Lab Owl Announcement:

Upon completion of this lab, log onto OWL. A Lab Owl section should now appear in your courses and your first assignment, Lab Owl: Exp 2, should appear in this section. You have until the next scheduled laboratory to complete this assignment. Three more assignments will appear here as the semester progresses. Remember, these Lab Owls are worth 25% of your laboratory grade.

Introduction:

Stoichiometry and Solids

When dealing with solids that one can weigh on a balance, determining the number of moles in a particular sample is simply:

Stoichiometry and Solutions

When dealing with solutions, one is usually concerned with concentration. Concentration is not an unfamiliar term, in that in the geographical and political context the term "people concentration" is often used in allocating funds and describing population densities in various areas of the continent. "People concentration" is simply the number of people in a given region divided by the area of that region. While the number of people in two given regions may be the same, the area of the regions may be vastly different, and thus represent different concentrations.

In the chemical world concentration is usually expressed in terms of the quantity of a substance (moles) per liter of solution and is called Molarity (M).

Concentration =
$$M = \frac{\text{mol of a substance}}{\text{Volume (Liters) of the solution}}$$

More often abbreviated to:

$$M = \frac{\text{mol}}{V(L)}$$

Determining the Molarity of a Sodium Hydroxide Solution

The reaction between an acid and base produces a salt and water, something that you may have heard before. In the reaction that you will be investigating, the acid is potassium hydrogen phthalate (KHP), and the base sodium hydroxide (NaOH).

$$NaOH(aq) + KHC_8H_4O_4(aq) = KNaC_8H_4O_4(aq) + H_2O(1)$$

As long as a reaction goes to completion, if one knows the following information:

- the balanced chemical equation for the reaction occurring (given above).
- the exact quantity of one of the reagents (the KHP, a white solid, the mass of which you will weigh)

then one can always determine the exact amount of any other substance involved in the reaction. This is the essence of the technique called quantitative chemical analysis. In this experiment we are going to determine the concentration of a sodium hydroxide solution by adding it to a known amount of KHP.

The process used to carry out this addition is called titration. This is where one of the reagents (in our case NaOH) is added slowly from a buret to a known quantity of the other reagent (KHP). The point at which sufficient reactant has been added to just complete the reaction is called the equivalence point. This is what we would like to determine since at this point we have added sufficient amount (moles) of NaOH to react with the entire amount (moles) of the KHP. A method to determine this visually, is to add a dye (referred to as an indicator) that changes

color at or extremely close to this point. The point at which the indicator actually changes color is referred to as the end point. As far as we are concerned at this stage, the end point and the equivalence point are the same. Later you will see that the criteria used for choosing an indicator in a particular titration is, how close the indicators end point (where it changes color) is to the equivalence point of the titration.

Calculating the Molarity of the Sodium Hydroxide Solution

In order to determine this we need to know the number of moles of NaOH that reacted with the KHP and the volume in liters of the NaOH solution that contained that said number of moles of NaOH.

You know the mass in grams of the KHP used in the titration:

We have a balanced chemical equation in which we now know the exact quantity of one of the reagents (KHP) – $NaOH(aq) + KHC_8H_4O_4(aq) = KNaC_8H_4O_4(aq) + H_2O(l)$ – thus we can determine the number of moles of NaOH that reacted with the KHP.

$$\frac{\text{\# mol KHP}}{\text{x KHP}} = \text{\# mol NaOH} \qquad \frac{\text{Where } x \text{ is to coefficient in front of the NaOH and}}{\text{the KHP respectively in the balanced chemical equation.}}$$

Finally, the molarity of the sodium hydroxide solution can be determined:

$$M = \frac{\text{\# mol NaOH}}{V(L)} \quad \begin{array}{c} \textit{Volume determined from the burst-final volume of the} \\ \textit{burst minus the initial volume-converted to liters.} \end{array}$$

Precision and Accuracy

These are two terms that often lead to some confusion. Precision is the degree to which a number of measurements are reproducible (how close each measurement is to one another). Accuracy, on the other hand, is how good the actual measurement is. How does it compare to the known value, or, in the case where it is not known, how close is it to identical measurements being made by other people on the same system. It is possible to have a high degree of precision yet lack accuracy. This is often either the failure in some aspect of the experimental design or of the operator. This lab is a case in point. You will endeavor to determine when the reaction has reached its equivalence point by observing a color change in a dye. It may well happen that for each trial you obtain a good degree of precision (reproducibility) but the color intensity to which you titrated each sample was a few drops beyond the actual equivalence point, hence poor accuracy.

As everyone makes up their own solution, we have no real test of accuracy on this experiment. However you will calculate a percent difference for each of your trials, which is a crude measurement of your reproducibility (precision). It will be crude due to the fact that a very small number of trials are used. Percent difference describes the difference between an experimentally determined value and a reference or accepted value. In this experiment, the molarity of sodium hydroxide is not actually known so the average value of the molarity is taken as the reference or accepted value. The percent difference in this experiment will, then, be determined by comparing the molarity in each separate titration trial with the average molarity.

% Difference =
$$\frac{\text{Trial '#' Molarity - Average Molarity}}{\text{Average Molarity}} \times 100$$
 $\frac{\text{You are aiming for a difference of } \pm 1\%.}{\text{You are aiming for a difference of }}$

Experimental Procedure

Making the NaOH Solution

- 1. Obtain about 26mL of ~1M sodium hydroxide in a, graduated cylinder.
- 2. Pour into a Florence flask, dilute with 174mL of distilled water, and mix thoroughly by swirling the contents.
- 3. In order to prevent evaporation, invert a 50mL beaker over the top of the Florence flask.

Trial Titration

This trial is used to get an estimate of the amount (volume) of NaOH that is required to completed react with the potassium hydrogen phthalate.

- 1. Weigh between 0.9 and 1.1g of potassium hydrogen phthalate into a 125mL Erlenmeyer flask and record the mass to the nearest milligram.
- 2. Add about 30mL of distilled water and two or three drops of phenolphthalein. Swirl the flask until the solid is completely dissolved.
- 3. Carefully place a stirring magnet into the flask.
- 4. Rinse a buret with distilled water and then with a 10-mL portion of the sodium hydroxide solution.
- 5. Now fill the buret with the sodium hydroxide solution and remove any air bubbles from the tip of the buret.
- 6. Take the initial burst reading to the closest 0.01 or 0.02mL. Record data to TWO decimal places, with no readings coarser than \pm 0.02mL.
- 7. While stiring the Erlenmeyer slowly add the sodium hydroxide from the buret until the solution in the Erlenmeyer turn pink. Try to stop it at the first hint of pink. Record the final volume of the buret.
- 8. Calculate the
 - i. Moles of NaOH added
 - ii. The molarity of the NaOH solution

Exact Titration

- 1. Weigh close to the same quantity*1 of potassium hydrogen phthalate as in the *Trial Titration* into a 125mL Erlenmeyer flask and record the mass to the nearest milligram.
 - *1 Its better to be a little over than under.
- 2. Add about 30mL of distilled water and two or three drops of phenolphthalein. Swirl the flask until the solid is completely dissolved.
- 3. Carefully place a stirring magnet into the flask.
- 4. Refill the buret with the sodium hydroxide solution.
- 5. Take the initial buret reading to the closest 0.01 or 0.02mL. Record data to TWO decimal places, with no readings coarser than \pm 0.02mL.
- 6. While stiring the Erlenmeyer quickly add the NaOH to within 5mL of the volume recorded in the *Trial Titration*.
- 7. Drop wise add the sodium hydroxide from the buret until the solution in the Erlenmeyer turns a faint permanent pink. Record the final volume of the buret.

- 8. Repeat this procedure with a second sample of potassium hydrogen phthalate, following the above procedure
- 9. Calculate the
 - i. Moles of NaOH added.
 - ii. The molarity of the NaOH solution.
 - iii. The average molarity of the NaOH
 - iv. A % Difference for each trial

Disposal

- 1. Once the titration is complete you may dispose of the contents of the Erlenmeyer flask down the sink. Take care not to lose the magnetic stir bar.
- 2. Any remaining sodium hydroxide solution should be discarded in the container provided in the fume hood. Ask your TA if you cannot locate it.