

Worksheet: Material balances in liquid-liquid extraction

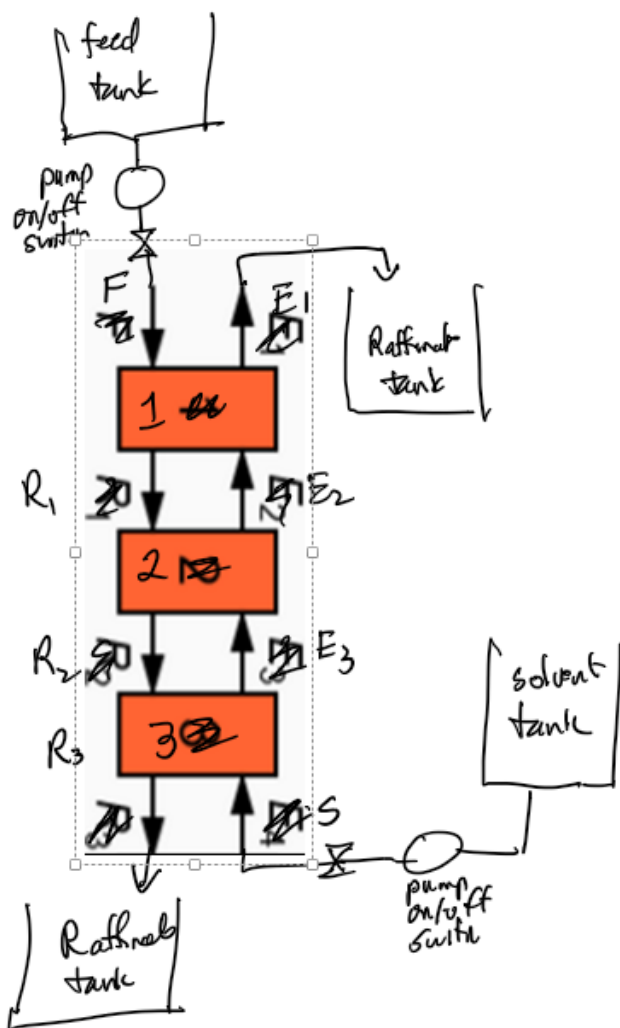
Name(s) _____

This experiment applies material balances to a liquid-liquid extraction process in which water as a solvent contacts an acetic acid/chloroform mixture in a steady-state counter flow experiment. Because acetic acid is much more soluble in water, it is extracted from the organic phase into the aqueous phase.

Student learning objectives

1. Be able to apply mass balances to a three-component system.
2. Be able to use phase equilibrium data to partially generate the phase envelope on a ternary phase diagram.
3. Be able to explain what happens during liquid-liquid extraction.

Equipment



The system consists of three stages where the aqueous phase contacts the organic phase in counter-current flow. The phases leaving each stage are in phase equilibrium.

S = solvent

F= feed

E = Extract

R = Raffinate

The water solvent extracts acetic acid solute from the organic feed (acetic acid, chloroform, and a small concentration of water), and the raffinate phase is enriched in chloroform because acetic acid is removed from it.

Questions to answer before starting the experiment

How many independent mass balances can you write for each stage of this liquid-liquid extraction? Explain.

The two phases contact each other in a counter-current flow. What might be the advantage of counter-current flow over co-current flow?

Would you expect the feed stream (acetic acid, chloroform, water) to increase or decrease its flow rate as it flows down the extraction system? Why?

Would you expect the water solvent stream system to increase or decrease its flow rate as it flows up through the extraction? Why?

Before starting

The feed inlet flow rate is 6.9 g/s and the mass fractions of its three components are:

Component	Mass fraction
Acetic acid	0.52
Chloroform	0.43
Water	0.05

The solvent inlet flow rate is 3.2 g/s, and it only contains water.

Procedure:

1. Turn on the two pumps and open the valves to start flow into the extraction system.
2. Allow time for the system to reach steady state, then determine the flow rates of the two exit streams by recording the change in the volumes in the extract and raffinate tanks while recording the time elapsed with your phone.

	Volume change	
Time	Extract tank	Raffinate tank

Calculate volumetric flow rates and mass flow rates. The density of the fluid in the extract tank is 1.04 g/cm^3 . The density of the fluid in the raffinate tank is 1.48 g/cm^3 .

Stream	Volumetric flow rate (cm^3/s)	Mass flow rate (g/s)
Extract		
Raffinate		

Measure the compositions of each of the streams by taking a sample and injecting it into a GC, which has been calibrated to yield mass fractions.

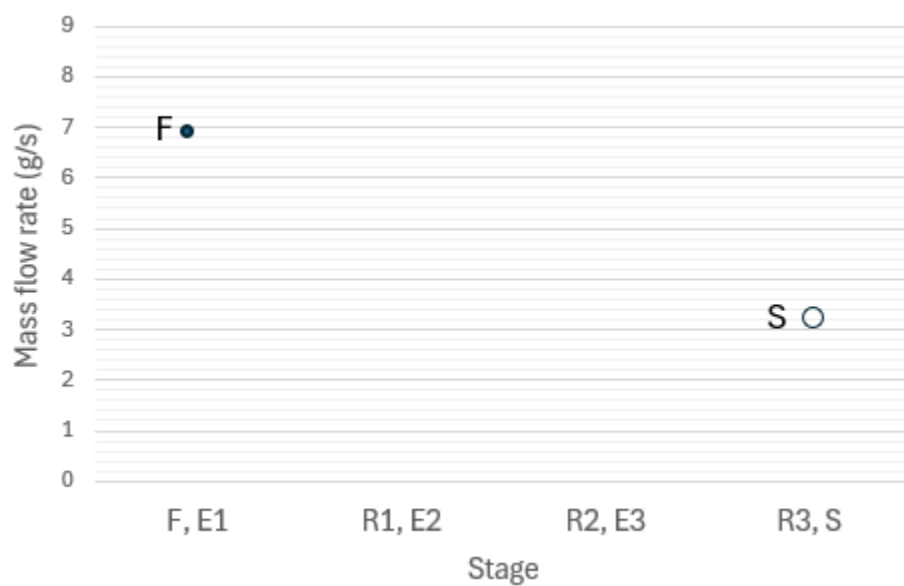
		Mass fractions		
Stream		Acetic acid	Chloroform	Water
E ₁				
E ₂				
E ₃				
R ₁				
R ₂				
R ₃				

Data Analysis:

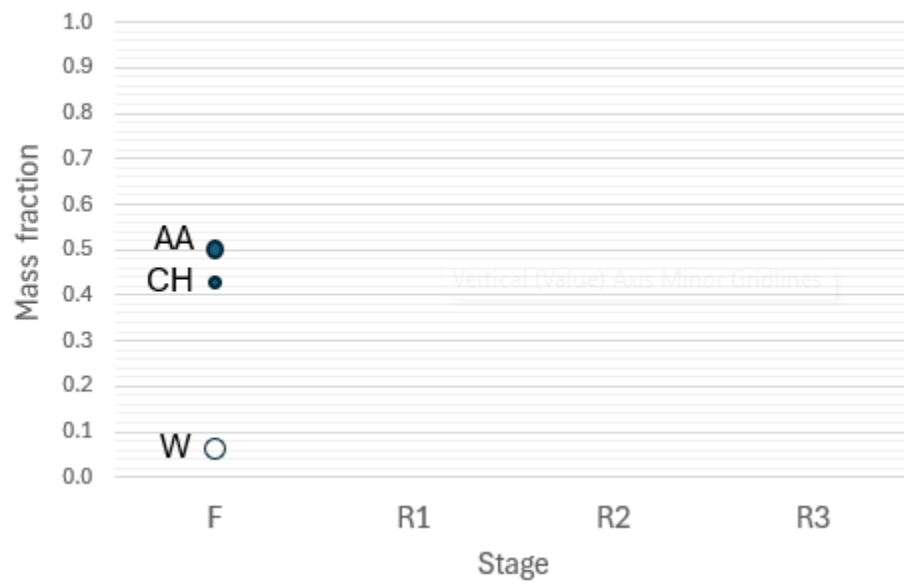
Carry out component mass balance for each stage in order to determine the flow rates of R₁, R₂, E₂, and E₃

Stream	Mass flow rate (g/s)
R ₁	
R ₂	
E ₂	
E ₃	

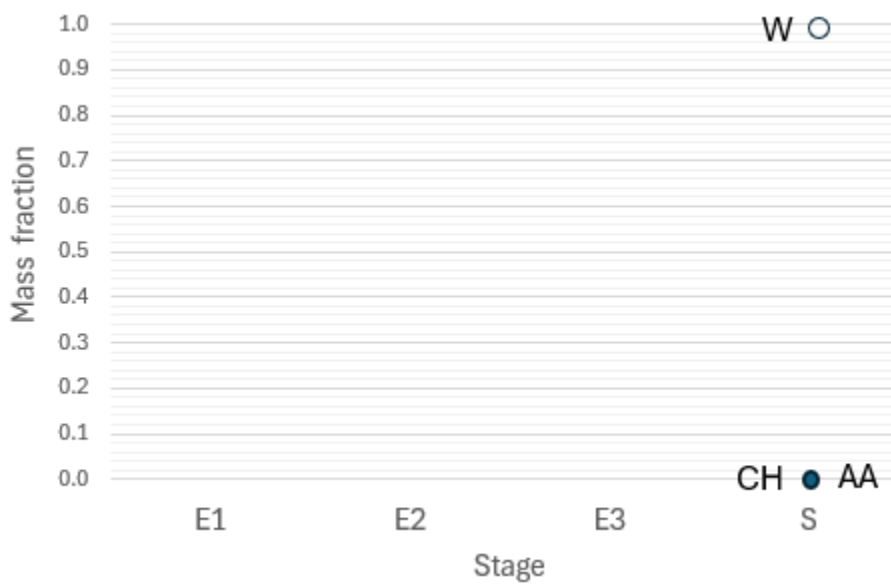
Plot the flow rates of the streams in each direction and explain the trends.



Plot the compositions of each component for the feed flow stream and explain the behavior.

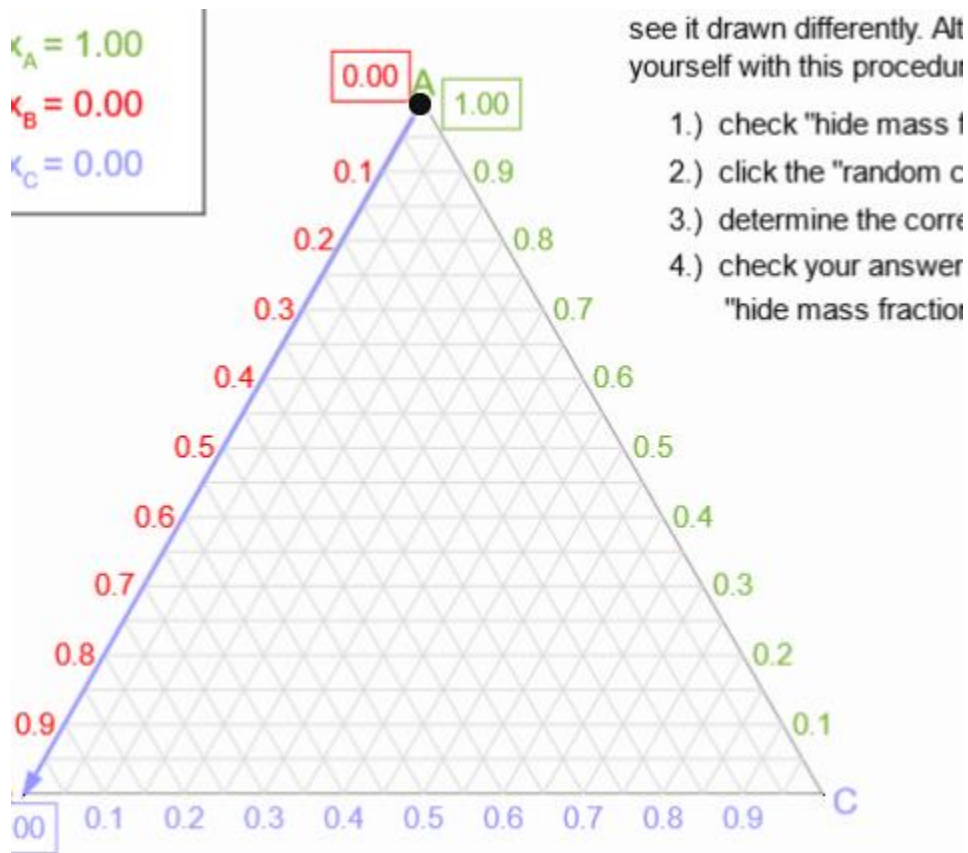


Plot the compositions of each component for the solvent flow stream and explain the behavior.



The streams leaving each stage (e.g., E_2 and R_2) are in equilibrium. This means tie lines connect the two composition points that are in equilibrium on a ternary phase diagram. The two points are on the phase envelope. From the exit compositions from the three stages, locate the six points on a ternary phase diagram and draw as much of the phase envelope as you can.

(Note, we want a diagram like the one below without the text or the black point. The A corner should be labeled acetic acid, the B corner (bottom left) should be labeled water, and the C corner should be labeled chloroform)



Questions to answer

1. Where might these measurement have errors?
2. What safety precautions would you take to carry out this experiment in the laboratory?