

## Extraction with Reflux for Type II Systems

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There is a type of systems with a different solubility chart from the Type I systems studied in the previous lecture notes. They are called Type II systems and their solubility charts have the following form:

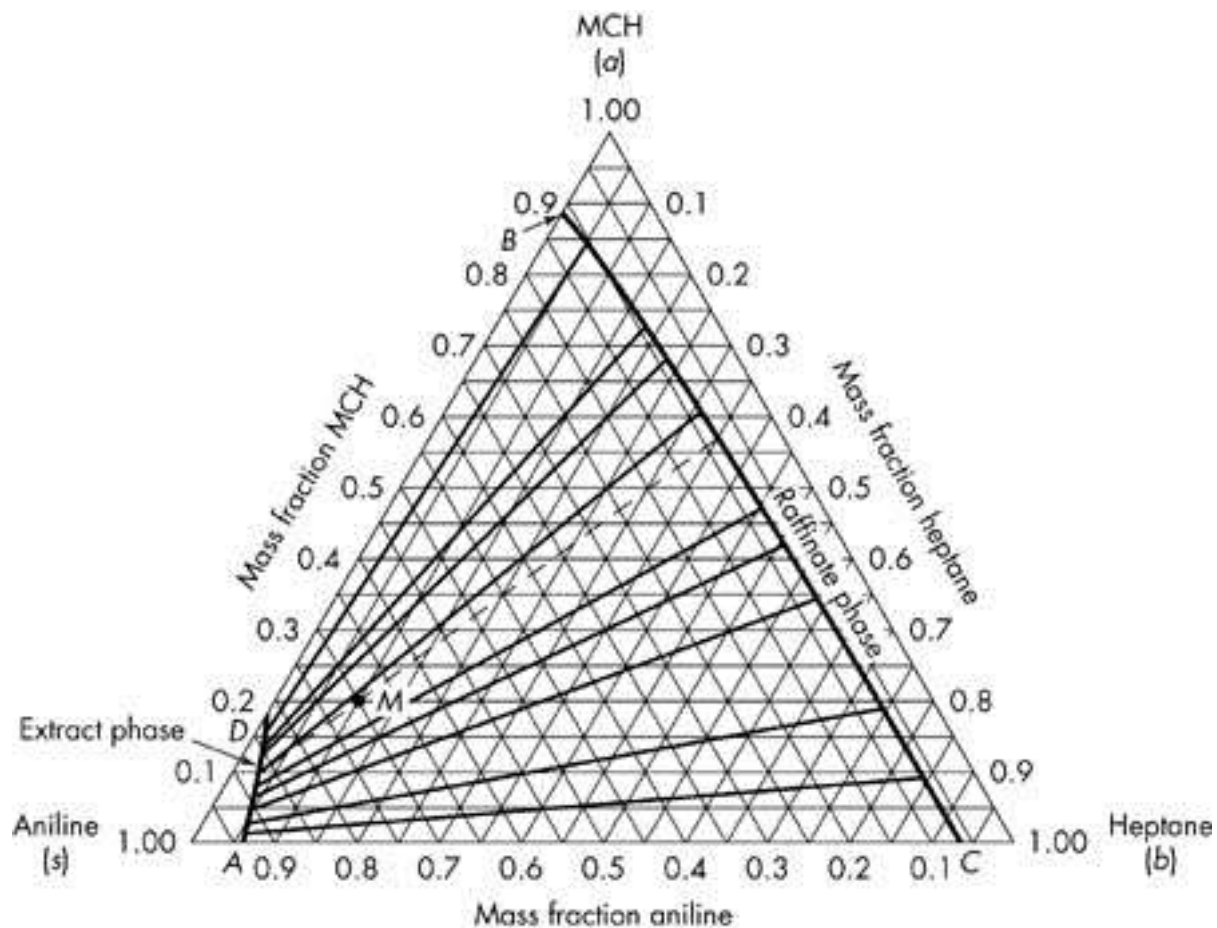


Figure 1. Solubility chart for a Type II system

In this system the solute is MCH (methyl-cyclo-hexane), the diluent is n-heptane, and the solvent is aniline.

The main difference from Type II systems is that there is no *plait point*, that is, the two-phase region extends from the AS axis to the SB axis. Notice that there is a tie line coinciding with the AS axis with points  $x_B = 0$ ,  $y_B = 0$ , and one coinciding with the SB axis with points  $x_A = 0$ ,  $y_A = 0$ . Notice also that the solvent and the solute are only partially miscible with each other. This should surprise you as the solvent is used to extract the solute even it is not even completely soluble in it.

As we shall see, it is possible to produce very pure extracts (on a solvent-free basis) by arranging two extraction batteries and returning some of the extract as reflux. The idea is borrowed from distillation and will show you that one of your jobs as a chemical engineer is to transfer ideas from one application to another.

## 1 Use Solvent-free Basis for the Calculations

The calculations for Type I systems are greatly simplified if they are carried out on a solvent-free basis. The following formulas convert mass fractions into solvent-free fractions:

$$X = \frac{x_A}{x_A + x_B} \quad Y = \frac{y_A}{y_A + y_B} \quad (1)$$

On a solvent-free basis it is not necessary to differentiate between components A and B because only one composition is required to represent a stream. The fractions of diluent are  $(1 - X)$  and  $(1 - Y)$ , and the solvent is expressed as mass of solvent per mass of solvent-free solution, calculated by

$$X_S = \frac{1 - x_A - x_B}{x_A + x_B} \quad \text{and} \quad Y_S = \frac{1 - y_A - y_B}{y_A + y_B} \quad (2)$$

The equilibrium data of Figure 1, taken from the MCH mass fraction at the end of the tie lines, can be plotted on solvent-free basis as follows:

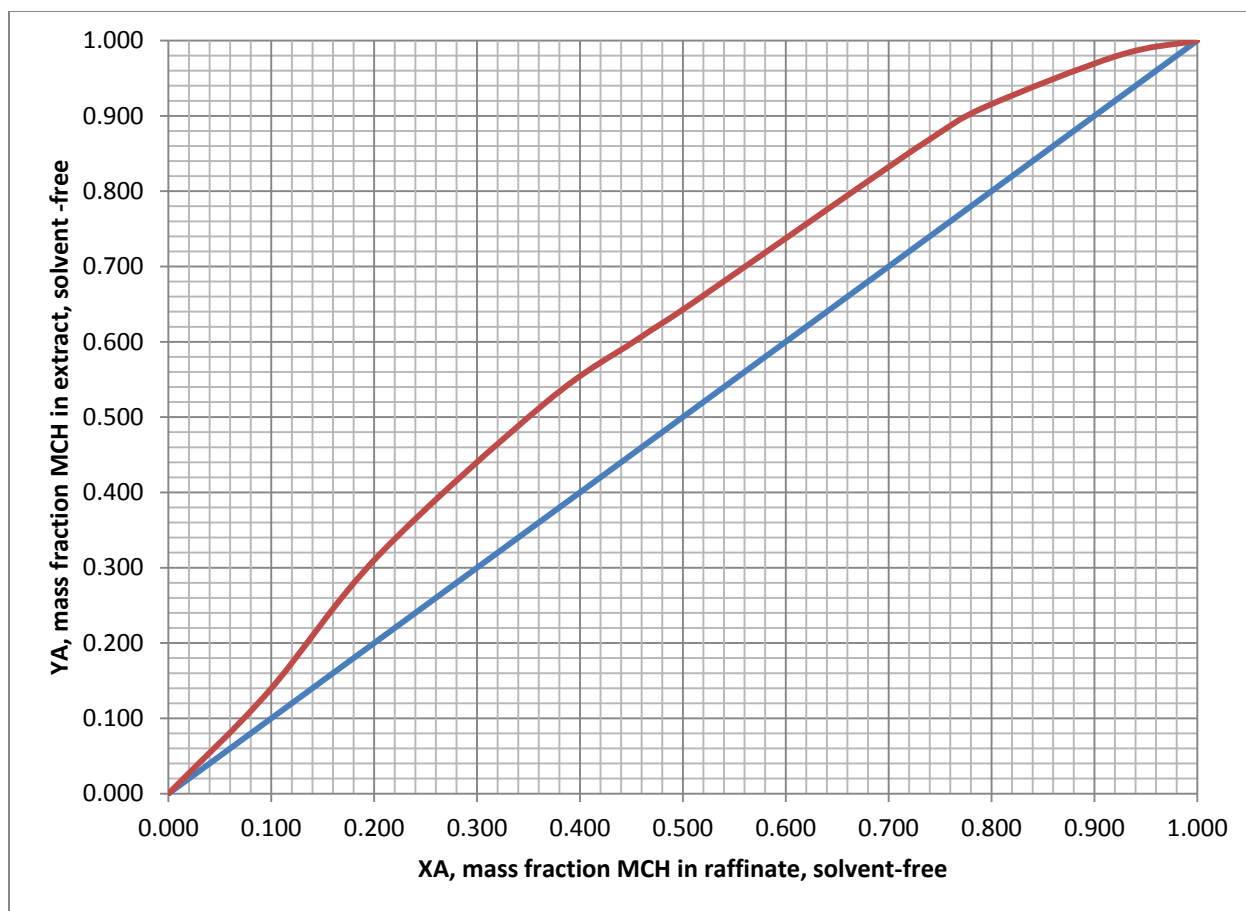


Figure 2. Equilibrium data for MCH-heptane-aniline on solvent-free basis

Similarly, the solubility of aniline in the raffinate from Figure 1 can be plotted on solvent-free basis:

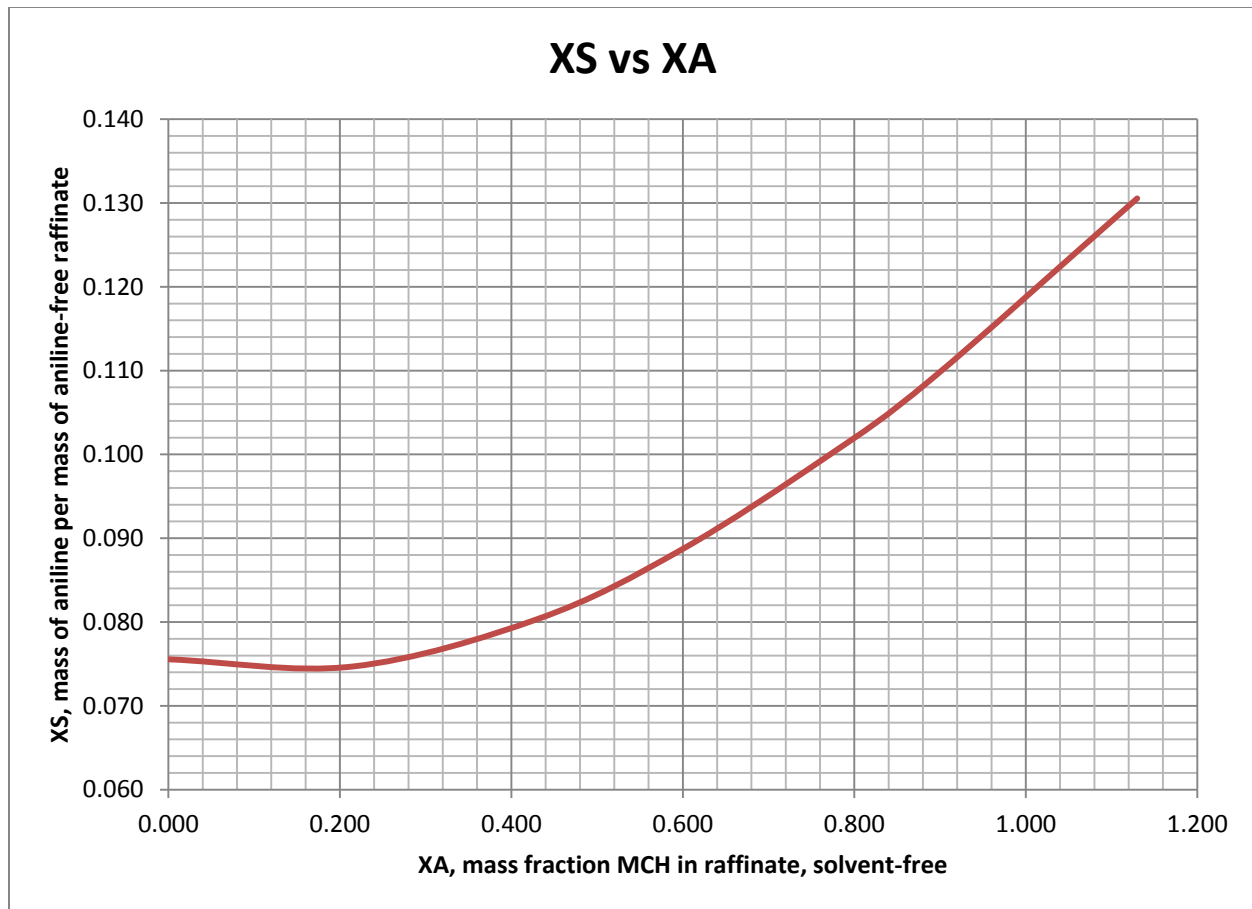


Figure 3. Solubility of aniline in the raffinate on solvent-free basis

The mass of solvent carried by unit mass of solvent-free extract can also be determined from Figure 1:

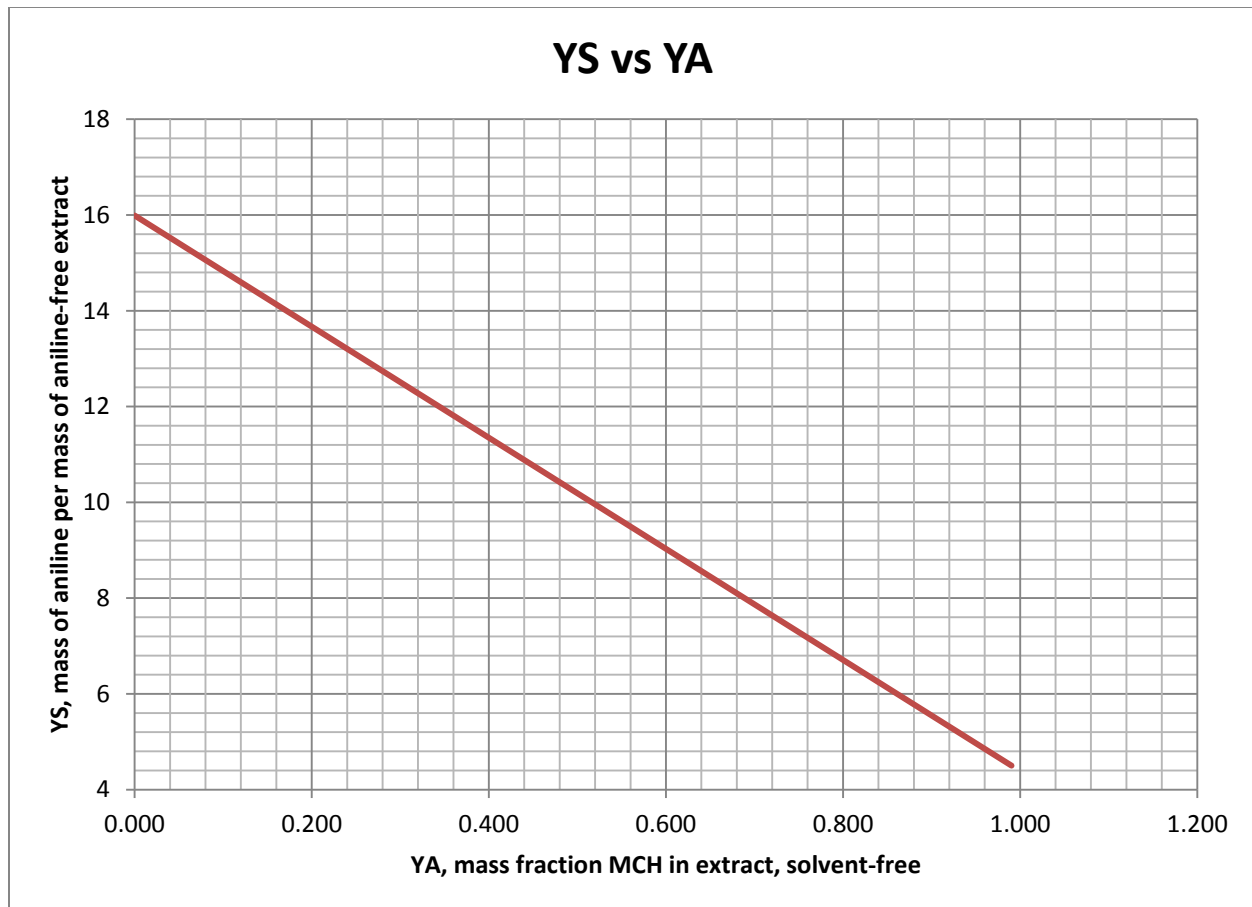


Figure 4. Mass of solvent carried by unit mass of solvent-free extract

The similarity of the equilibrium data of Figure 2 with the equilibrium curves for binary distillation suggests that extraction of Type II systems can be designed like a distillation column to produce a near pure extract on a solvent-free basis. We will look at this idea next.

## 2 Extraction of Type II Systems with Reflux

Figure 5 shows the schematic for extraction of Type II systems with reflux:

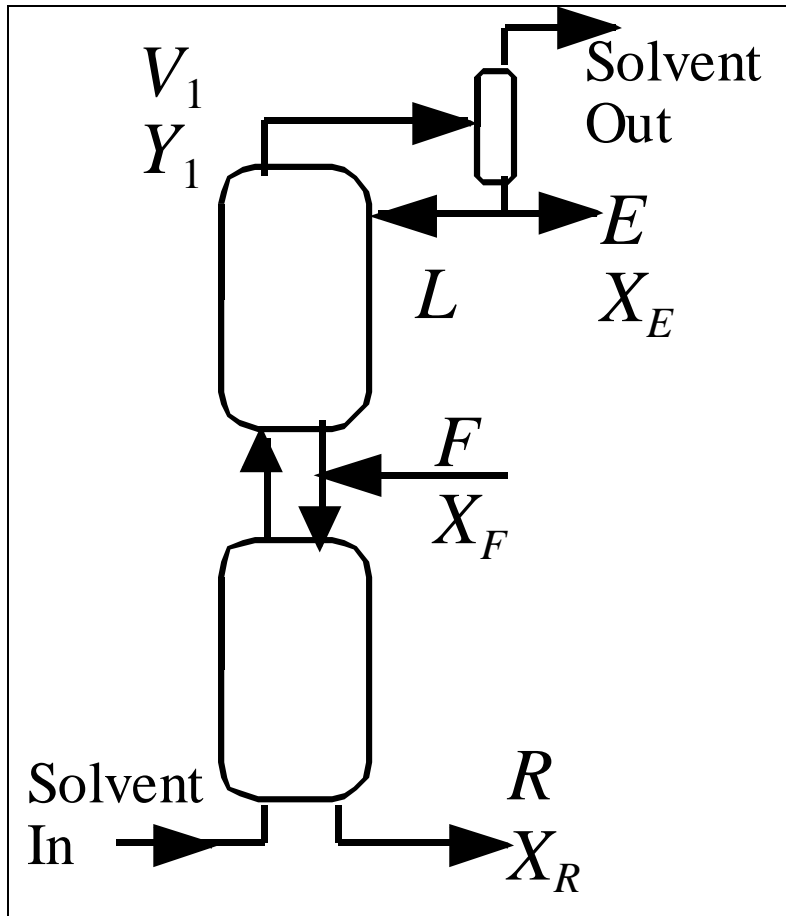


Figure 5. Extraction of a Type II system with reflux

The bottom box in Figure 5 is a countercurrent contact column with the feed entering at the top of this box; the solvent enters at the bottom and the raffinate  $R$  leaves at the bottom. The other stream leaving this bottom box is the extract; this is fed to the top box which is another countercurrent contact column. The extract  $V_1$  leaving the top column is fed to a distillation column where the solvent is separated from it producing a top product  $E$  which is the extract actually free of solvent. Part of this extract is the reflux  $L$  fed to the top column to remove the diluent from the stream flowing up. The solvent removed by the distillation column at the top is recycled back and makes up the majority of the solvent entering at the bottom. Only the solvent dissolved in the raffinate  $R$  is

lost and must be replaced with fresh solvent. The complete operation is simply a single column with the portion above the feed is called the *extract-enriching section* and the portion below the feed called the *raffinate-stripping section*.

You are encouraged to study Figure 5 and recognize the following parallels between stripping with reflux and distillation:

Stripping with Reflux	Distillation
Extract phase flow	Vapor flow
Raffinate phase flow	Liquid flow
Solvent removal column	Condenser
Extract-enriching section	Rectifying section
Raffinate-stripping section	Stripping section
Solvent added at bottom	Heat to reboiler
Solvent-free feed	Saturated-liquid feed

### 3 Over-all Mass Balances

From Figure 4 we can write the following over-all mass balances on a solvent-free basis:

$$\text{Total mass balance:} \quad F = E + R \quad (3)$$

$$\text{Balance on the condenser:} \quad V_1 = E + L \quad (5)$$

### 4 Operating Lines

The enriching operating line is obtained from a solute balance around  $n$  stages in the extract-enriching section:



$$Y_{n+1} = \frac{L_n}{V_{n+1}} X_n + \frac{EX_E}{V_{n+1}} \quad (6)$$

Where  $L_n = V_{n+1} - E$ , and  $V_{n+1}$  is determined from the solvent balance:

$$V_{n+1} = \frac{L_n X_{S,n} - EX_{SE}}{Y_{S,n+1}} \quad (7)$$

Similarly, the stripping operating line results from a solute balance on  $m$  stages in the raffinate-stripping section:

$$Y_{m+1} = \frac{L_m}{V_{m+1}} X_m - \frac{RX_R}{V_{m+1}} \quad (8)$$

Where  $L_m = V_{m+1} + R$ , and  $V_{m+1}$  is determined from the solvent balance:

$$V_{m+1} = \frac{L_m X_{S,m} - RX_{SR}}{Y_{S,m+1}} \quad (9)$$

Equations (7) and (9) must be solved by iteration. The assumption of equi-mass counter-diffusion results in constant phase flows in each section and straight operating lines. At the feed tray the flows change as follows:

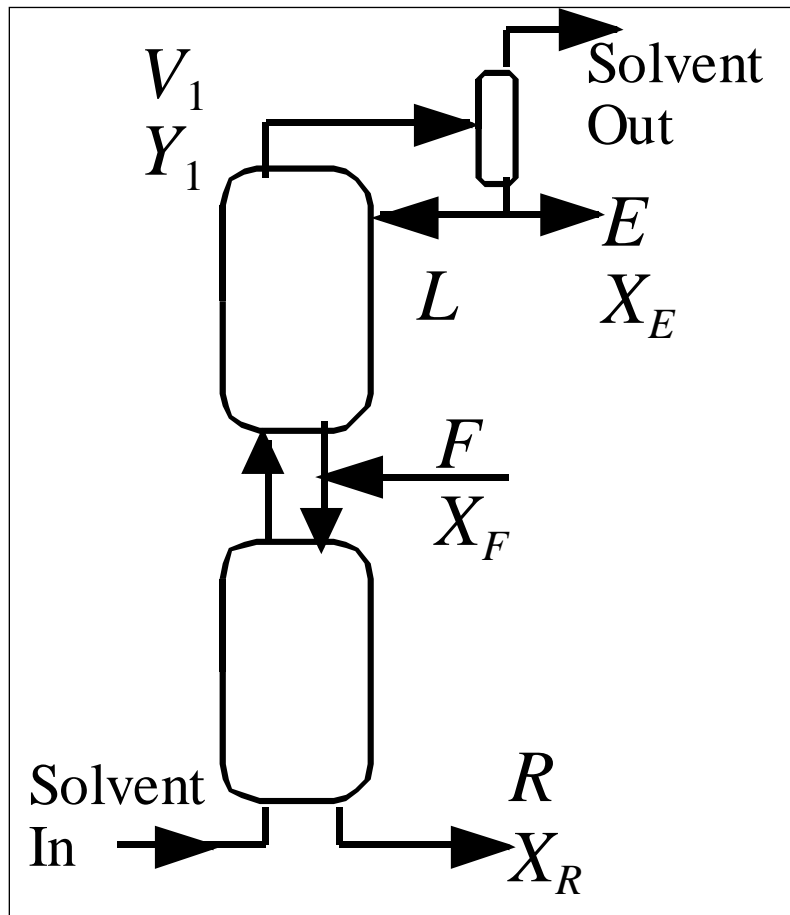
$$L_m = L_n + F \quad (10)$$

Where  $L_m$  and  $L_n$  are the raffinate phase flows in the stripping and enriching sections, respectively, and the extract phase flow does not change at the feed plate.

### Example 1. Design of an Extraction Column of a Type II System with Reflux

A solution of 45 weight% methyl-cyclo-hexane (MCH) and the balance heptane is fed to a column to extract 96% of the MCH and produce an extract product with no more than 3 weight% heptane on a solvent-free basis. The solvent is aniline. An extract-enriching section is used with a reflux to extract product ratio of 3. Determine the number of equilibrium stages required and the location of the feed tray. Report also how much solvent must be fed at the bottom and the make-up solvent required if the solvent recovered from the extract is recycled.

*Solution.*



Basis:  $F = 100 \text{ kg}$

$X_F = 45\%$

96% Recovery of MCH

$X_E = (100 - 3)\% = 97\%$

$L/E = 3$

Recovery of MCH:

$$E = \frac{0.96FX_F}{X_E} = \frac{0.96(100)(0.45)}{0.97} = 44.5 \text{ kg}$$

Total mass balance:

$$R = F - E = 100 - 44.5 = 55.5 \text{ kg}$$

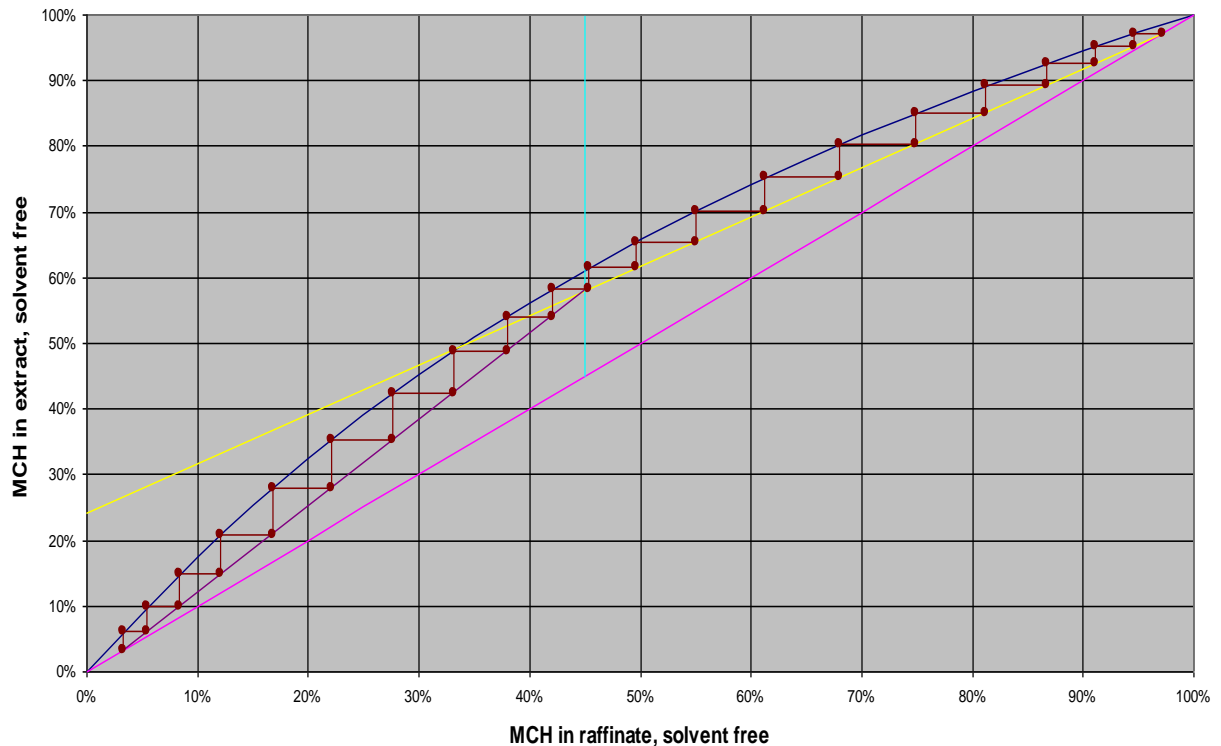
Solute balance: 
$$X_R = \frac{FX_F - EX_E}{R} = \frac{(100)(0.45) - (44.5)(0.97)}{55.5} = 0.0324$$

Reflux flow: 
$$L = 3E = 3(44.5) = 133.5 \text{ kg}$$

Extract flow: 
$$V_1 = L + E = 133.5 + 44.5 = 178.0 \text{ kg}$$

Intercept of enriching operating line on Y-axis:

$$\frac{EX_E}{V_1} = \frac{(44.5)(0.97)}{178.0} = 0.242$$



The McCabe-Thiele solution, assuming constant phase flows in each section (straight operating lines) results in 20 equilibrium stages, 10 in each section. The feed line is always vertical on a solvent-free basis.

From Figure 4, the solvent leaving with the extract at  $Y_1 = X_E = 0.97$  contains  $Y_S = 4.8 \text{ kg aniline/kg aniline-free extract}$ . So, the aniline that must be removed by distillation is:

$$V_1 Y_S = (178.0)(4.8) = 854.4 \text{ kg}$$

From Figure 3, the solvent leaving with the raffinate at  $X_R = 0.0324$  contains  $X_S = 0.076$  kg aniline/kg of aniline-free raffinate. So, the amount of aniline lost with the raffinate is:

$$RX_S = (55.5)(0.076) = 4.2 \text{ kg}$$

Total aniline to be fed at the bottom:  $854.4 + 4.2 = 858.6 \text{ kg}$

Of this, 854.4 kg are recycled and 4.2 kg must be made up with fresh solvent. All these values are on the basis of 100 kg of feed.

## Summary

These notes have shown how an extraction column with recycle can be designed to produce a purer extract than with a simple extraction column. The design is very similar to the design of a binary distillation column when the calculations are carried out on a solvent-free basis. Extraction with reflux can only be carried out with Type II systems. This is because Type I systems have a plait point in their solubility chart. The plait point is similar to an azeotrope in distillation making it impossible to produce a pure extract by extraction.

## Review Questions

1. What is the main difference between Type I and Type II systems?
2. On what basis are the calculations for Type II systems easier to carry out?
3. Why is the design of extraction with reflux for Type II systems similar to the design of a binary distillation column?
4. In comparing extraction with reflux to distillation, what corresponds to the vapor rate in a column? What corresponds to the condenser? What corresponds to the liquid flow? What corresponds to the reboiler?
5. What section of the extraction with reflux column similar to the rectifying section of a distillation column?