Experiment

INTRODUCTION TO ACIDS, BASES AND TITRATION

The CCLI Initiative

Computers in chemistry Laboratory Instruction

LEARNING OBJECTIVES

The objectives of this experiment are to . . .

- introduce the nature of acids and bases.
- introduce acid-base indicators, e.g., litmus, wide range indicator papers and specific titration indicators.
- Introduce various acid/base reactions
- introduce titration as a means of determining the amount of an acid or base present.

BACKGROUND

An acid was originally defined by Svante Ahrennius as a substance which furnishes hydrogen ion in solution, and a base as a substance which furnishes hydroxide ion in solution. Bronsted and Lowry further defined an acid as a proton donor and a base as a proton acceptor.

Indicators

We determine whether a substance is an acid or base by using "indicators." An indicator is an organic compound which can either accept or donate a proton, depending upon the hydrogen ion concentration of the solution it is placed in. Indicators have different colors depending on the hydrogen ion concentration. For example, litmus, a naturally occurring dye with this capability, is red in acid and blue in base. Other indicators have other colors. In addition, different indicators will change color at different hydrogen ion concentrations, or acidities. We will also utilize buffer solutions, which are solutions which have a specific acidity and tend to resist a change in that acidity.

Titration

Titration is a process in which a solution of known concentration is quantitatively added to a known volume of a solution of unknown concentration in order to determine its concentration. It requires the use of a buret, which is a glass tube of uniform bore with accurate calibrations capable of measuring any dispensed volume to a precision of 0.02 ml. The known volume of solution of unknown concentration to be titrated is known as the "analyte," and the solution in the buret of known concentration is termed the "titrant." The titration takes place by incrementally adding the titrant to the analyte solution.

As the titration proceeds, it is necessary to have some mechanism to identify when the proper amount of titrant has been added. Stoichiometrically, this occurs when chemically equivalent amounts of titrant have been added to the analyte. Macroscopically, this point is not generally observable, and it is necessary to measure it by some artificial means. In an acid-base titration such as we are doing here, there is a change in the acidity of the solution, and monitoring this change can serve the purpose. In practice, several methods

are available for determining this change in acidity. One is the use of indicators, as discussed above. The point in the titration at which the indicator changes from the acid color to the base color (titrating with a base) is defined as the "end point," and signals the end of the titration.

Another method of determining when the correct amount of titrant has been added is the use of a pH meter. pH is defined as the negative logarithm of the hydrogen ion concentration,

$$pH = - \log [H^+]$$

and ranges from 1 for very acid solutions ($[H^+] = 10^{-1}$), to 14 for very basic solutions

($[H^+] = 10^{-14}$). A pH meter measures the pH of the solution as the titration proceeds. We will use the ***MicroLAB*** interface and computer with a pH probe to measure the pH, and the system will graph the progress of the titration on the screen as the titrant is added

In this experiment, our titrant is sodium hydroxide, a strong base. We will be titrating acetic acid, a weak acid that only ionizes to about 2% in solution. We will then examine the titration curve as to its general shape, the initial and final pH values, and the pH at the equivalence point.

We will first explore the interaction of different acid concentrations with different indicators, then we will explore the interaction of different acids with different substances, then we will investigate the nature of the change in acidity of an acid by the incremental addition of a base.

SAFETY PRECAUTIONS

SAFETY GOGGLES MUST BE WORN AT ALL TIMES DURING THIS EXPERIMENT

Hydrochloric Acid solution:	Toxic by ingestion and inhalation, strong irritant to eyes and skin.
Nitric Acid solution:	Very dangerous to eyes and skin. Severe tissue damage upon ingestion.
Sulfuric Acid solution:	Severely corrosive to eyes, skin and other tissue. Toxic, strong skin irritant. Powerful dehydrator causing blistering of the skin.
Acetic Acid solution:	Corrosive liquid, skin burns are possible, very dangerous to eyes.
Sodium Hydroxide solution:	Corrosive liquid, skin burns are possible, very dangerous to eyes.

The other chemicals are innocuous; however you should keep all chemicals away from eyes and mouth, wash hands after use and before leaving the laboratory, and use prudent laboratory practices at all times.

BEFORE PERFORMING THIS EXPERIMENT . . .

...you will need a ***MicroLAB*** program capable of timing the collection of 5 ml titrant at about 2 - 4 seconds per drop. Your program should also be designed to collect pH data in relation to time and load this information into a spreadsheet. Use the ***pH.Time.exp*** selected from the **Titrations** tab.

EXPERIMENTAL PROCEDURE

Acidity and Indicators: Determining the pH Acid, Transition and Base Range of Indicators

1. Add about 1 ml (1 plastic dropper full) of each buffer solution, 2 through 12, in separate wells of the well plate.

2. Dip a stirring rod into each buffer and touch it to a short piece of Alkacid paper, then match the color of the Alkacid paper against the chart on the side of the tube.

3. Under each buffer value on the report sheet, indicate whether the color you observed on the Alkacid paper lighter or darker than the color on the chart.

4. Add one drop of methyl orange indicator to each well, stir with a clean toothpick and note the pH **Acid, Transition** and **Base Range** over which the indicator makes its color transition. Indicate this on the buffer chart on the report sheet.

5. Empty the well plate into a large beaker, rinse and wipe dry, and refill with one ml each of buffer.

6. Repeat step four using bromothymol blue, then, then phenolphthalein, noting the **Acid, Transition** and **Base Range** for each on the report sheet.

Acid/base reactions (using a 24-well plate)

1. Arrange six (6) small pieces each of marble chip, zinc, and iron wire in the 24 well plate as indicated in Figure 1.

2. Add 1 dropper of 6 *M* HCL, 1 dropper of 6 *M* H2SO4, and 1 dropper of 0.6 *M* H_2SO_4 to each of the first three samples of marble, zinc and iron wire as indicated in Figure 1.

3. Observe the reaction of each substance with each of the acid solutions and record your observations in the report sheet table. Pay particular attention to differences in the reactions and to differences in reaction rates.

4. Hold a lighted match close to the surface of each of the liquids that show any bubbling in the wells and observe closely any change in the nature of the flame.

5. Add 1 dropper of 6 *M* NaOH, 1 dropper of sat'd $Ca(OH)_2$, and 1 dropper of 0.1 *M* Na(OH) to each of the last three samples of marble, zinc and iron wire as indicated in Figure 1.

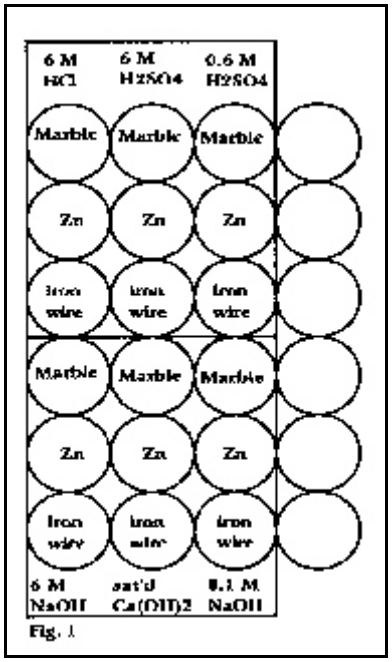

Fig. 1

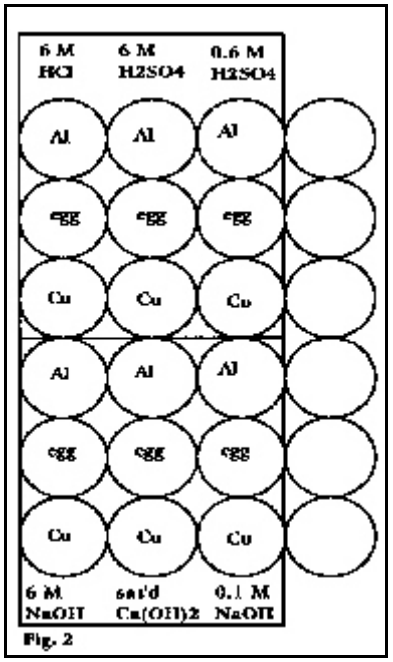

Fig. 2

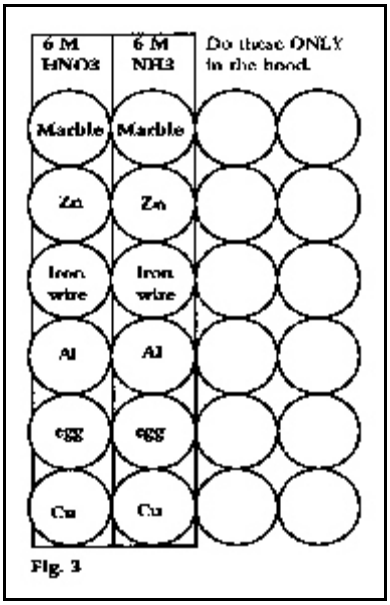

Fig. 3

6. Again, observe the reaction of each substance with each of the base solutions and record your observations in the report sheet table. Pay particular attention to differences in the reactions and to differences in reaction rates.

7. Again, hold a lighted match close to the surface of each of the liquids that show any bubbling in the wells and observe closely any change in the nature of the flame.

8. Empty the contents of the wells into the discard beaker and rinse the cells out well with tap water, then a light rinse of distilled water and wipe dry with Kim Wipes.

9. Now arrange six (6) small pieces each of aluminum, six (6) sets each of a few drops of egg albumin and six (6) small pieces of copper in the 24 well plate as indicated in Figure 2.

10. Repeat steps two through eight above and record your observations on the report sheet.

11. Place two (2) pieces each of marble chips, zinc, iron wire, aluminum, 2 sets of a few drops of egg albumin and copper in separate wells as indicated in Figure 3. Move the well plate to a fume hood and add 1 ml of 6 *M* HNO3 and 1 ml of 6 *M* NH4OH to each well and record your observations on the report sheet. Pay particular attention to differences in the reactions and to differences in reaction rates.

Caution: Do not put your face close to the well plate because a poisonous gas may be produced.

12. **Do not remove your well plate from the fume hood** but dump the solutions into the waste beaker in the hood labeled HNO3 waste, rinse the well plate into the beaker with your wash bottle, then rinse in the sink and wipe dry.

Titrations

1. Check the buret for cleanliness and proper drainage as directed by your instructor.

2. Rinse the buret three times with about 5 ml amounts of the standard NaOH solution, tipping and rotating the buret to cover all of the inside surface of the buret with each rinse. Be sure to have a stopper in the end of the buret so you don't spill NaOH on the floor or counter top.

3. Fill the buret above the 0 mark with the NaOH solution.

4. Measure exactly 25 ml portions of the 0.1 *M* $HC_2H_3O_2$ solution (the analyte solution) in a graduated cylinder and pour into each of two or three 250 ml beakers.

5. To the first beaker of analyte, add eight drops of phenolphthalein indicator, to the second add eight drops of bromothymol blue indicator. If you have time to do a third titration, use the methyl orange indicator so you can see a complete comparison. As you are doing your titration, ***be sure to note the pH at which the indicator changed color***.

6. Connect your pH probe to the pH input on the back of the ***MicroLAB*** interface and press the **Power On** button.

7. Open the ***MicroLAB Experiment program*** in the normal manner, select the experiment named ***pH.Time.exp*** from the **Titrations** tab, then press **Enter** to open the program. Click on **pH** in the **Variables View** and recalibrate your probe with the pH 4, 7 and 10 buffers supplied. Be sure to rinse the pH probe with distilled water after each buffer and before you place it in your analyte solution. Between titrations, the probe should be stored in the pH 7 buffer, then rinsed well with distilled water before inserting into your titration beaker.

8. Place a stirring bar in the 250 ml beaker and position the beaker on top of the stirrer. Place the pH probe in the analyte solution at the side of the beaker and the buret above it to drop the titrant directly into the analyte. Now remove this beaker assembly and temporarily replace it with a "waste solution" beaker to get the titration started.

9. Set the drop rate at two to four seconds per drop. When the drop rate is properly set, while one student quickly replaces the “waste beaker” with the “titration beaker,” the other student clicks the start button so that the program has started at the instant the first drop falls into the “titration beaker.” Immediately turn on the magnetic stirrer and set it to a moderate rate.

10. Be sure to give a descriptive name for each titration when asked for it in the program. ***Do not use*** **data,** as that will be overwritten with new data each time.

11. The pH vs. Time data will immediately appear in **Graph, Digital Display** and **Spreadsheet Views**.

12. While one partner continues to maintain the titrant level between 0 and 10 with the wash bottle of 0.100 *M* NaOH, ***the other partner should watch for the change of color of the indicator. Be sure to record on the report sheet the pH and the time at which the indicator changes color at the time that the color change occurs***.

13. Continue the titration until the curve has flattened out at the top. Then turn off the stopcock (turn it

perpendicular to the length of the buret) and stop the data collection by clicking on **"Stop**."

14. Repeat the entire process from step 10, using bromothymol blue in the second titration and methyl orange as the indicator for the third titration, if there is time.

    Be sure to use a different file name for each of the titrations and record this on your report sheet so you can recover them for printing and calculating later.

Discard all titration solutions into a large beaker. At the end of the experiment, this should be neutralized and flushed down the drain with large amounts of water. Be sure solids are **NOT** washed into the sink. Any solids from this experiment may be disposed of in the container so labeled.

DATA ANALYSIS

1. When the titrations are completed, reload each of your data files in turn into the ***MicroLAB*** program and perform the following analyses.

2. Click on the **Analysis** button, click on **Plot a Derivative**, use the current variables, **pH** and **Time**, click **OK**, The **Derivative** of pH vs. Time will appear on the **Graph View** in a fine line in red.

3. In the **Variables View**, scroll down to **Analysis (pH vs. Time),** and "click drag" the **f'(x)** to the **Y2** Axis. This will show the derivative in blue, and with data points.

4. Now **Right Click** on the **f'(x)** line in the **Variables View** and click on **Hide this Derivative** to remove the red derivative line.

5. **Print** your graph. **DO NOT** print your data table, as that will require too much time and paper. However, using the scroll button on the right of the **Spreadsheet View**, scroll up and down to examine how the data in the table is changing.

6. Be sure to add your names or initials and a descriptive title to each graph that you print and submit. (The title should be added to the graph ***before*** you print.)