

Distillation 101

Dan Summers / March 8, 2011



What's is Distillation?

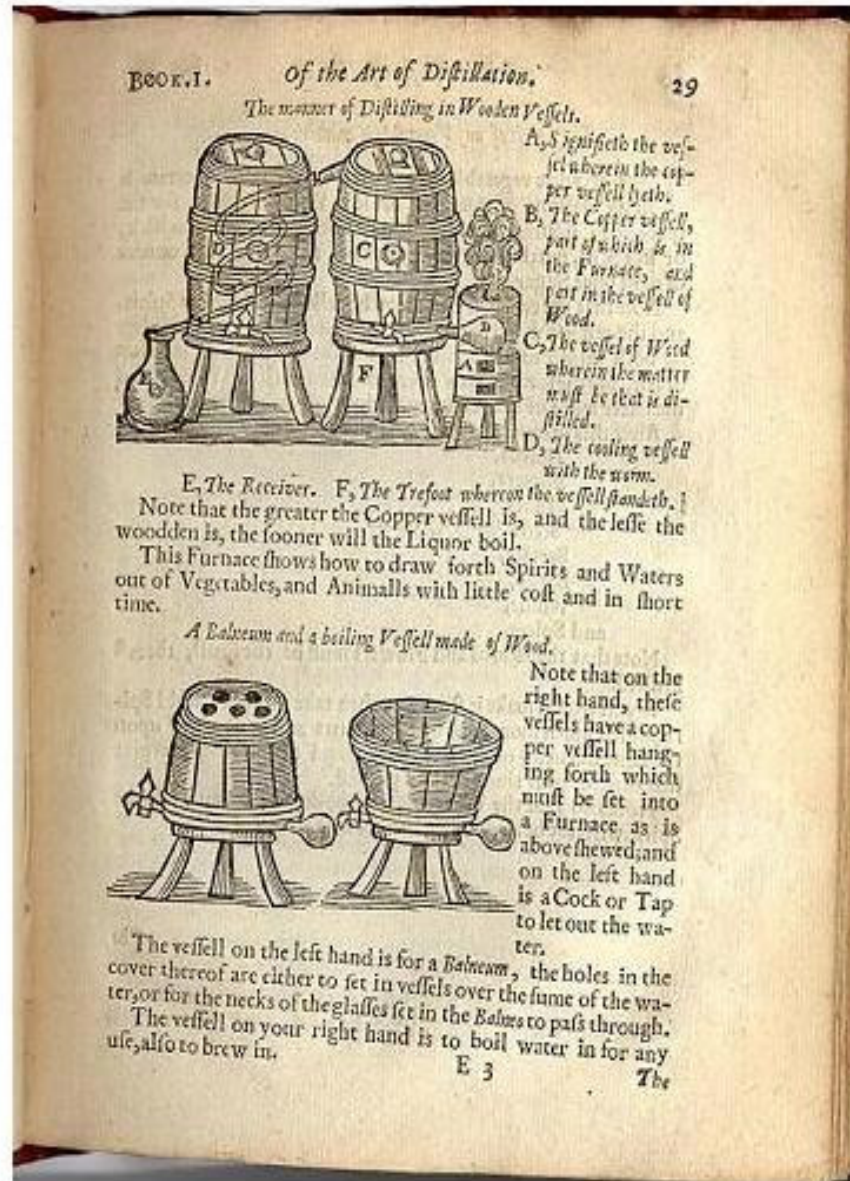
Distillation is purification of gases or liquids by playing with their boiling points.

The earliest known distillation was between ethanol and water

What's is Distillation?



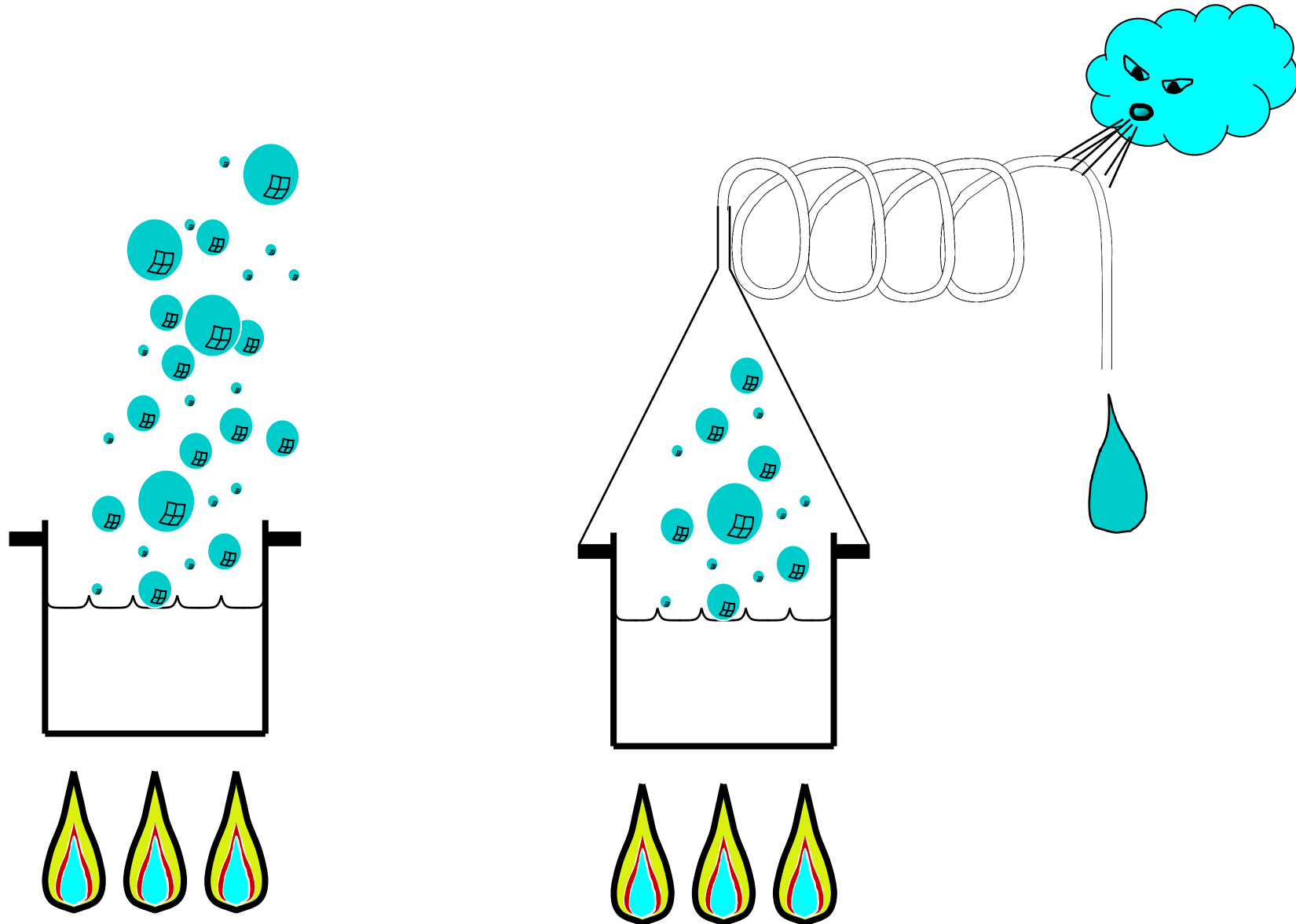
What's is Distillation?



What's is Distillation?

Early Distillation was typically a single stage – or more simply a pot boiling with a condenser.

What's is Distillation?

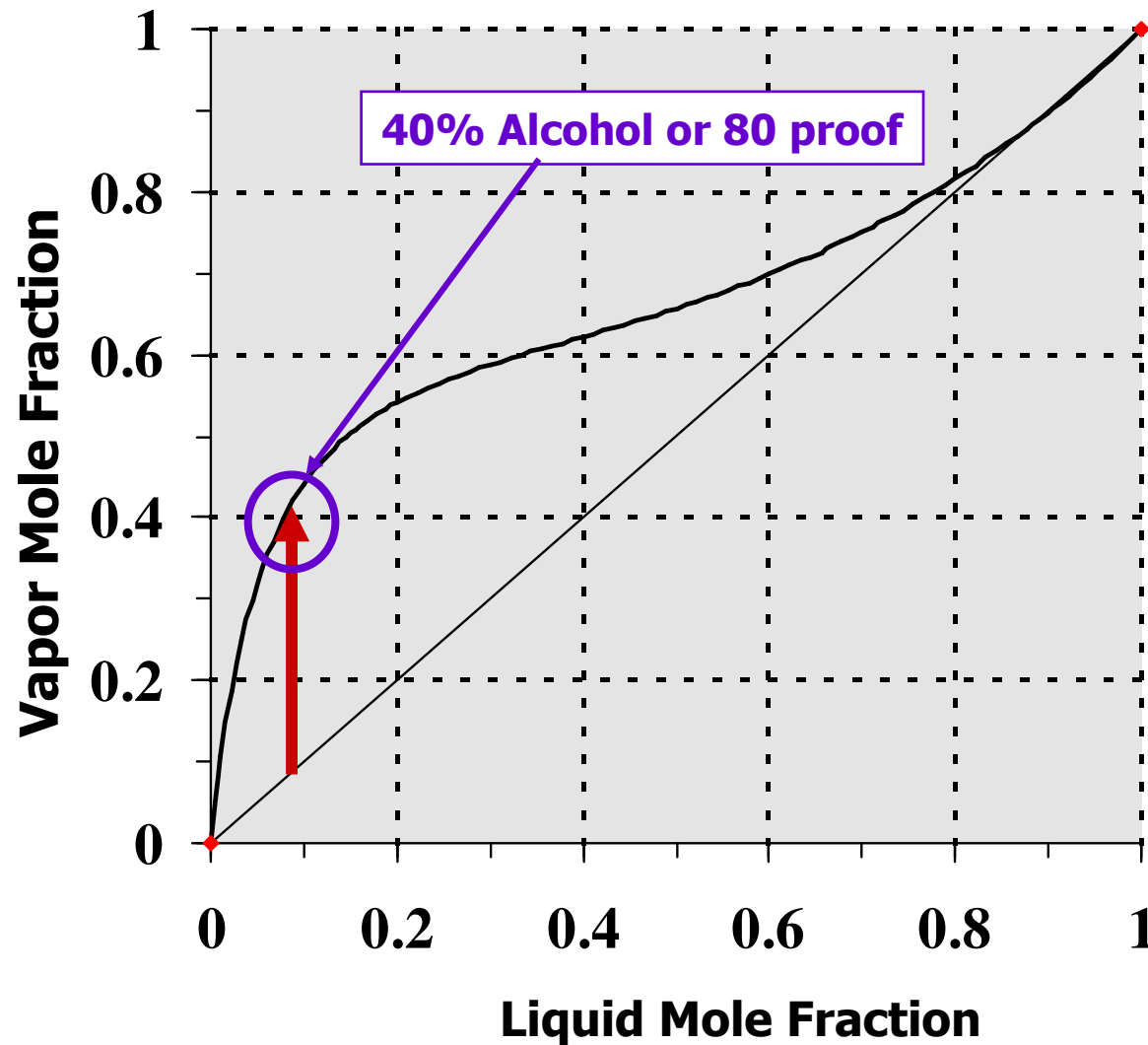


What's is Distillation?

The composition of the overhead condensed vapors was found to have a much higher concentration of the lighter component.

For example, a boiling pot of 6 to 8% ethanol has about 40% ethanol (80 proof) in the condensed vapors.

Ethanol and Water



Pressure = 1 atm

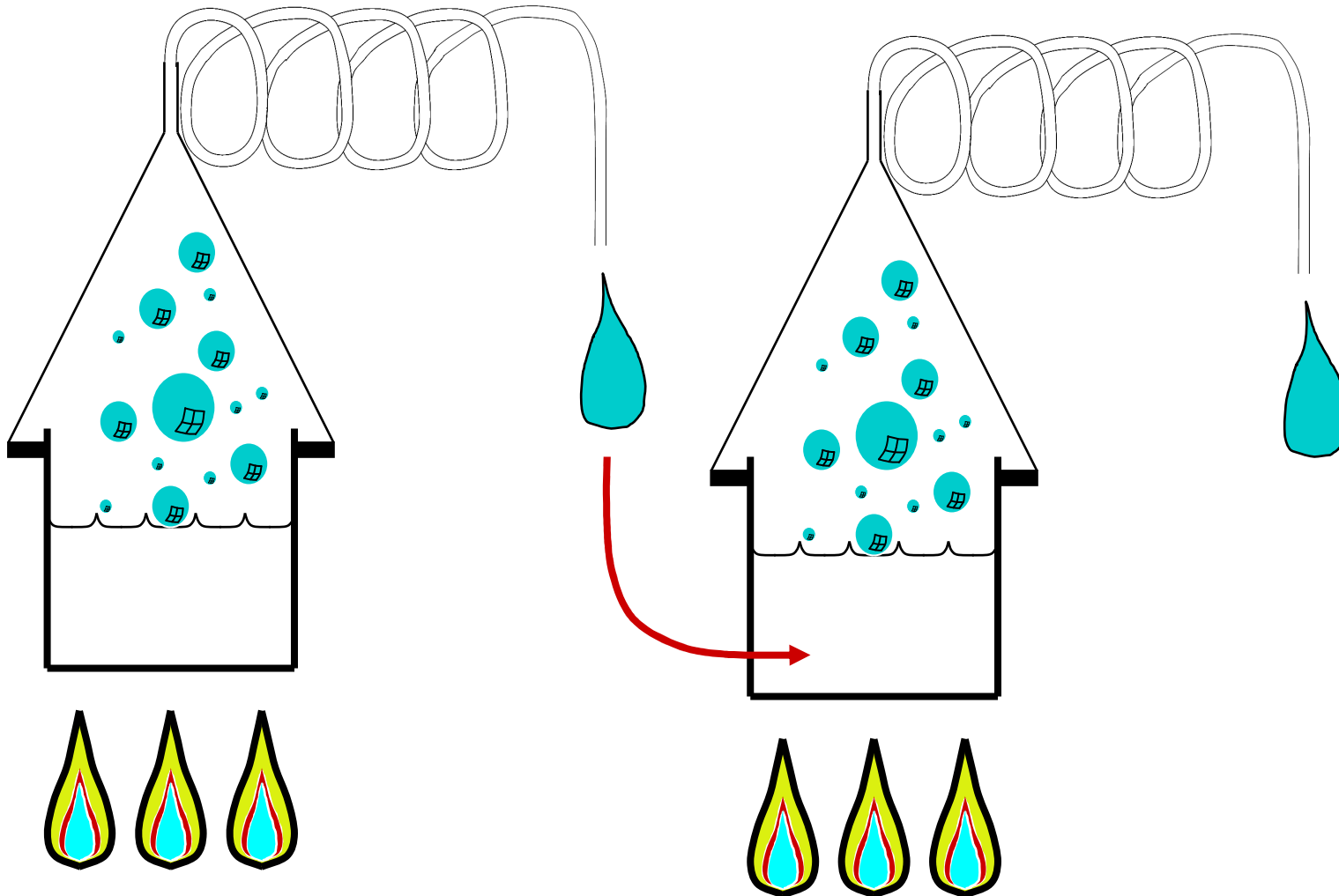


Example of a Home Distillation unit on a stove. Note cooling water hookups.

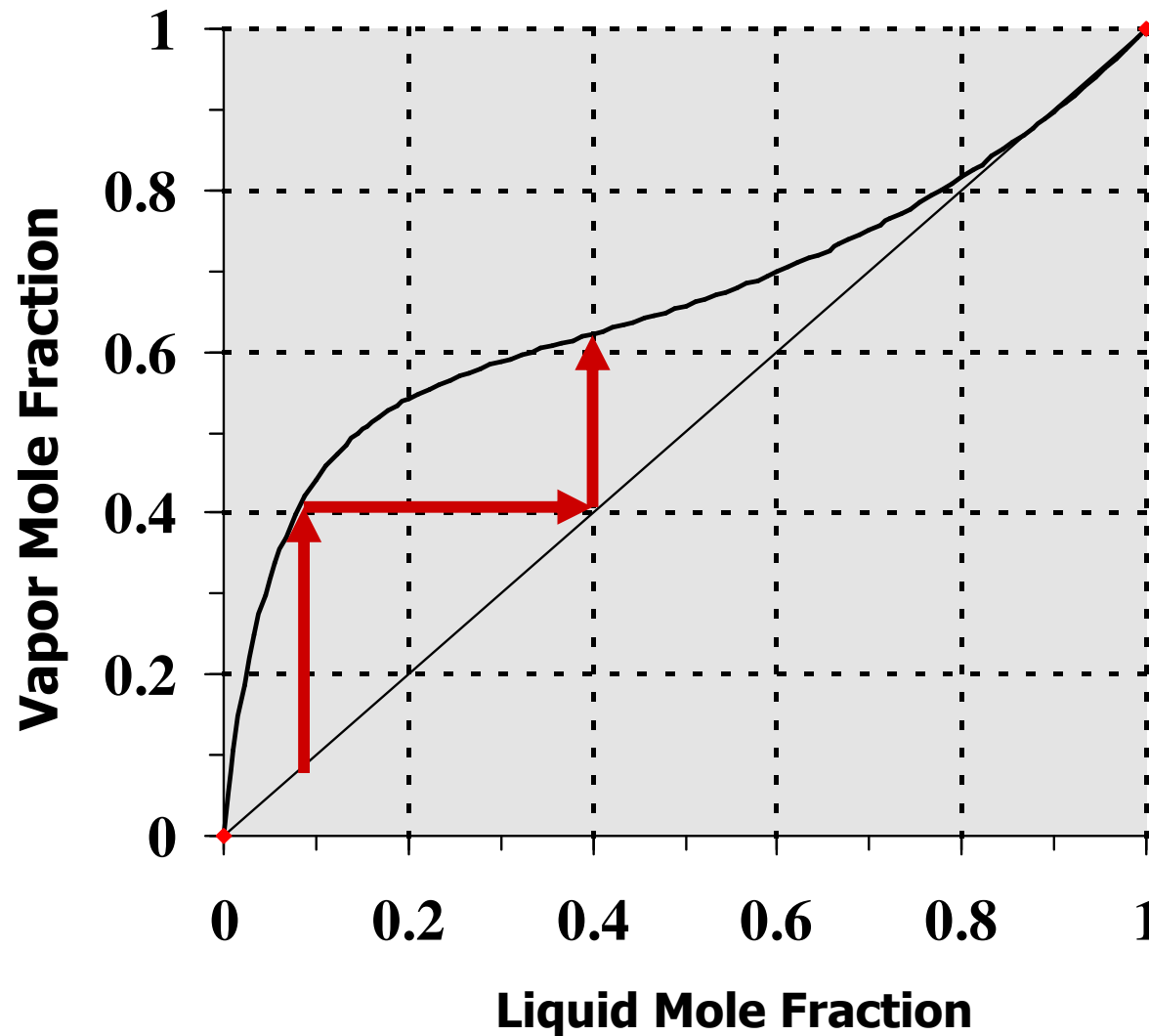
Internals are copper scrubbing pads.

Note – Not legal in the US!

What if you do this twice?



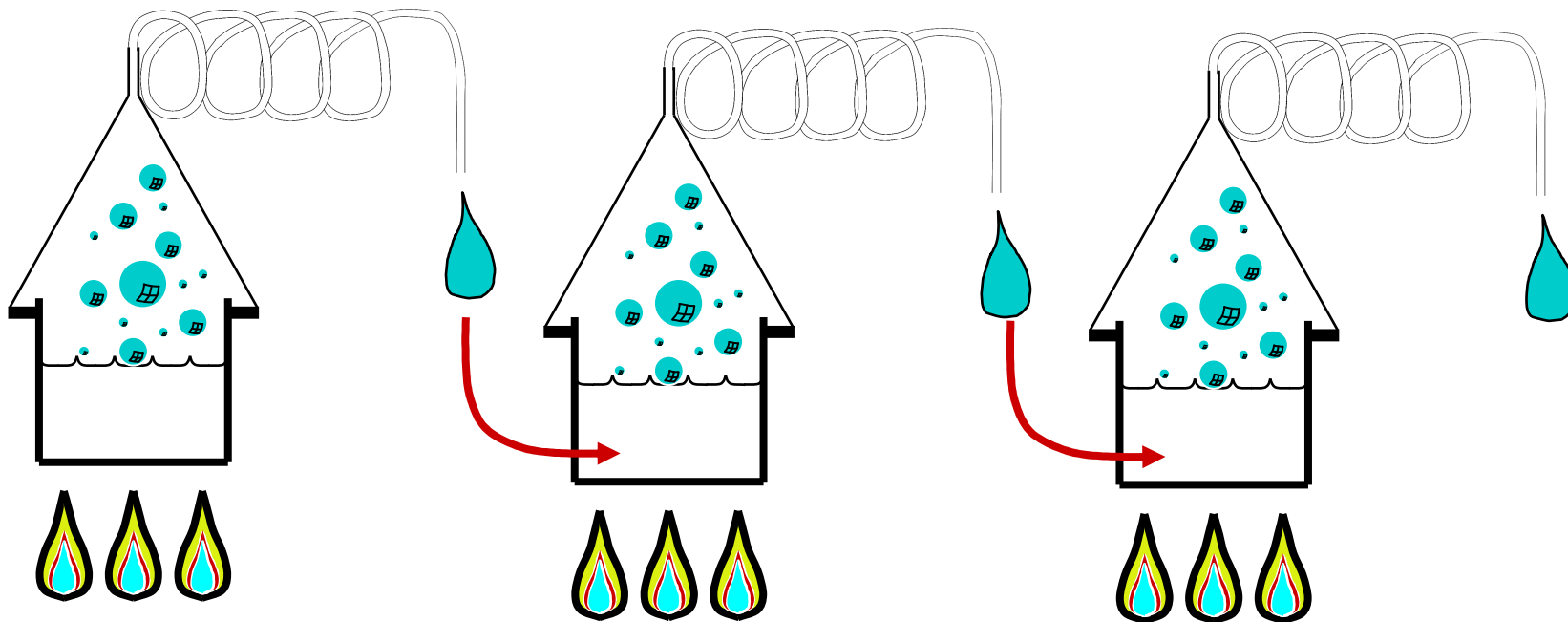
62% Ethanol!



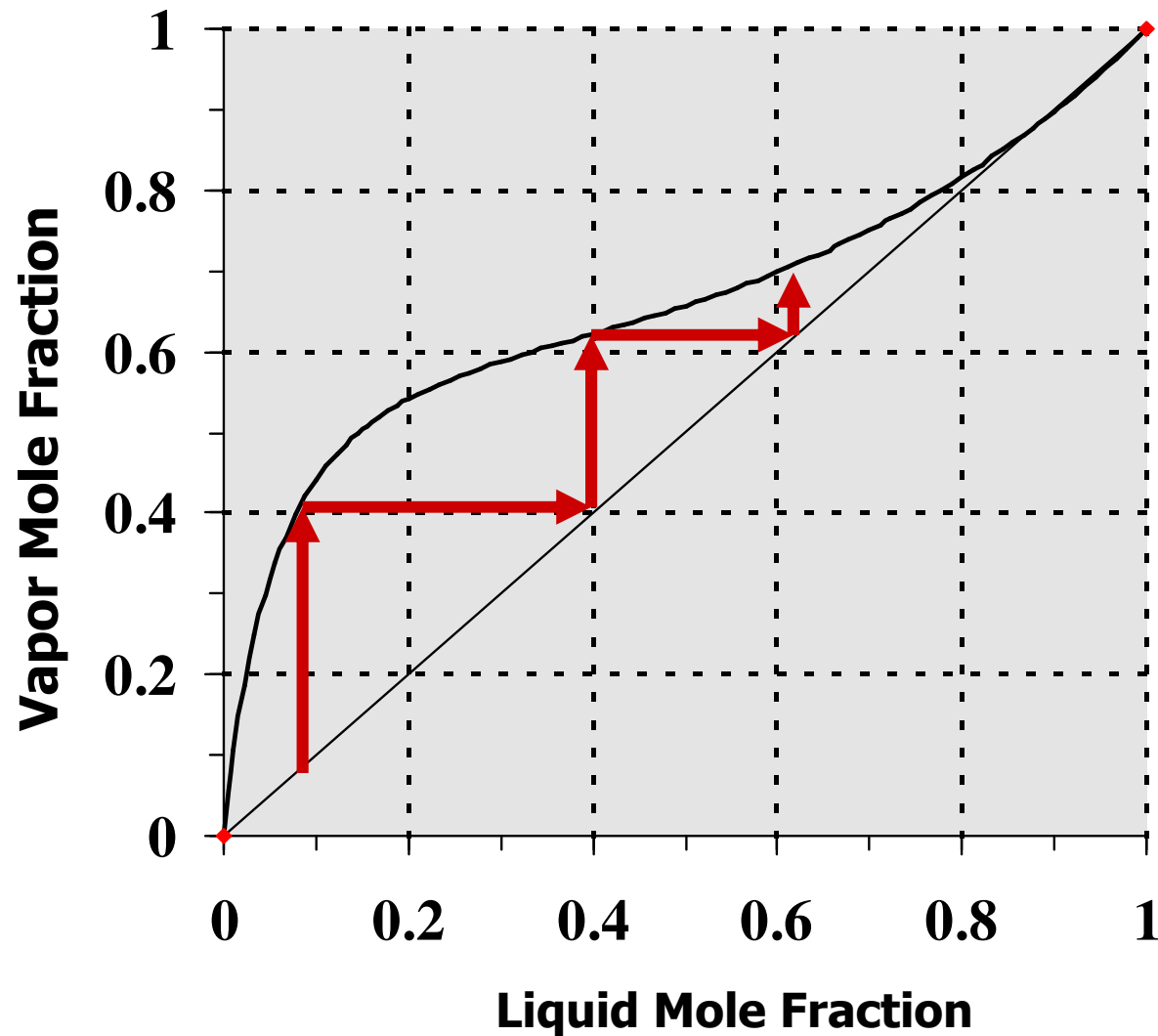
To make cognac
you distill twice

Pressure = 1 atm

How about 3 times?



You get 70% Ethanol!



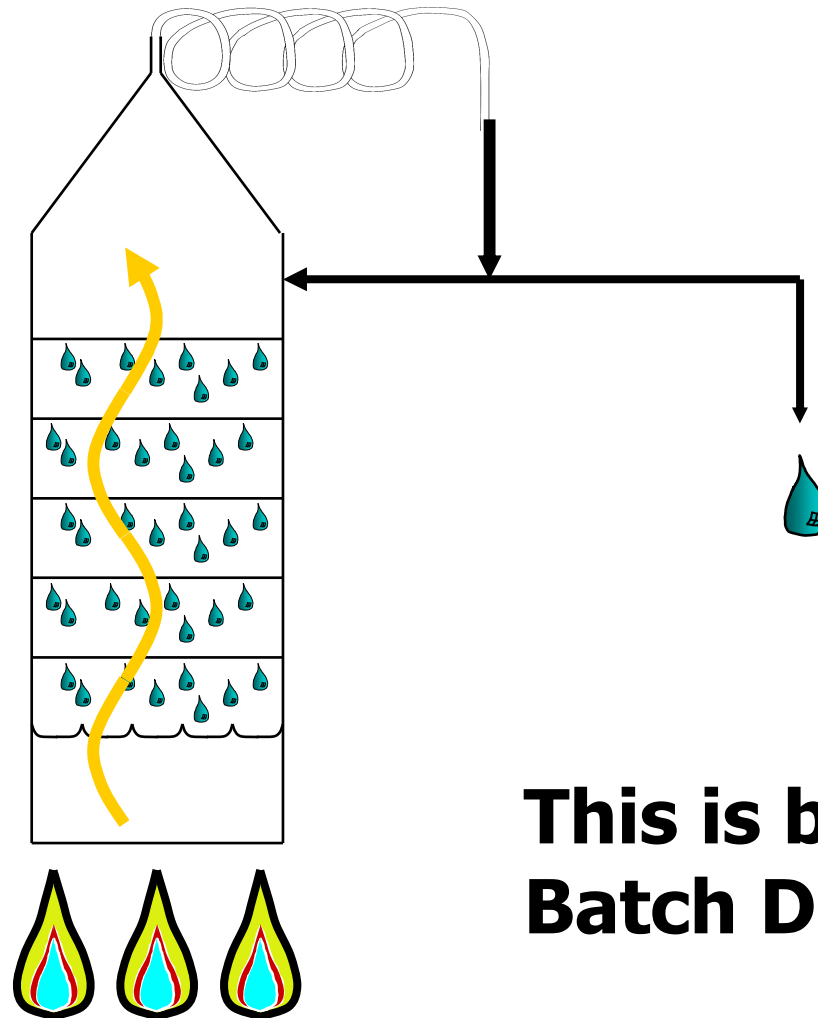
Pressure = 1 atm

What's is Distillation?

It was soon discovered that by placing a series of discrete trays in a pipe, the heat from one tray to the next could duplicate several boiling pots.

In addition if some of the condensate was added back to this pipe, the purities were vastly improved.

What's is Distillation?

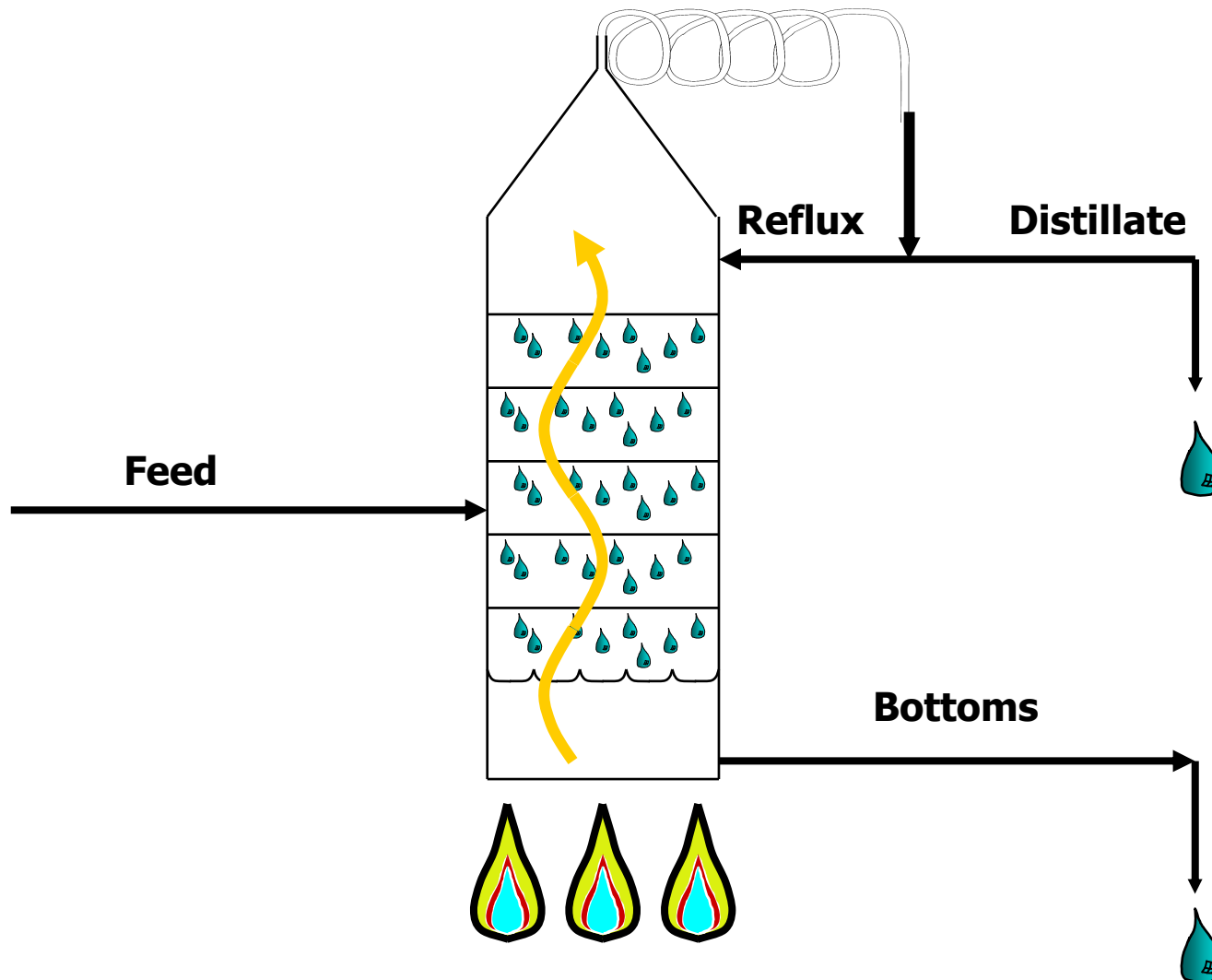


**This is basically
Batch Distillation?**

What's is Batch Distillation?

A tank or vat of liquid, with a distillation tower on top, is heated to boiling. As the liquid boils, the compounds with the lowest vapor pressure (lowest boiling temperature) will leave the vat first and concentrate at the top of the tower. After these first compounds exit the tower, the next "heavier" compounds concentrate at the top of the tower, etc.

Continuous Distillation



What's is Continuous Distillation?

This is a steady state operation where components have the opportunity to establish a concentration profile in the tower such that the "lightest" compound will concentrate in the top of the tower and the "heaviest" compound will concentrate in the bottom of the tower.

What's is Continuous Distillation?

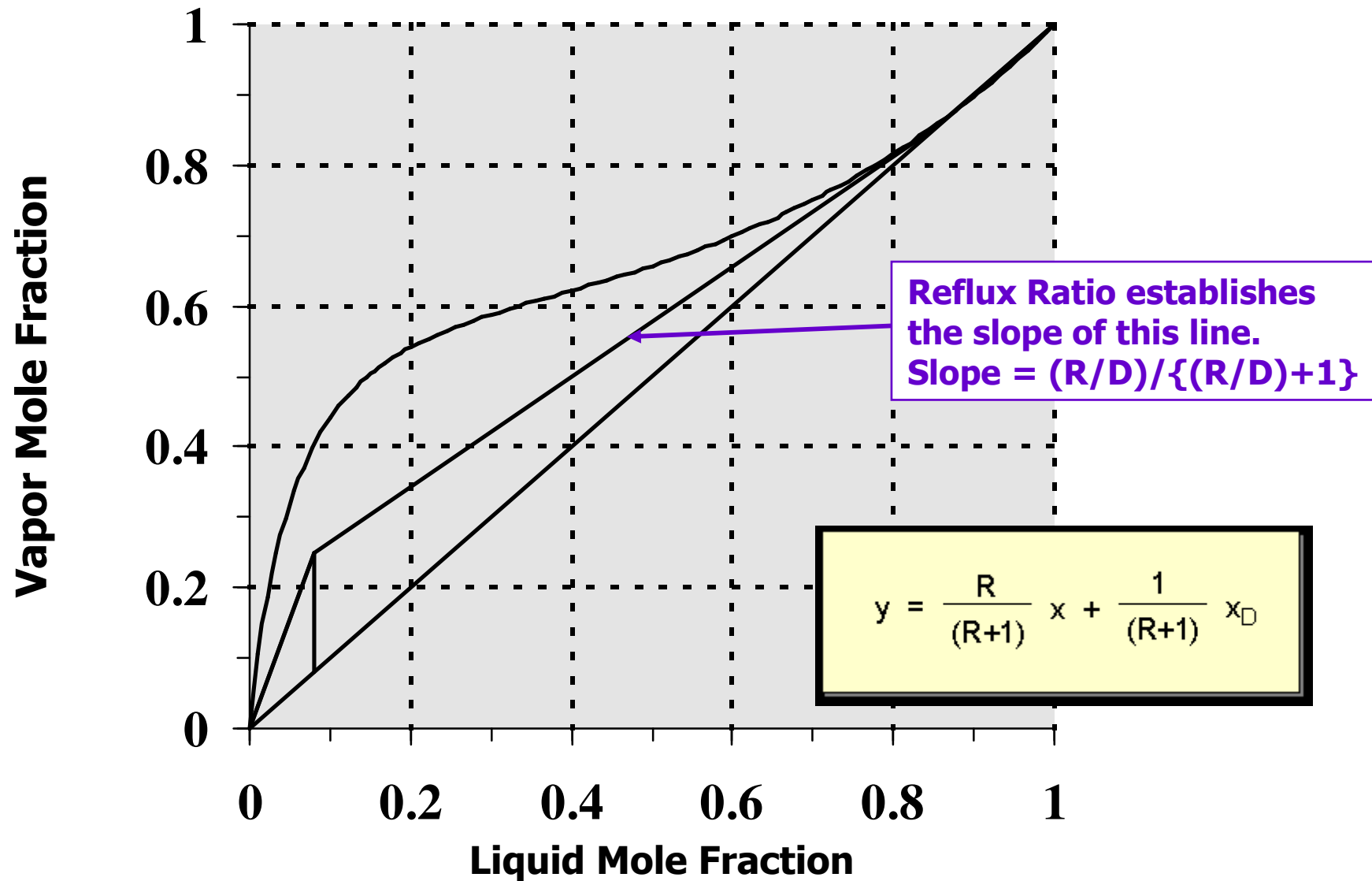
There is always products being drawn out of the tower and there is a relationship between the reflux back to the tower and the product rate. This is called reflux ratio.

What's is Continuous Distillation?

The reflux ratio establishes the internal loads and heat duty on the tower. It also establishes the number of theoretical trays to make a particular separation.

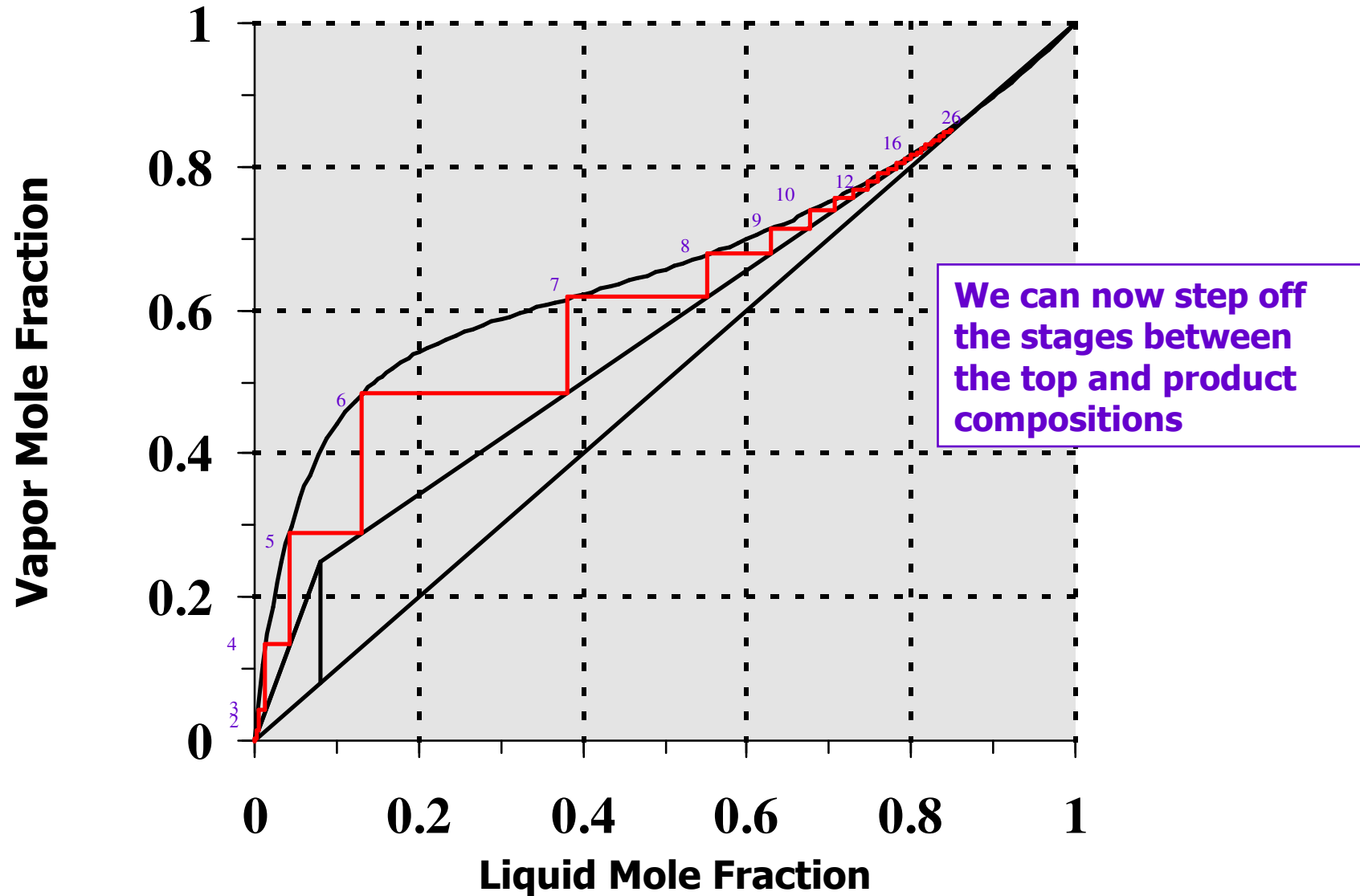
Operating Conditions

Pressure = 1 atm



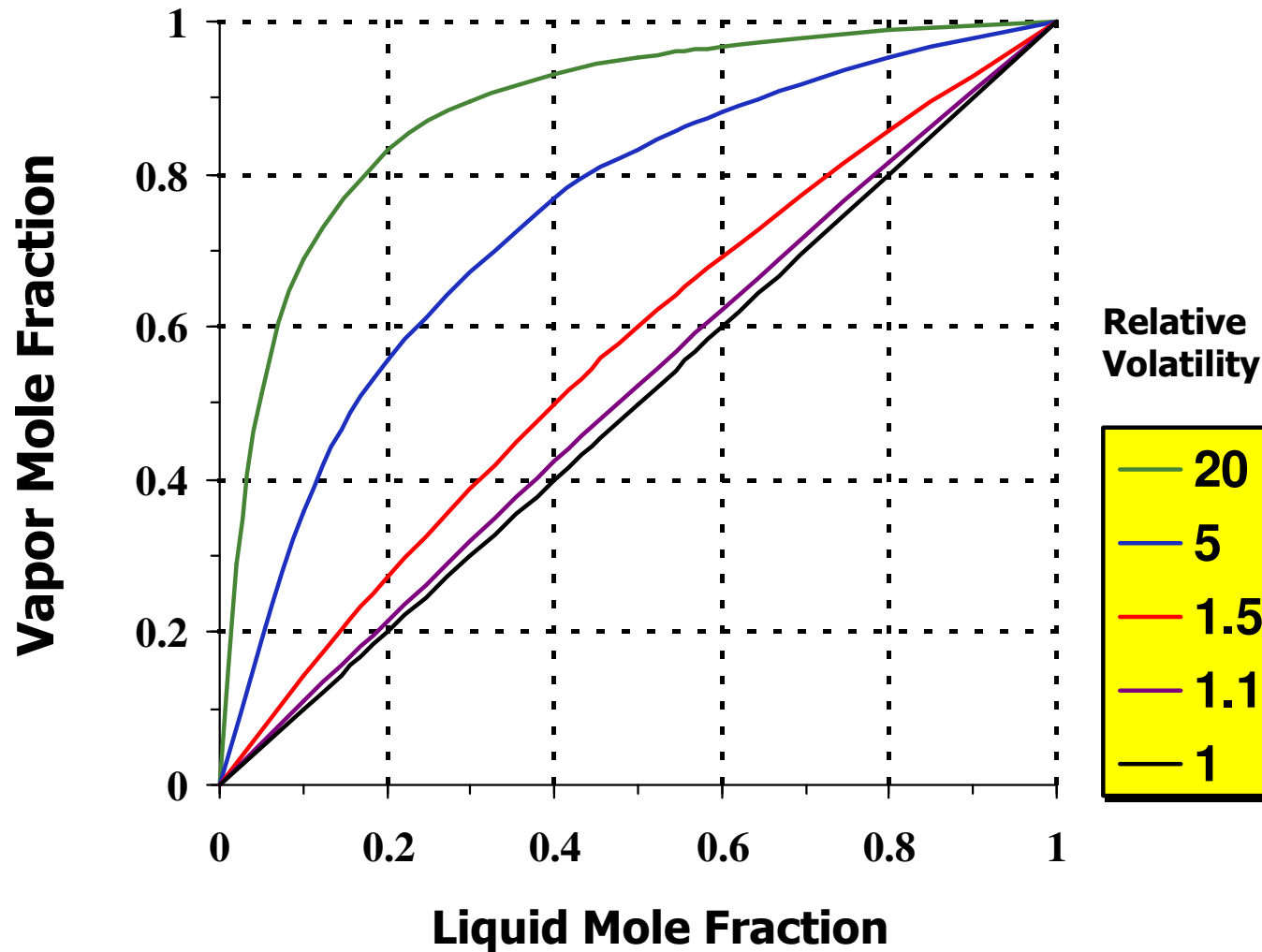
Operating Conditions

Pressure = 1 atm



Most Hydrocarbons do not act like Ethanol and Water. Their equilibrium curves look more like this.

Hydrocarbons



Relative Volatility is a relationship of composition ratios.

$$\text{Composition Ratio} = K_1 = Y_1/X_1$$

$$\text{Relative Volatility} = \alpha_{12} = K_1/K_2$$

The higher the value of Relative Volatility, the easier the separation and the fewer stages that are needed.

Hydrocarbon Example

Relative Volatility = 5.0

Top Purity = 97% Light Component

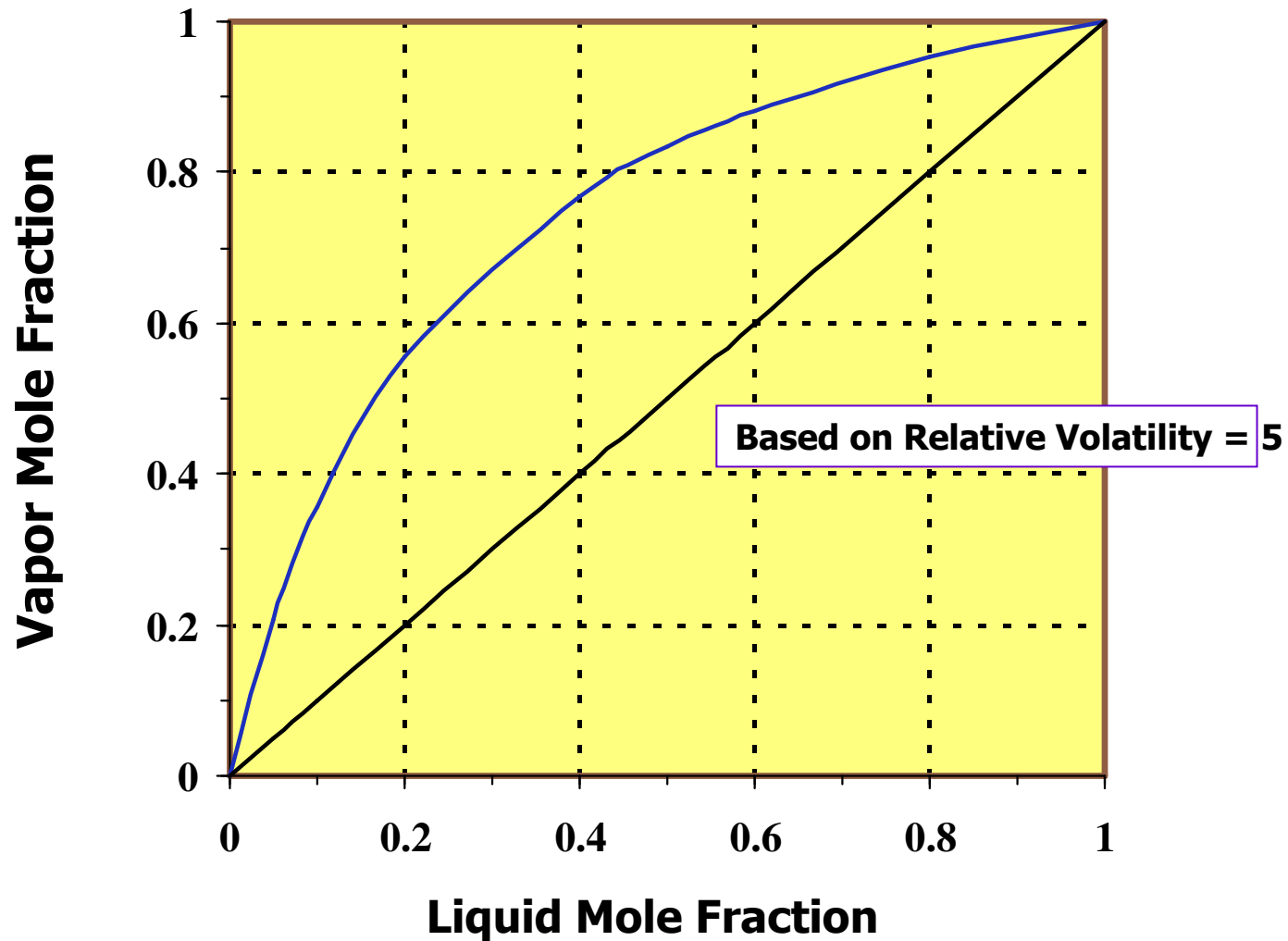
Bottom Purity = 93% Heavy Component

Feed = 45% Light Component & Saturated Liquid

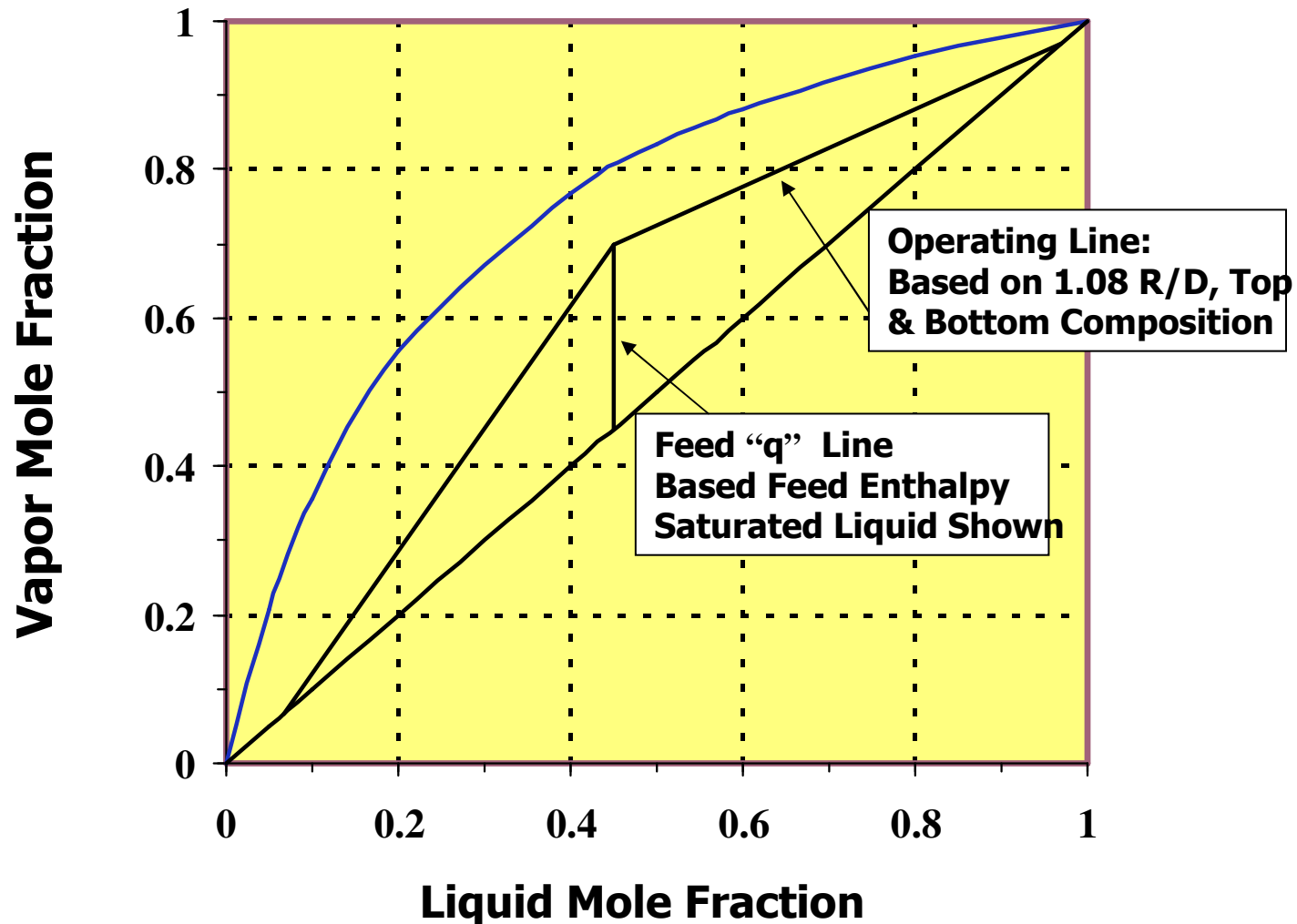
Reflux Ratio = 1.08

Resulting No. of Theoretical Stages = 6

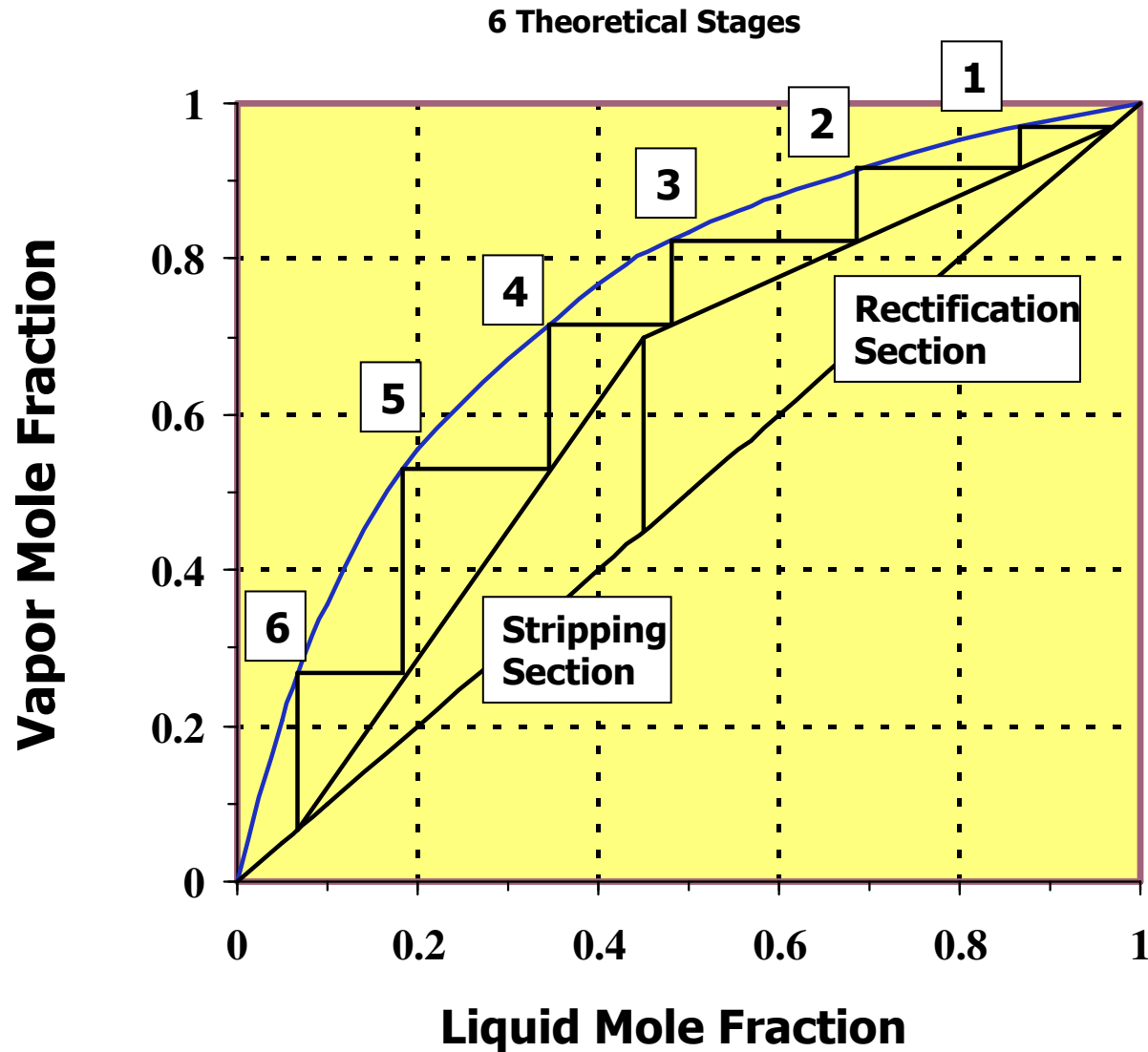
Equilibrium Curve



Equilibrium Curve



Isobaric McCabe Thiele Diagram



Simulation Tools do little more than what the previous McCabe-Thiele diagram did except add in Heat effect and how to handle multicomponent mixtures.

The key to a good simulation is getting the equilibrium data correct. The set of Equilibrium data is called a Model

Differences in boiling point is a good measure of the difference in relative volatility.

Increased pressure will make separations more difficult by driving the Relative Volatility lower

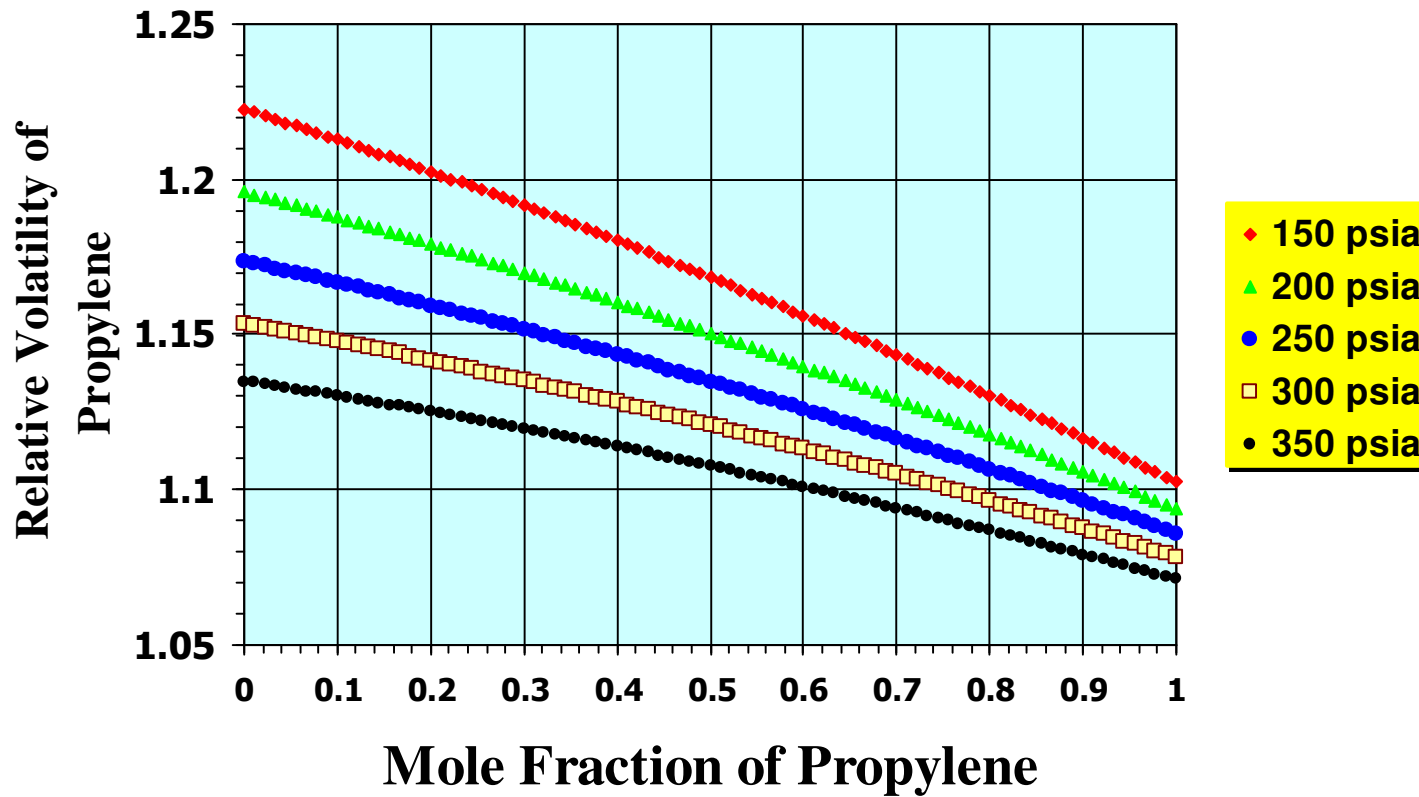
Typical Sizing – C4 Hydrocarbons

1 atmosphere

COMPOUND	Boiling Temp °C	Rel. Volatility to Butane
IsoButane	-11.85	1.650
IsoButylene	-6.85	1.269
1-Butene	-6.25	1.222
1,3 Butadiene	-4.45	1.136
n-Butane	-0.45	1.000
trans 2-Butene	0.85	0.869
cis 2-Butene	3.75	0.811
Vinyl Acetylene	8.05	0.704
1,2 Butadiene	10.85	0.585
Cyclobutane	12.55	0.414*
2-Butyne	27.05	0.244*

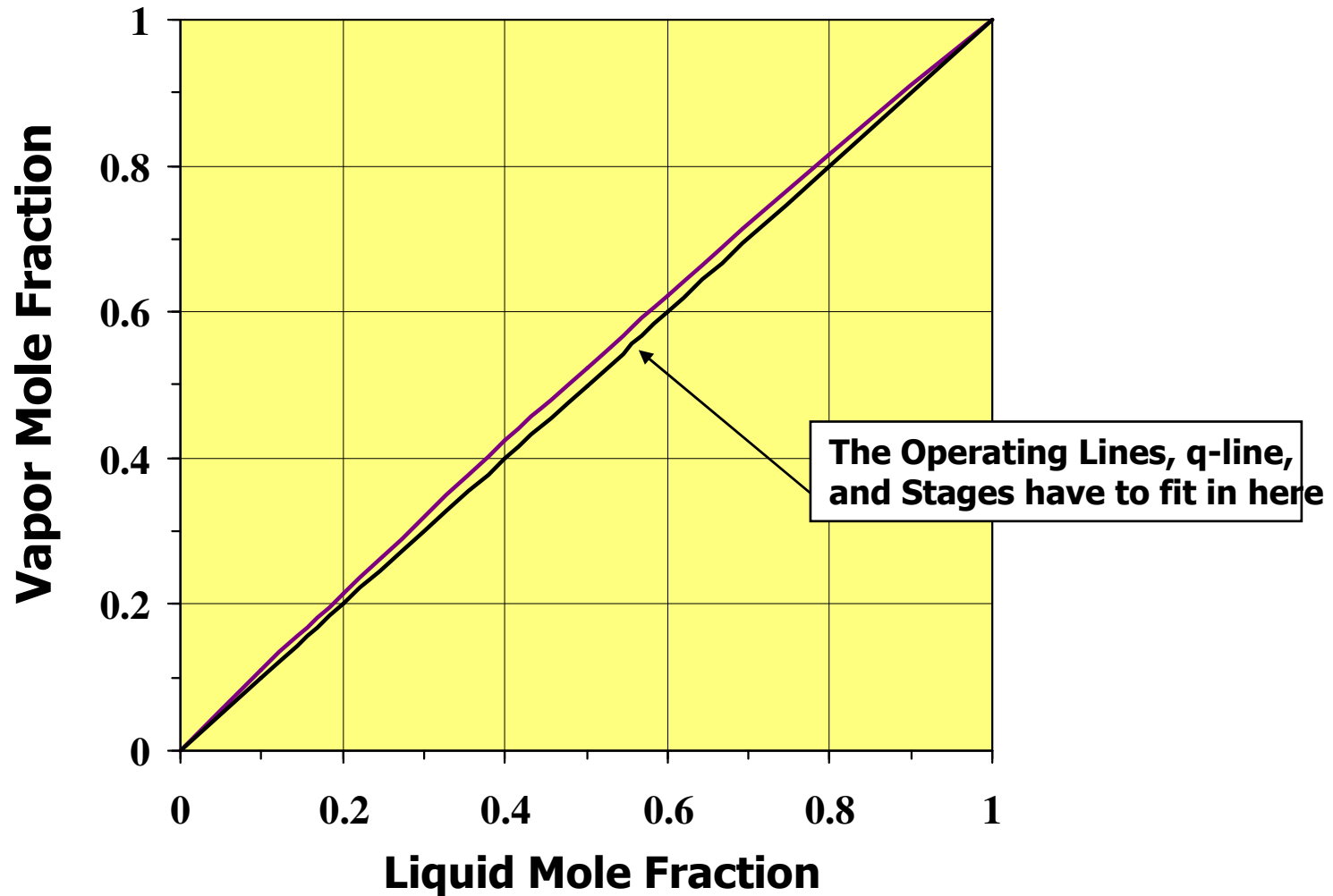
VLE Model Development

Propylene/Propane Binary

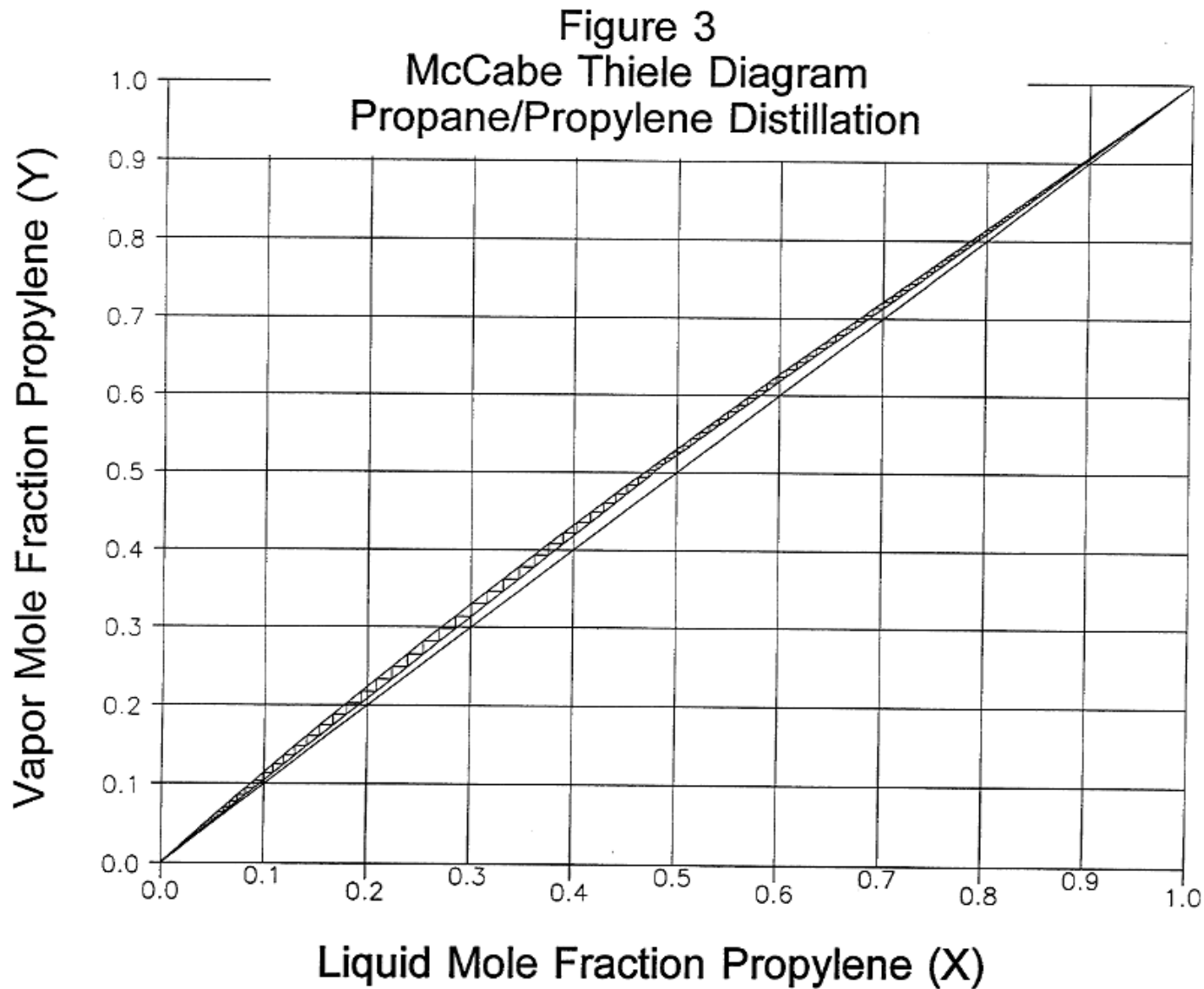


Process Considerations

1.10 Relative Volatility



Process Considerations



Distillation is:

The most common separation technique practiced today

It consumes enormous amounts of energy in both heating and cooling.

Can contribute to more than 50% of a plant's operating costs

Has been the focus of numerous studies by the US DOE for the past 30 years to reduce energy consumption.

Is Distillation the Best method of Separation?

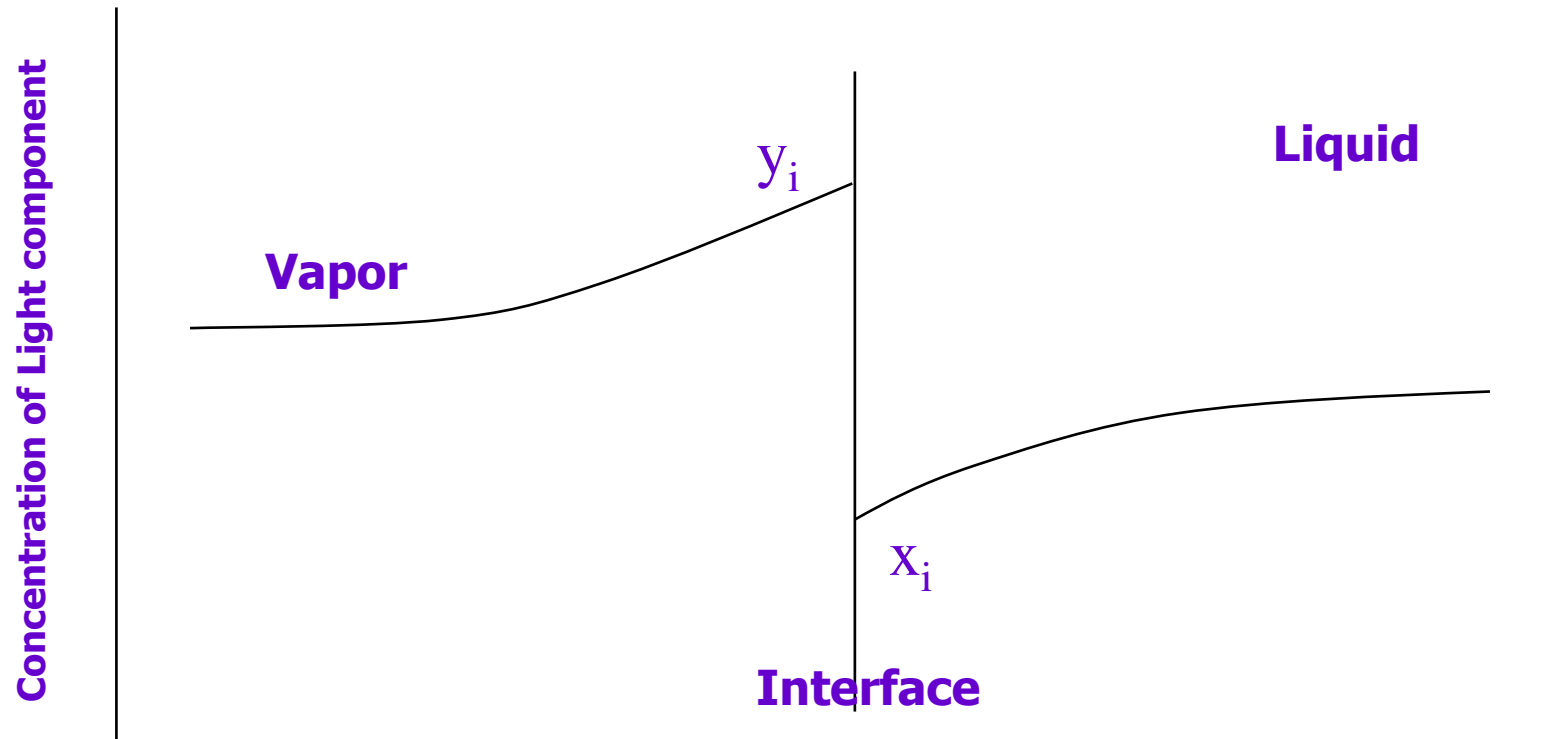
Are the boiling temperatures significantly different between the compounds you wish to separate ?

Are you trying to recover a small quantity of heavy material ?

Do your compounds thermally degrade?

Is there high corrosion or significant fouling?

The "Gory Details" are that mass transfer only occurs at the interface between the liquid and vapor phase. In a trayed tower this occurs at the bubble wall, in a packed tower this occurs at the thin film.



Simulations

Most Critical Aspect
of
Distillation Technology !

Process Considerations

Design

The previous diagrams can be made from Hand Generated McCabe-Thiele Diagrams or Computer Generated.

Not very often is a separation between a pure binary and at a pressure where VLE data is available

Computer Simulations (i.e. HYSYS, Pro-II, ASPEN) are usually used to determine the No. of Stages

Process Considerations

Design

Be careful with Computer Simulations. They are only as good as the data used to make them.

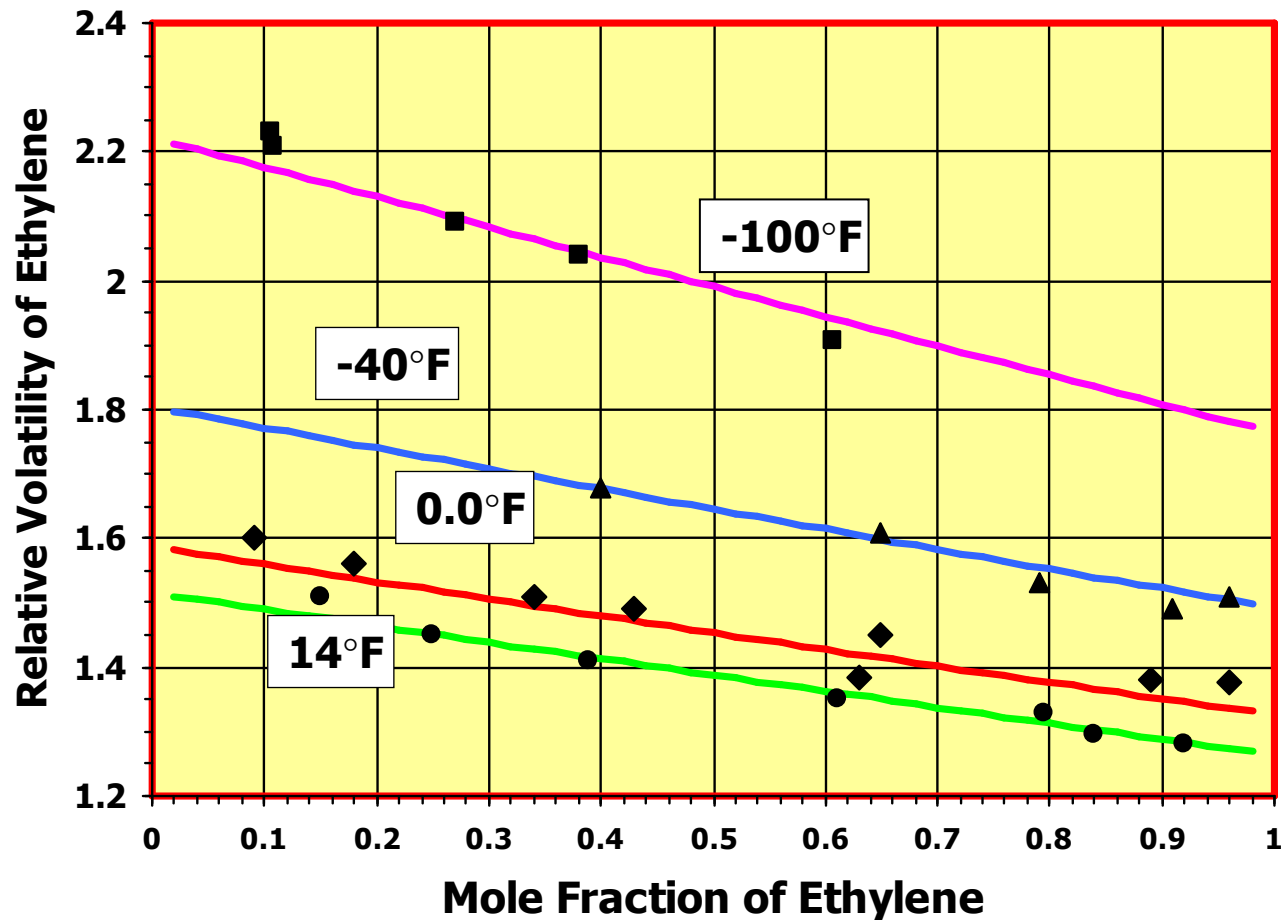
Superfractionators (i.e. C₂, C₃, & Xylene Splitters) simulations need to be checked against or fitted to available VLE data ! These towers have relative volatilities around 1.05 to 1.10 and small errors will result in a large effect on the number of Stages to use for design.

Watch out for Pinch Points and convergence problems!

Equilibrium Data – How good is it?

Ethylene/Ethane Binary

Isothermal Data from Hanson et. al. and Fredenslund et. al.



Detailed Design

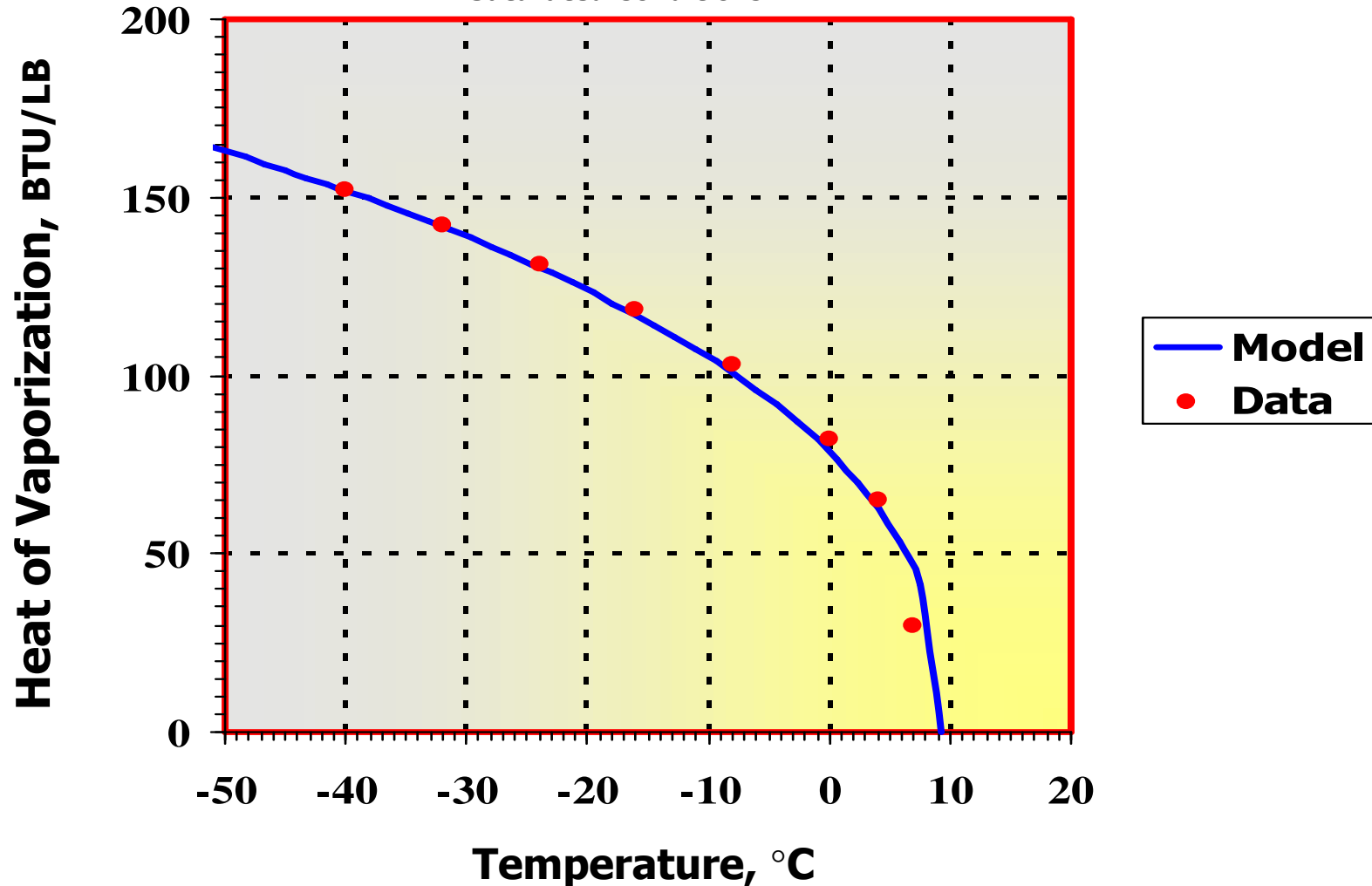
Most Simulation Packages will allow you to look at the physical properties that it uses. It is important to know if the Enthalpy Difference (Heat of Vaporization), for example, between the Vapor and Liquid Phases is correct.

Also important, is the Liquid Specific Heat for Proper calculation of Subcooled Feeds and Refluxes.

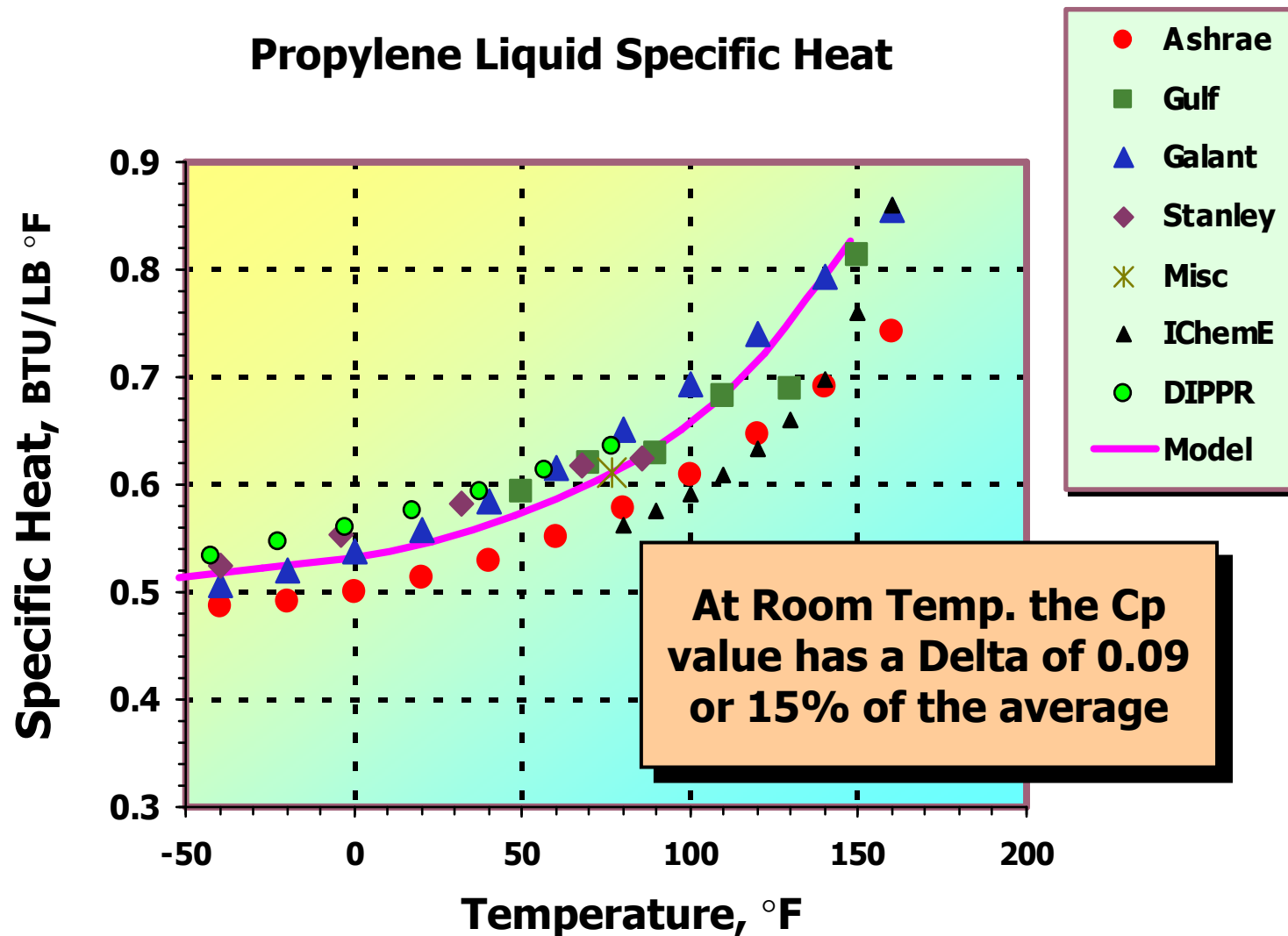
Check the Physical Properties !

Ethylene Heat of Vaporization

Saturated Conditions

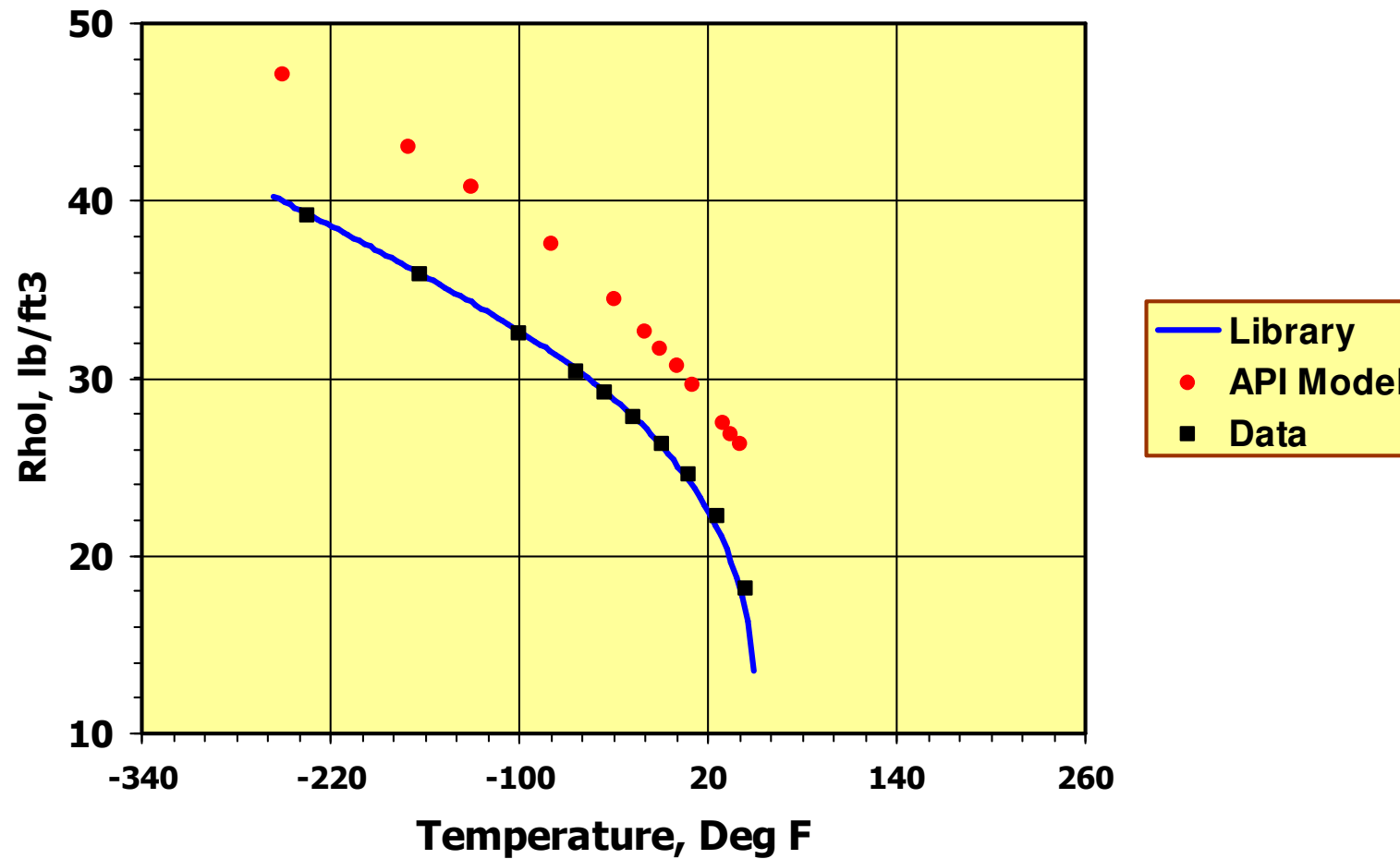


How does the model compare to data?



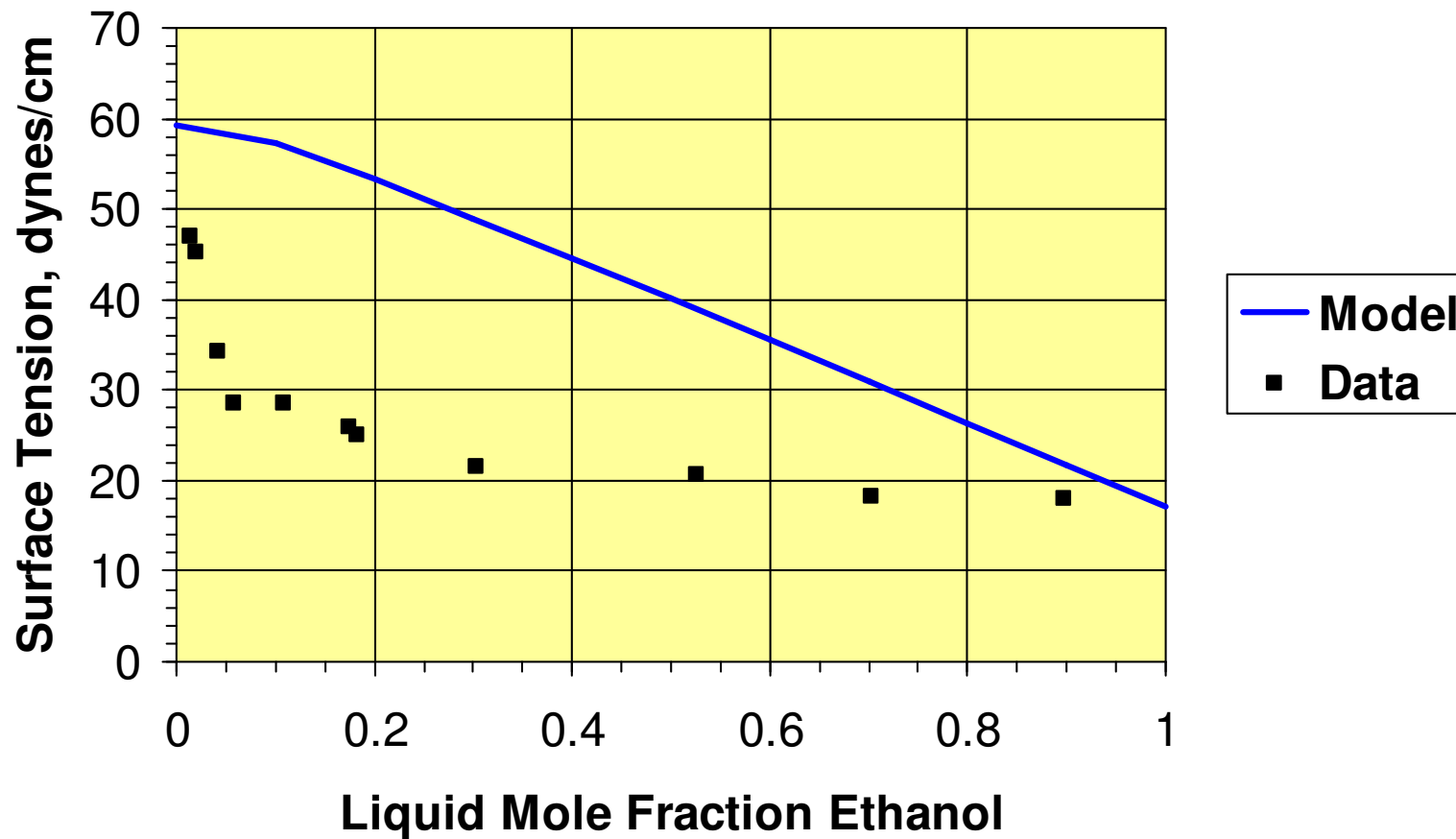
What correlations do we pick?

Ethylene Liquid Density



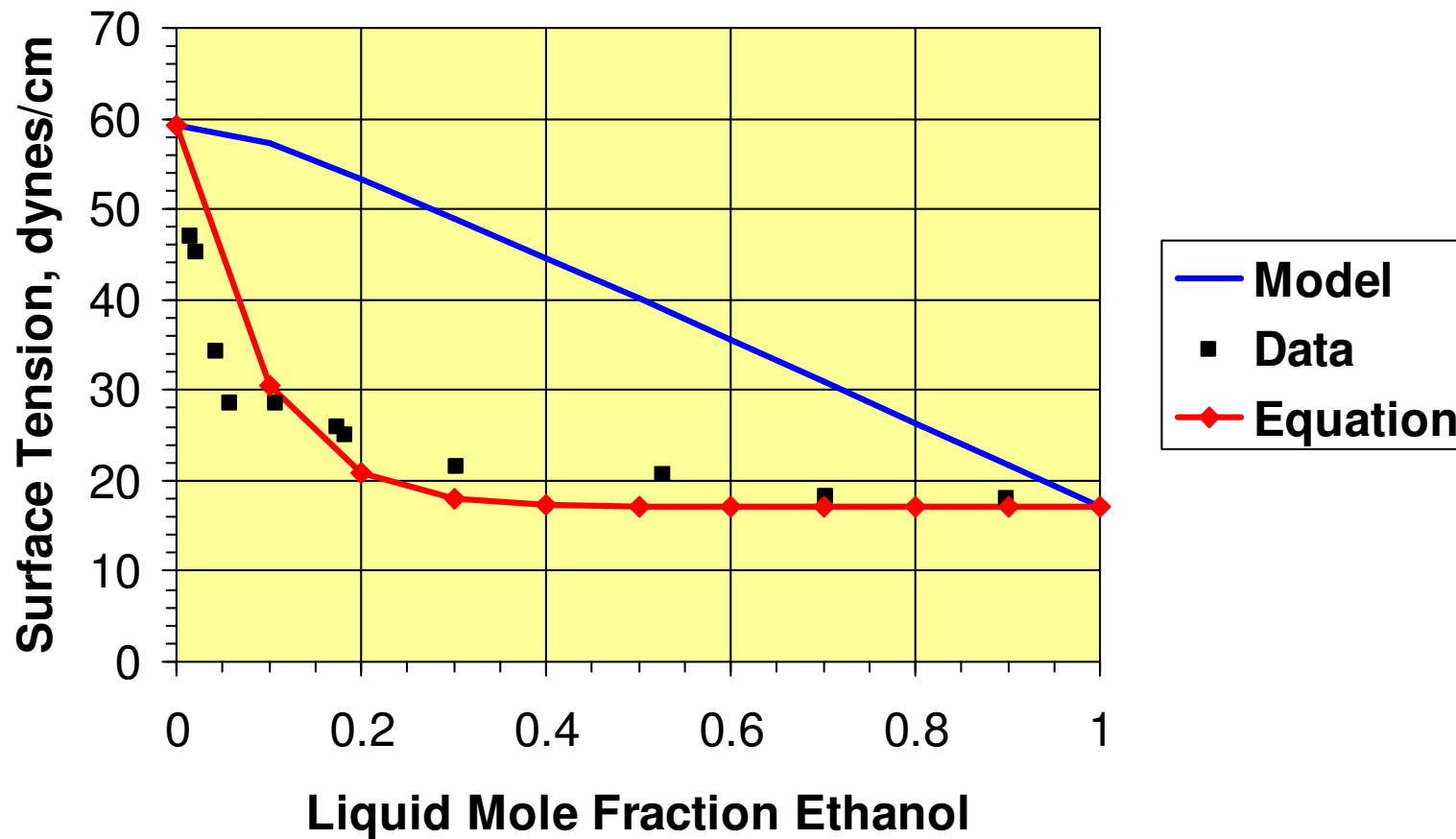
What about Mixing Rules?

Mixture Surface Tension
Ethanol-Water



Be careful – Model is only as good as the data.

Mixture Surface Tension
Ethanol-Water



$$\sigma = (X_{H_2O}^{11}) * \sigma_{H_2O} + ((1 - (X_{H_2O}^{11})) * \sigma_{EtOH})$$

The Whole Purpose

Once a Number of Theoretical Stages has been established, a **Heat & Material Balance** can be made for a given Feed and Product Rate.

The **Heat & Material Balance** Simulation allows the designer to determine the **Internal Loads and Physical Properties** needed for Design/Evaluation.