg/h}$$

3. step

$$\frac{\dot{L}}{\dot{R}_2} = \frac{\overline{R_2M_3}}{\overline{M_3L}} = \frac{1.100}{1.405{,}15} = 0{,}783$$

$$\overline{R_2M_3} + \overline{M_3L} = 171\,\text{mm}$$

$$0{,}783 \cdot \overline{M_3L} + \overline{M_3L} = 171\,\text{mm}$$

$$\overline{M_3L} = \frac{171}{1{,}783} = 95{,}9\,\text{mm}$$

amount of mixing point:

$$\dot{M}_3 = \dot{R}_2 + \dot{L} = 1.405{,}15 + 1.100 = 2.505{,}15\,\text{kg/h}$$

concentrations:

$$w_{C,E_3} = 0{,}097 \qquad w_{C,M_3} = 0{,}16 \qquad w_{C,R_3} = 0{,}225$$

raffinate flow:

$$\dot{R}_3 = \frac{\dot{M}_3 \cdot \left(w_{C,M_3} - w_{C,E_3}\right)}{w_{C,R_3} - w_{C,E_3}} = \frac{2.505{,}15 \cdot (0{,}16 - 0{,}097)}{0{,}225 - 0{,}097} = 1233\,\text{kg/h}$$

extract flow:

$$\dot{E}_3 = \dot{M}_3 - \dot{R}_3 = 2.505{,}15 - 1.233 = 1272{,}15\,\text{kg/h}$$

4. step

$$\frac{\dot{L}}{\dot{R}_3} = \frac{\overline{R_3M_4}}{\overline{M_4L}} = \frac{1.100}{1233} = 0{,}892$$

$$\overline{R_3M_4} + \overline{M_4L} = 175\,\text{mm}$$

$$0{,}892 \cdot \overline{M_4L} + \overline{M_4L} = 175\,\text{mm}$$

$$\overline{M_4L} = \frac{175}{1{,}892} = 92{,}5\,\text{mm}$$

amount of mixing point:

$$\dot{M}_4 = \dot{R}_3 + \dot{L} = 1.233 + 1.100 = 2.333\,\text{kg/h}$$

concentrations:

$$w_{C,E_4} = 0{,}07 \qquad w_{C,M_4} = 0{,}117 \qquad w_{C,R_4} = 0{,}173$$

raffinate flow:

$$\dot{R}_4 = \frac{\dot{M}_4 \cdot \left(w_{C,M_4} - w_{C,E_4}\right)}{w_{C,R_4} - w_{C,E_4}} = \frac{2.333 \cdot (0{,}117 - 0{,}07)}{0{,}173 - 0{,}07} = 1.064{,}6\,\text{kg/h}$$

extract flow:

$$\dot{E}_4 = \dot{M}_4 - \dot{R}_4 = 2.333 - 1.064{,}6 = 1.268{,}4\,\text{kg/h}$$

5. step

$$\frac{\dot{L}}{\dot{R}_4} = \frac{\overline{R_4M_5}}{\overline{M_5L}} = \frac{1.100}{1.064{,}6} = 1{,}033$$

$$\overline{R_4M_5} + \overline{M_5L} = 180\,\text{mm}$$

$$1{,}033 \cdot \overline{M_5L} + \overline{M_5L} = 180\,\text{mm}$$

$$\overline{M_5L} = \frac{180}{2{,}033} = 88{,}54\,\text{mm}$$

amount of mixing point:

$$\dot{M}_5 = \dot{R}_4 + \dot{L} = 1.064{,}6 + 1.100 = 2.164{,}6\,\text{kg/h}$$

concentrations:

$w_{C,E_5} = 0{,}045 \qquad w_{C,M_5} = 0{,}084 \qquad w_{C,R_5} = 0{,}128$

raffinate flow:

$$\dot{R}_5 = \frac{\dot{M}_5 \cdot \left(w_{C,M_5} - w_{C,E_5}\right)}{w_{C,R_5} - w_{C,E_5}} = \frac{2.164{,}6 \cdot (0{,}084 - 0{,}045)}{0{,}128 - 0{,}045} = 1017{,}1\,\mathrm{kg/h}$$

extract flow:

$$\dot{E}_5 = \dot{M}_5 - \dot{R}_5 = 2.164{,}6 - 1017{,}1 = 1147{,}5\,\mathrm{kg/h}$$

6. step

$$\frac{\dot{L}}{\dot{R}_5} = \frac{\overline{R_5 M_6}}{\overline{M_6 L}} = \frac{1.100}{1017{,}1} = 1{,}082$$

$$\overline{R_5 M_6} + \overline{M_6 L} = 183\,\mathrm{mm}$$

$$1{,}082 \cdot \overline{M_6 L} + \overline{M_6 L} = 183\,\mathrm{mm}$$

$$\overline{M_6 L} = \frac{183}{2{,}082} = 87{,}9\,\mathrm{mm}$$

amount of mixing point:

$$\dot{M}_6 = \dot{R}_5 + \dot{L} = 1017{,}1 + 1.100 = 2117{,}1\,\mathrm{kg/h}$$

concentrations:

$w_{C,E_6} = 0{,}033 \qquad w_{C,M_6} = 0{,}06 \qquad w_{C,R_6} = 0{,}094$

raffinate flow:

$$\dot{R}_6 = \frac{\dot{M}_6 \cdot \left(w_{C,M_6} - w_{C,E_6}\right)}{w_{C,R_6} - w_{C,E_6}} = \frac{2.117{,}1 \cdot (0{,}06 - 0{,}033)}{0{,}094 - 0{,}033} = 937{,}1\,\mathrm{kg/h}$$

extract flow:

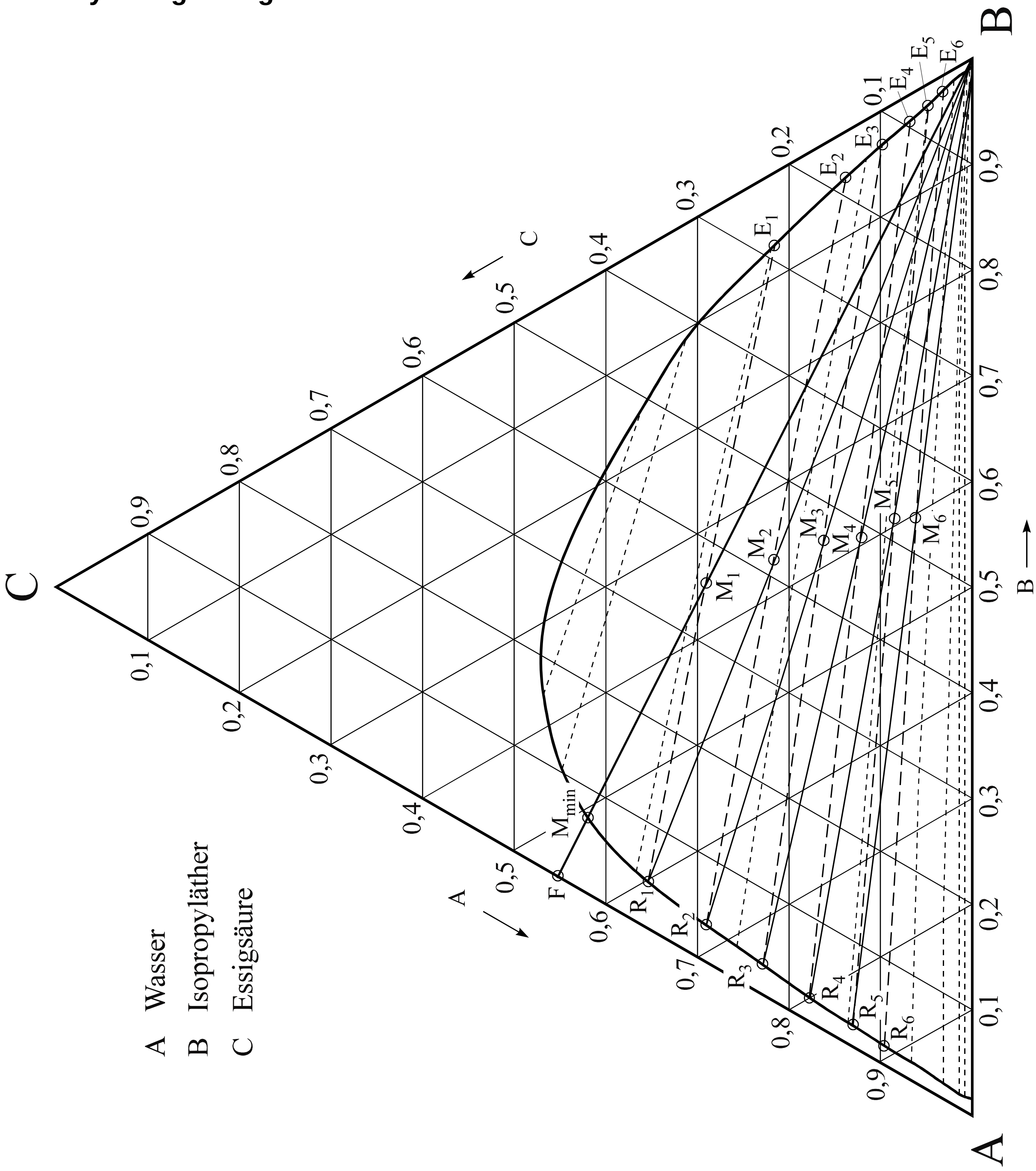
$$\dot{E}_6 = \dot{M}_6 - \dot{R}_6 = 2.117{,}1 - 937{,}1 = 1180\,\mathrm{kg/h}$$

The concentration of the raffinate of this 6. step is lower than the necessary concentration so that the extraction can be stopped.

necessary number of steps: :

$$\boxed{N_{th} = 6}$$

Ternary triangle diagram



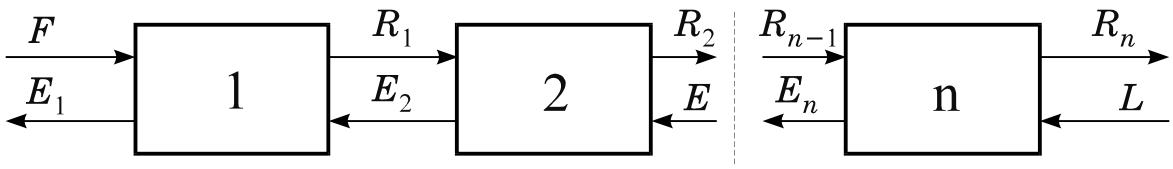
Example 5: Multi Step Countercurrent Extraction

The acetic acid / water mixture of example 4 has to be extracted in a multi step countercurrent extraction cascade with isopropyl ether as solvent. The residual acetic acid concentration is also given with 10 wt%.

You have to determine:

a) the necessary amount of theoretical extraction steps in the triangle diagram for the case that the effective amount of solvent is 2649 kg/h.

Phase equilibria data see example 4.



a) number of theoretical steps in the triangle diagram

The number of theoretical steps can be determined in the triangle diagram by a method developed by Hunter and Nash.

It has to be considered that:

- points of coexisting phases in equilibrium are on a connode
- points of phases, which contact at a cross section of the extractor, have to be on a pole line.

construction:

$w_{C,F} = 0{,}45$ $\quad w_{C,E_1} = 0{,}227$ $\quad w_{C,E_{max}} = 0{,}291$

The mixing point *M* is given by the ratio of feed *F* and solvent *B* and has to be on the connection line $\overline{FB}$. The amount of the mixing point *M* can be determined by a total balance:

$$\dot{M} = \dot{F} + \dot{B} = \dot{R}_n + \dot{E}_1$$

The point E_1 has to be on the binodal curve and on the connection line $\overline{R_n M}$.

Attention: The line $\overline{R_n E_1}$ represents no connode but only a balance line!!

The points F and E_1 are connected by the upper pole line. F represents the feed, which enters the first extraction step and E_1 is the extract which leaves this first step. The lower pole line is given by the connection of the solvent L and the raffinate R_n leaving the extraction plant.

The pole is fixed by crossing the two pole lines $\overline{F E_1}$ and $\overline{B R_n}$.

For the case that the first extraction step is a theoretical step the leaving phases have to be in equilibrium. R_1 as point of the leaving raffinate phase $\dot{R}_1$, has to be on the binodal curve and has to be on a connode through the extract E_1.

The raffinate phase $\dot{R}_1$ and the extract phase $\dot{E}_2$ contact in the next extraction step. The point E_2 of the extract phase $\dot{E}_2$ has to be on the binodal curve and further on the pole line, which goes through R_1.

Doing the construction for all points R_2, R_3 … and E_3, E_4 by this method finally the necessary number of theoretical steps N_{th} for the extraction can be determined.

$$\boxed{N_{th} = 6{,}5}$$

For the calculation of the amounts of E_i and R_i the concentrations are determined from the triangle diagram.

step	1	2	3	4	5	6	7
w_{C,R_i}	0,383	0,335	0,285	0,233	0,171	0,119	0,029
w_{C,E_i}	0,227	0,175	0,134	0,100	0,069	0,036	0,008

balance:

$$\dot{F} \cdot w_{C,F} + \dot{L} \cdot w_{C,L} = \dot{E}_1 \cdot w_{C,E_1} + \dot{R}_n \cdot w_{C,R_n}$$

$$\dot{M}_{\text{ges}} = \dot{E}_1 + \dot{R}_n = \dot{F} + \dot{L} = 2.000 + 2.649 = 4.649 \text{ kg/h}$$

$$\dot{F} \cdot w_{C,F} + \dot{L} \cdot w_{C,L} = \left(\dot{M}_{\text{ges}} - \dot{R}_n\right) \cdot w_{C,E_1} + \dot{R}_n \cdot w_{C,R_n}$$

$$\dot{R}_n = \frac{\dot{F} \cdot w_{C,F} + \dot{L} \cdot w_{C,L} - \dot{M}_{\text{ges}} \cdot w_{C,E_1}}{w_{C,R_n} - w_{C,E_1}}$$

$$= \frac{2.000 \cdot 0{,}45 + 2.649 \cdot 0 - 4.649 \cdot 0{,}227}{0{,}1 - 0{,}227} = 1.223 \text{ kg/h}$$

$$\dot{E}_1 = \dot{M}_{\text{ges}} - \dot{R}_n = 4.649 - 1.223 = 3.426 \text{ kg/h}$$

1. step

$$\dot{F} + \dot{E}_2 = \dot{R}_1 + \dot{E}_1$$

$$\dot{F} \cdot w_{C,F} + \dot{E}_2 \cdot w_{C,E_2} = \dot{R}_1 \cdot w_{C,R_1} + \dot{E}_1 \cdot w_{C,E_1}$$

$$\dot{F} \cdot w_{C,F} + \dot{E}_2 \cdot w_{C,E_2} = \left(\dot{F} + \dot{E}_2 - \dot{E}_1\right) \cdot w_{C,R_1} + \dot{E}_1 \cdot w_{C,E_1}$$

$$\dot{E}_2 = \frac{\dot{F} \cdot \left(w_{C,F} - w_{C,R_1}\right) + \dot{E}_1 \cdot \left(w_{C,R_1} - w_{C,E_1}\right)}{w_{C,R_1} - w_{C,E_2}}$$

$$\dot{E}_2 = \frac{2.000 \cdot (0{,}45 - 0{,}383) + 3.426 \cdot (0{,}383 - 0{,}227)}{0{,}383 - 0{,}175} = 3.214 \text{ kg/h}$$

$$\dot{R}_1 = 2.000 + 3.214 - 3.426 = 1.788 \text{ kg/h}$$

The following steps are calculated analogous

2. step

$$\dot{E}_3 = \frac{\dot{R}_1 \cdot \left(w_{C,R_1} - w_{C,R_2}\right) + \dot{E}_2 \cdot \left(w_{C,R_2} - w_{C,E_2}\right)}{w_{C,R_2} - w_{C,E_3}}$$

$$\dot{E}_3 = \frac{1.788 \cdot (0{,}383 - 0{,}335) + 3.376 \cdot (0{,}335 - 0{,}175)}{0{,}335 - 0{,}134} = 3.114 \text{ kg/h}$$

$$\dot{R}_2 = 1.788 + 3.114 - 3.214 = 1.688 \text{ kg/h}$$

3. step

$$\dot{E}_4 = \frac{\dot{R}_2 \cdot \left(w_{C,R_2} - w_{C,R_3}\right) + \dot{E}_3 \cdot \left(w_{C,R_3} - w_{C,E_3}\right)}{w_{C,R_3} - w_{C,E_4}}$$

$$\dot{E}_4 = \frac{1.688 \cdot (0{,}335 - 0{,}285) + 3.114 \cdot (0{,}285 - 0{,}134)}{0{,}285 - 0{,}100} = 2998\,\mathrm{kg/h}$$

$$\dot{R}_3 = 1.688 + 2998 - 3.114 = 1.572\,\mathrm{kg/h}$$

4. step

$$\dot{E}_5 = \frac{\dot{R}_3 \cdot \left(w_{C,R_3} - w_{C,R_4}\right) + \dot{E}_4 \cdot \left(w_{C,R_4} - w_{C,E_4}\right)}{w_{C,R_4} - w_{C,E_5}}$$

$$\dot{E}_5 = \frac{1.572 \cdot (0{,}285 - 0{,}233) + 2998 \cdot (0{,}233 - 0{,}100)}{0{,}233 - 0{,}069} = 2.930\,\mathrm{kg/h}$$

$$\dot{R}_4 = 1.572 + 2.930 - 2998 = 1.504\,\mathrm{kg/h}$$

5. step

$$\dot{E}_6 = \frac{\dot{R}_4 \cdot \left(w_{C,R_4} - w_{C,R_5}\right) + \dot{E}_5 \cdot \left(w_{C,R_5} - w_{C,E_5}\right)}{w_{C,R_5} - w_{C,E_6}}$$

$$\dot{E}_6 = \frac{1.504 \cdot (0{,}233 - 0{,}171) + 2.930 \cdot (0{,}171 - 0{,}069)}{0{,}171 - 0{,}036} = 2.905\,\mathrm{kg/h}$$

$$\dot{R}_5 = 1.504 + 2.905 - 2.930 = 1.479\,\mathrm{kg/h}$$

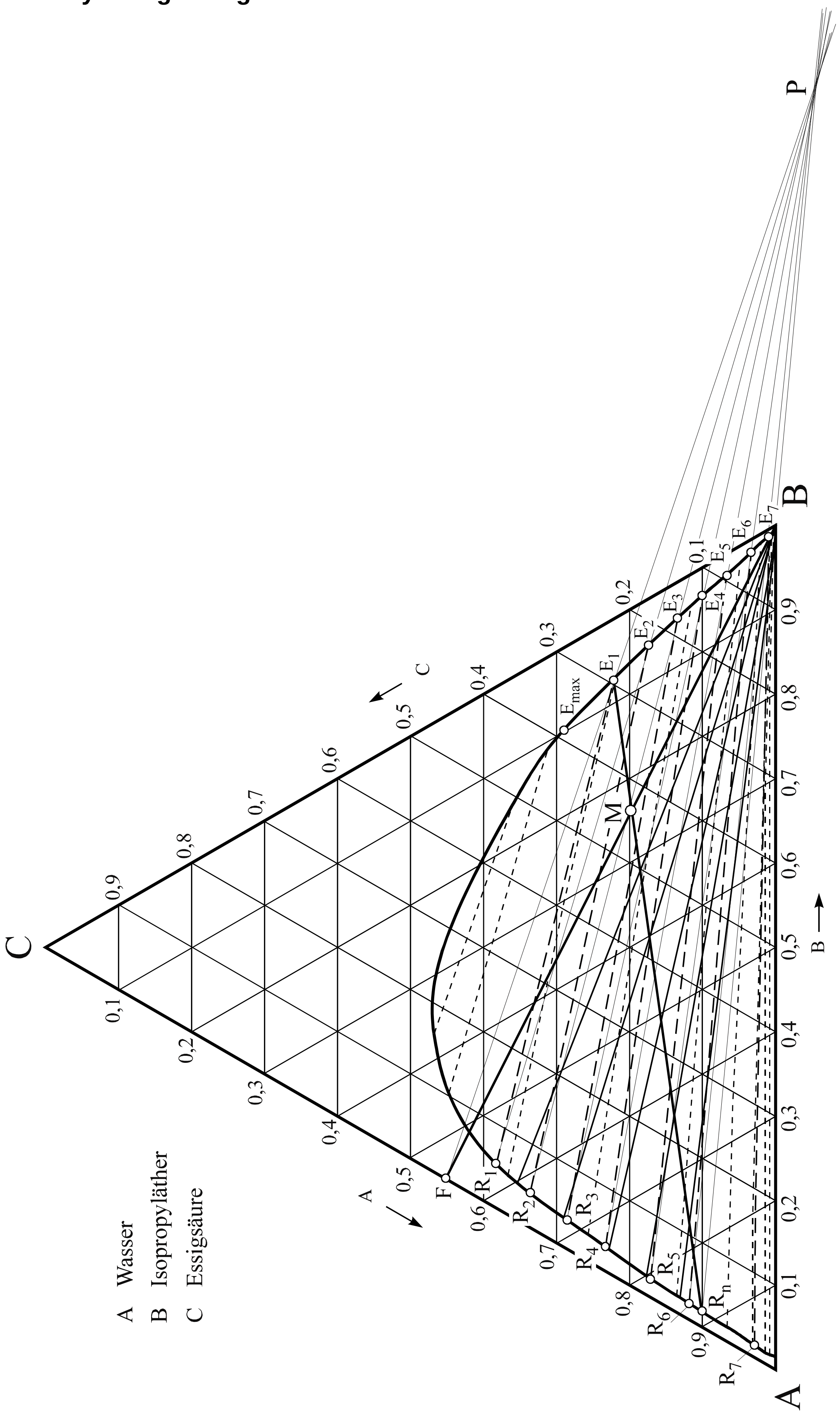
6. step

$$\dot{E}_7 = \frac{\dot{R}_5 \cdot \left(w_{C,R_5} - w_{C,R_6}\right) + \dot{E}_6 \cdot \left(w_{C,R_6} - w_{C,E_6}\right)}{w_{C,R_6} - w_{C,E_7}}$$

$$\dot{E}_7 = \frac{1.479 \cdot (0{,}171 - 0{,}119) + 2.905 \cdot (0{,}119 - 0{,}036)}{0{,}119 - 0{,}008} = 2.865\,\mathrm{kg/h}$$

$$\dot{R}_6 = 1.479 + 2.865 - 2.905 = 1.439\,\mathrm{kg/h}$$

Ternary triangle diagram



Appendix: Triangle Diagrams