

Experiment 28B

MLE 8/16/16

DETERMINATION OF THE RELATIVE SOLUBILITY OF CROTONIC ACID¹

MATERIALS: Each hood needs: 250. mL Erlenmeyer flasks (4), 125. mL Erlenmeyer flask (1), rubber stoppers (4), polyethylene “slipcover” seals (4), 150. mL beakers (3), 50.0 mL graduated cylinders (4), 50.00 mL buret (1), ring stand and buret clamp (1), 25.00 mL pipet (1), 120 mL 0.5 M crotonic acid, 100 mL 0.1 M NaOH, 100 mL 1-pentanol, 100 mL toluene.

PURPOSE: To determine the partition ratio of crotonic acid in several two-phase solutions and to predict and determine the best solvent for dissolution of the acid.

OBJECTIVES: By the end of this experiment, the student should be able to demonstrate proficiencies in the following areas:

1. Understanding of intermolecular forces
2. Proper creation and management of a two-phase organic solution.
3. Titration of an acidic aqueous solution.
4. Calculation of solubility partition ratios.
5. Interpretation of solubility partition ratios.

PRE-LAB: Read this entire document before coming to lab.

DISCUSSION:

Chemical substances dissolve in solvents according to the nature, similarity, and strength of the intermolecular forces which can be formed between solute and solvent. It is possible to measure absolute solubility, and thus gain insight into the solute-solvent interactions. Alternatively, it is often useful to assess relative solubility of a solute in two non-miscible solvents that are in physical contact with one another. These two-phase solvent systems are composed of a water-based aqueous phase and an organic phase based on a non-polar or slightly polar organic solvent. The ability to partition solutes between the two non-miscible solvents is a fundamental separation technique in organic chemistry. Mathematically, the propensity of a solute to dissolve in one solvent vs. another is given as the partition ratio K_D (also called the distribution constant). By convention, the concentration in organic solvent is the numerator².

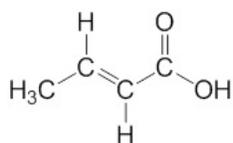
$$K_D = \frac{[\textit{solute}]_{\textit{organic phase}}}{[\textit{solute}]_{\textit{aqueous phase}}}$$

¹ Based on Koubek, E., Johnstone, P., *Journal of College Science Teaching*, 3, 241 (1978)

² IUPAC Gold Book Compendium of Chemical Terminology (<http://goldbook.iupac.org/P04440.html>) accessed 11/18/10

Remember the intermolecular principle of “like dissolves like”. K_D is higher than 1 for a solute with intermolecular forces more similar to the organic solvent. K_D is lower than 1 for a solute with intermolecular forces more similar to water. In this experiment, the solute to be examined is *trans*-2-butenoic acid, an organic acid which has the common name crotonic acid. Its structure is shown below. The carboxylic acid region of crotonic acid (-COOH) is polar, while the hydrocarbon region (CH₃CHCH) is non-polar, making this an amphipathic molecule. Due to the differing intermolecular forces of the two ends, the compound is soluble to some extent in both water and organic solvents. In this experiment the relative solubility of crotonic acid will be studied using water, 1-pentanol, and toluene as solvents. Neither pentanol nor toluene is significantly soluble in water, though pentanol and toluene are significantly soluble in each other. Therefore, the comparisons will be made between pentanol and water or between toluene and water. By comparing the relative amount of acid in the aqueous phase of these systems, it will be possible to determine K_D values and a relative order for crotonic acid solubility in the three solvents. The amount of crotonic acid present in the aqueous phase at equilibrium will be determined by titration. (Titrations of organic solutions are difficult and, in this lab, unnecessary.)

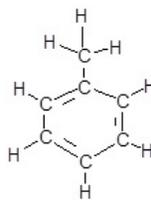
crotonic acid
CH₃(CH)₂COOH



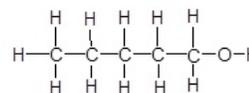
water
H₂O
density = 1.0 g/mL



toluene
C₆H₅CH₃
density = 0.87 g/mL



1-pentanol
C₅H₁₁OH
density = 0.81 g/mL



PROCEDURE:

1.) Make up four solutions in 250. mL flasks (in the hood) as outlined in the following table.

Solution #	0.5 <u>M</u> crotonic acid	H ₂ O	organic solvent
1	20.0 mL	30.0 mL	50.0 mL toluene
2	40.0 mL	10.0 mL	50.0 mL toluene
3	20.0 mL	30.0 mL	50.0 mL pentanol
4	40.0 mL	10.0 mL	50.0 mL pentanol

- 2.) Stopper each flask with a rubber stopper (protected from the solution with a polyethylene slipcover) and shake well for 5 minutes. Vent the solution periodically, particularly early on, but take care not to lose any of the solution.
- 3.) Allow the solution to settle until the phases have separated.
- 4.) Remove 25.00 mL of the aqueous phase (is that the top layer or the bottom layer?) from each solution with a 25.00 mL pipet and place in a 125 mL Erlenmeyer flask.
- 5.) Add a few drops of phenolphthalein indicator to the aqueous phase sample and titrate each with 0.1 M NaOH using a buret. Be sure to record the ACTUAL NaOH concentration and crotonic acid concentration on your data sheet on the next page.
- 6.) The contents of the titration flask may be poured into the sink. The remainder of each solution contains organic material and must be disposed of in the instructor hood.

EXPERIMENTAL DATA:

Name: _____

Record the actual crotonic acid concentration: _____

Record the actual NaOH concentration: _____

Toluene

mix #1 _____ initial buret reading

_____ final buret reading

_____ vol. of 0.1 M NaOH

mix #2 _____ initial buret reading

_____ final buret reading

_____ vol. of 0.1 M NaOH**Pentanol**

mix #3 _____ initial buret reading

_____ final buret reading

_____ vol. of 0.1 M NaOH

mix #4 _____ initial buret reading

_____ final buret reading

_____ vol. of 0.1 M NaOH**DATA TREATMENT:** (Show your work for solution #1)

1. Calculate total moles of crotonic acid delivered for solution #1.
2. Calculate moles of crotonic acid present in the aqueous phase of solution #1. Remember that you only titrated 25 mL of the 50 mL aqueous phase!
3. Calculate moles of crotonic acid that must have been in the organic phase of solution #1.
4. Calculate the concentration of crotonic acid in the organic phase of solution #1
5. Calculate the concentration of crotonic acid in the aqueous phase of solution #1.

6. Calculate the K_D of crotonic acid in solution #1.

7. Perform similar calculations for the other three solutions and record the results in the tables below:

Toluene

Exp.	Total moles of crotonic acid	moles of crotonic acid in organic phase	moles of crotonic acid in aqueous phase	$[\text{crotonic}]_{\text{org}}$	$[\text{crotonic}]_{\text{aq}}$	K_D
1						
2						

Pentanol

Exp.	Total moles of crotonic acid	moles of crotonic acid in organic phase	moles of crotonic acid in aqueous phase	$[\text{crotonic}]_{\text{org}}$	$[\text{crotonic}]_{\text{aq}}$	K_D
3						
4						

8. Calculate the average K_D for each solvent.

QUESTIONS:

1. For each solvent, you performed two different determinations of K_D using different amounts of crotonic acid. Should the K_D values for the two runs be the same? Why?

2. Use the partition ratios you found for crotonic acid in the pentanol/water and toluene/water solvent systems to rank the solubility of crotonic acid in the three solvents.

least solubility: _____

greatest solubility: _____

3. Explain the order of relative solubilities determined in #1 in the context of the type and strength of intermolecular forces.

4. Both water and 1-pentanol contain $-O-H$ in their structures and are both capable of dipole and hydrogen-bond intermolecular forces. Why then are these apparently similar solvents not soluble in one another?