Chemical Reactions: Titrations

ORGANIZATION

- Mode: laboratory work, work in pairs
- Grading: lab notes, lab performance (titration accuracy), and post-lab report
- Safety: goggles, lab coat, closed-toe shoes, long pants/skirts and sleeves; latex or nitrile gloves recommended, use care with acids and bases

GOAL:

To titrate standard and unknown samples of acids with a strong base, and also to use data to determine the concentration of unknown samples.

Chemical Classification and Potential Hazards of Chemicals Used in Experiment

CHEM 115 Expt. 10	*Chemical Classification*													*Possibility Of:*					*NFPA Codes*			
Acid-Base Titrations	Poison A	Flammable Gas	Flammable Liquid	Combustable Liquid	Reacts with Water	Oxidizer	Organic Peroxide	Poison B	Corrosive Acid	Corrosive Base	Irritating or Harmful	Misc. Hazard	No Hazard	Fire	Sudden Release of Pressure	Reactive	Immediate (Acute) Health Hazard	Delayed (Chronic) Health Hazard	Fire	Health	Reactivity	Special Precautions
Sodium Hydroxide, 0.2 *M*										X							X		0	3	1	ALK
Hydrochloric Acid, 0.1 *M*								X	X								X		0	3	1	COR
Sulfuric Acid, 2 *M*								X	X								X		0	3	1	COR
Acetic Acid, 0.1 *M*									X								X		2	3	0	COR
Citric Acid, 0.1 *M*									X			X							1	2	0	COR
Phenolphtalein												X							3	2	0	

I: BACKGROUND

A *titration* is a procedure commonly performed in almost any chemistry lab. The titration is the process of determining the specific amount of a standard solution required to react with a certain amount of unknown. The standard solution is one whose concentration is already accurately known. A buret[1] is commonly used to deliver the standard solution, because it measures volume dispensed. The solution used in the buret is called the *titrant*, and the solution of the unknown is called the *analyte*. A titration can be performed with any chemical reaction in solution, but it is most commonly performed with acids and bases. A typical situation would be where a standard solution of base is titrated with an unknown acid and the concentration of acid is determined, or vice versa.

The key to any titration is *stoichiometry*. For this reason concentrations of both titrant and analyte are usually expressed in molarity.

$$M = \text{moles/L} \tag{1}$$

Then, using molar ratios, the moles of the unknown can be calculated.

$$\begin{aligned} \text{moles analyte} = (&\text{mol analyte/mol titrant}) \cdot \text{volume of titrant (L)} \\ &\cdot \text{concentration of titrant } (M) \end{aligned} \tag{2}$$

During the titration, we must stop right at the point where stoichiometric amounts of acid and base react. This is called the *equivalence point* or *stoichiometric point*.

The problem with acids and bases is that nothing obvious happens at the equivalence point to tell you to stop the titration. An *indicator* is used to solve this problem. The *indicator* is a molecule that changes color depending on the acidity of the solution. Near the equivalence point, the acidity will change very rapidly. When the indictor changes color, this is called the *endpoint* (because you end the titration then). We will use an indicator called **phenolphthalein** in this lab. Phenolphthalein is colorless in acidic solution and pink in basic solution.

Dilutions

When carrying out a titration, there is frequently a small range of concentrations that can be titrated given the concentration of the titrant. If the analyte solution is more concentrated than that, it must be diluted. The following equation can be used when trying to calculate concentrations of solutions that have been diluted.

$$M_1V_1 = M_2V_2 \tag{3}$$

M_1 is the molar concentration of the solution with volume, V_1, and M_2 is the molar concentration of the solution with volume, V_2. Note that this equation can only be used in dilutions using pure solvent. A variation of this expression can be used when two solutions of different strength are involved. This is the expression below, where n_1 is the number of moles of Solution 1 and n_2 is the number of moles of Solution 2.

$$n_2M_1V_1 = n_1M_2V_2 \tag{4}$$

[1] http://www.youtube.com/watch?v=_VsKF_NCE3I

Many times a *dilution factor* is used to express how much a sample is diluted by. A sample that is diluted by a factor of 2 contains 1 part concentrated solution for every 1 part solvent. In other words, the final solution is twice the volume of the inital, concentrated solution, and its concentration is half that of the initial, concentrated solution. This is also expressed as a 1:2 dilution. A sample that is diluted by a factor of 10 contains 1 part concentrated solution for every 9 parts solvent. Likewise, this is known as a 1:10 dilution.

Overview of Lab

You will be carrying out a titration of four different acids—two strong acids and two weak acids. A strong acid is also a strong electrolyte. It dissociates completely into a proton (H^+) and its conjugate base when dissolved in water. A weak acid in contrast is a weak electrolyte and dissociates only partially in water.

Each pair will titrate known concentrations of hydrochloric acid (HCl), acetic acid (CH_3COOH), and citric acid ($H_3C_6H_5O_7$). The equivalence point will be determined using phenolphthalein, an indicator dye. After the titrations are complete, students will calculate the experimentally determined concentration of each. If your calculated concentrations are very different from the known concentration, your instructor may ask you to repeat the titration. Once a group has mastered the titration of the acids with known concentrations and its associated calculations, each pair will be given a modestly concentrated solution of sulfuric acid. After carrying out three dilutions of your sample, you will follow your procedures for the known samples, and you will calculate the concentration of the diluted samples. Depending on the concentration of your initial solution, one or two of your dilutions may not be able to be titrated using the 0.2 *M* NaOH.

Before you begin the lab, you should *estimate* how much 0.2 *M* NaOH will be needed to titrate the 0.1 *M* acid solutions (note that citric acid is triprotic). Begin by writing a balanced equation for the chemical reactions between each acid and NaOH.

II: EXPERIMENTAL PROCEDURE

Part A: Preparation

1 Label a 400-mL beaker as “Waste”.

2 Label a 100-mL beaker as “NaOH”. Take the beaker to the hood and pour about 50 mL of 0.2 *M* NaOH into it. Write down the exact concentration of the titrant as marked on the container.

3 Set up the titration equipment as demonstrated by your lab instructor. Put a white paper towel on the base of the ring stand. This will help you see the color changes more easily.

4 Close the stopcock and use a plastic pipet to pour 1 to 2 mL of NaOH into the buret. Open the stopcock and allow most of the NaOH to drain into the waste beaker.

5 Repeat the rinse. Add NaOH and allow most of it to drain out.

6 Carefully pour NaOH into the buret until it is mostly full. (Somewhat below the 0 mL mark, but above the 5 mL mark.)

Part B: Titrations Using Known Solutions

Follow the same procedure for 0.1 *M* HCl, 0.1 *M* acetic acid, and 0.1 *M* citric acid.

1 Prepare the acid for titration.

- **a.** Label a clean, dry 50-mL beaker labeled with the name of the acid.
- **b.** Take the beaker to the hood and pour about 20 mL of acid into the beaker.
- **c.** Get a clean, dry 125-mL Erlenmeyer flask. A beaker can be used if a flask is not available.
- **d.** Measure 20.0 mL of distilled water with a graduated cylinder. Pour it in to the flask.
- **e.** Use a pipette to measure 10.0 mL of acid. Add the acid to the flask.
- **f.** Use pH paper to estimate the pH of the solution. (Use a glass rod or a pipet tip to wet the pH paper. Do not insert the pH paper into the flask.)
- **g.** Add 2 drops of phenolphthalein to the flask, and mix it by swirling gently.
- **h.** Record the color of the solution.

2 Perform the titration.

- **a.** Record the initial volume of NaOH in the buret. Slowly add NaOH to the acid solution. Swirl the flask as the base is added to ensure complete mixing.
- **b.** Stop periodically to check the color of the solution.
- **c.** Record the buret volume *when the color of the acid solution first starts to change color* and the volume *when the color persists after swirling.*
- **d.** *Stop* the titration when the color of the acid solution is no longer changing. Record the final volume of NaOH.

3 Change acids.

- **a.** Pour the acid mixture into the waste container. Rinse the flask and the graduated pipette with distilled water in the waste container.
- **b.** Wipe the tip of the buret to avoid contamination, and then refill the buret before starting with a new sample. If you have to add more NaOH during a titration, ask your lab instructor for help.
- **c.** Follow the steps in Parts A and B with another acid.

4 Once you've shown your calculations to your lab instructor, you may move on to Part C.

Part C: Titrations Using an Unknown Solution

1 Each pair will be assigned a concentrated acid to dilute and titrate.

2 Dilute the acid sample 1:2, 1:5, and 1:10 to create 20.0 mL of each dilution. Use a pipet and a volumetric flask if available, or a pipet and a graduated cylinder with an appropriate volume, to prepare these diluted samples. (See the Background section for information about dilution ratios.)

3 Repeat the procedure in Part B for the diluted samples.

4 Calculate the concentration of your samples based on your titrations for those samples you were able to titrate. Using your dilution ratios, calculate the initial concentration of your samples. Show your calculations to your instructor before you leave.

: Clean Up

When you have titrated all of your acids, clean up.

1 Pour all remaining NaOH into the waste container.

2 Rinse the buret with distilled water and return it. Rinse the pipette.

3 Pour the last acid into the waste container. Clean the flask.

4 Empty the waste container into the hazardous waste bottle.