Partition coefficient of acetic acid

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Objectives:

1) Use titrations to determine K_D, the partition equilibrium constant.
2) Understand the effect of solvent layer volumes on K_D and extraction.
3) Gain experience using separatory funnels and doing titrations in different solvents.

Introduction:

In this experiment, we will be using titrations to determine an equilibrium constant, K_D. This equilibrium constant describes the **partitioning** of a compound between two immiscible liquids, usually water and an immiscible organic solvent such as methylene chloride or ether. In essence, it describes what is going on when you use a separatory funnel in the organic lab to do an **extraction**!

We will observe how acetic acid partitions between water and octanol.

The equilibrium reaction is

$$\text{Acetic acid}_{(\text{water})} \leftrightarrows \text{Acetic acid}_{(\text{octanol})}$$

where the acetic acid in water is the "reactant". So the corresponding equilibrium expression is

$$K_D = \frac{[\text{AcOH}]_{\text{oct}}}{[\text{AcOH}]_{\text{wat}}}$$

Notice that K_D is a ratio of concentrations. The convention is that when water is one of the solvents, the concentration in water is in the denominator. Octanol/water partitioning is important in the pharmaceutical industry, and the notation $\boldsymbol{K_{ow}}$ is often used specifically for the partition coefficient between those two solvents. To make matters just a bit more confusing, the **base 10 log** of $\boldsymbol{K_{ow}}$ is often reported; *that* is called **log P**!

We're also going to determine the effect of using different volumes of octanol and water on the partitioning of acetic acid between the two solvents. We will measure the concentrations of the acid in each layer by titrating each layer with NaOH. Just like in our first experiment, we will be looking for the pink color of the phenolphthalein end point, but we won't be using the pH meters this time.

You've titrated acetic acid in water before. It is a bit tricky to titrate the octanol layer, so we will practice that in a control titration, and we will discuss what is going on in this titration in class. You will likely want to take notes, so that this information can appear in your final lab report.

Procedure:
These titrations will be done using NaOH as the titrant and phenolphthalein as the indicator. Be sure to coat the inside of your burette with the standardized 0.10xx M NaOH solution and rinse before filling the burette. You'll need to clear bubbles from the burette tip. Refill the burette as needed and **don't let the burette tip drain.** You do not have to start at the 0.00 mL mark. Always record your starting volume in your lab notebook.

Each group will do both control titrations; the titration in the presence of octanol requires some practice. Then each group will then be assigned several volumes ratios for the partitioning measurement.

Waste from water layer titrations can go down the drain. All waste from octanol layer titrations should go into the large waste separatory funnel.

Control titrations

acetic acid in water

1. Add roughly 25 mL of DI water to a 250 mL Erlenmeyer flask using a graduated cylinder. Add a medium-sized stir bar.
2. Using a micropipette, add 2.00 mL of white vinegar to the water.
3. Add ~2 drops of phenolphthalein indicator.
4. Adjust the stirrer to 500 rpm.
5. Read and record the starting volume of NaOH on your burette to two decimal places.
6. Titrate using 0.10xx M NaOH to a **light pink** endpoint. If it turns hot pink, you've gone too far.
7. Record the final volume of NaOH to two decimal places (four sig figs).
8. Determine the volume of NaOH needed to reach the endpoint.
9. The titration solution can be rinsed down the sink

acetic acid in octanol

1. Add roughly 10 mL of DI water and roughly 25 mL of **octanol** to a 250 mL Erlenmeyer flask using a graduated cylinder. Add a medium sized stir bar.
2. Using a micropipette, add 2.00 mL of white vinegar to the mixture.
3. Add ~2 drops of phenolphthalein indicator.
4. Adjust the stirrer to 500 rpm.
5. Read and record the starting volume of NaOH on your burette to two decimal places.
6. Titrate using 0.10xx M NaOH to a **light pink** endpoint. If it turns hot pink, you've gone too far.
7. Record the final volume of NaOH to two decimal places (four sig figs).
8. Determine the volume of NaOH needed to reach the endpoint.
9. Mixtures with octanol cannot go down the sink. Pour the titration mixture with octanol into the large waste sep funnel.

Note: Before your endpoint occurs, the solution may turn hot pink. If this occurs, let the solution stir for about ~1.5 minutes. If it goes back to colorless, then you have not passed your endpoint and you should continue your titration. If it stays hot pink, then you have passed your endpoint.

Partition measurement

acetic acid partitioned between water and octanol

You will be assigned two or three volume ratios for the two layers, water and octanol. Measure the volumes with a grad cylinder. The total volume of the mixture should be about half the volume of the sep funnel you are using for the partition. For example, if you use a 125 ml sep funnel, then the total volume of the mixture should be 40 to 80 mls.

1. Fill your burette with the NaOH titrant.
2. Add the assigned volumes of DI water and octanol to a separatory funnel. Make sure the stopcock is closed before adding the liquids. If you are unfamiliar with the use of a separatory funnel, ask your instructor for help.
3. Using a micropipette, add 2.00 mL of white vinegar to the mixture. Cap the sep funnel and shake it vigorously for at least one minute. This will distribute the acetic acid between the two layers. Thorough mixing is important to reach equilibrium.
4. Set the sep funnel in the ring stand and allow the mixture to settle and the layers to become clearly separated.
5. Once the layers have separated, remove the cap (so you don't create a vacuum!), open the stopcock, and allow *just* the bottom layer to flow into an Erlenmeyer flask. *When the bottom layer is almost completely drained, try doing quick half-rotations of the stopcock to make sure that you remove the whole bottom layer without removing part of the top layer.*
6. Titrate the bottom layer as you did the appropriate control. (This solution can be rinsed down the sink.)
7. **Do not refill your burette!**
8. Drain the top layer into an Erlenmeyer flask.
9. Rinse the separatory funnel with about 10 mL of DI water and drain the rinse water into the same Erlenmeyer flask. *Why are you doing this?*
10. Titrate the top layer as you did the appropriate control. (This mixture should go into the large waste sep funnel.)

Add the volumes of titrant used in the two titrations. What is the total volume of titrant consumed by **both layers**?

Compute and record K_D, taking into account the volume of each layer.

What quantities usually associated with a titration are not important in computing K_D?

Proceed to the next partition experiment with a different ratio of volumes. If you have finished all the assigned measurements, empty your burette, rinse it with DI water, and hang it upside down. Dispose all mixtures containing octanol into the large waste sep funnel.