Purpose

You will perform one of the basic types of quantitative analysis - the volumetric analysis or titration. You will determine the molar concentration of acetic acid in an acetic acid solution of unknown concentration.

Equipment

- 50mL buret
- Buret holder and stand
- Erlenmeyer flasks, 125 mL and/or 250 mL (5)
- Volumetric pipet, 10 mL
- Gtraduated cylinder, 50 mL
- Small funnel (glass or plastic)

Chemicals required

- NaOH solution (approximately 0.25 mL - check bottle for exact concentration)
- Phenolphthalein indicator solution
- Deionized water

Theoretical

Overview of the experiment

Titration takes advantage of the chemistry between substances to determine the amount of a substance present. If you know the identity but not the concentration of the components of a mixture, you can use titration to find the concentration. Titration is usually a fast analysis, but is not as precise as some other kinds of analysis.

In titration, you determine the amount of a substance present in an unknown by reacting it with a solution of another substance whose concentration *is* known. By measuring the amount of known solution required for the unknown to be completely reacted away, you can determine the amount of unknown present using a simple chemical calculation.

This experiment relies on the reaction of acetic acid ($HC_2H_3O_2$) with sodium hydroxide, a **neutralization** reaction.

$$HC_2H_3O_2(aq) + NaOH(aq) \rightarrow NaC_2H_3O_2(aq) + H_2O(l)$$

Your unknown will be a solution of acetic acid, and you will react it with sodium hydroxide until all the acid is consumed. There is one issue we should address before going on:

How do you know when all of the acetic acid is consumed?

Acetic acid and sodium hydroxide are both colorless in solution, and so are the products of the reaction. It is necessary to add an **indicator** to the mixture that will let you know when the reaction is complete. There are many different indicators for titrations, and what you would select as an indicator depends on what kind of chemistry you are using for your titration. Today, we will use **phenolphthalein** indicator - a substance that is pink in solutions that are basic, and clear in solutions that are acidic. In this titration, when there is unreacted acetic acid present, the solution will be colorless. When there is unreacted sodium hydroxide present, the solution will be pink. You want to stop the reaction when a **single drop** of sodium hydroxide causes the solution to change from colorless to a light shade of pink. (The light shade of pink must remain if the mixture is stirred!) This is called the **endpoint**.

Procedure

Setup

Set up your stand (built into your desk in Room 5402), buret, and buret holder as shown in Illustration 1, but do not yet put the sample flask below the buret.

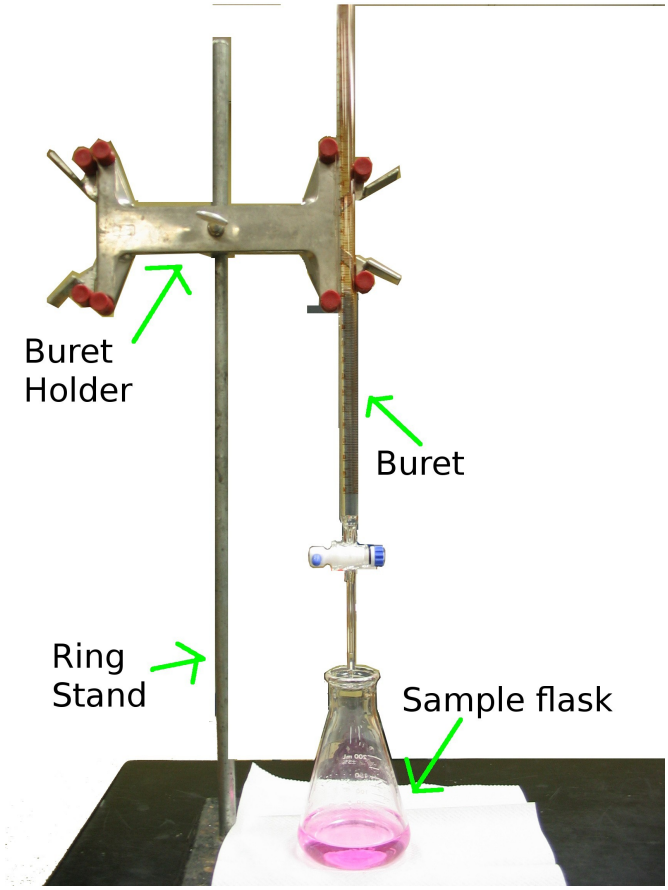

Illustration 1: A setup for titration. This setup shows what the flask would look like after titrating an acetic acid sample with sodium hydroxide.

Prepare your buret

Fill a 50 mL buret to the zero mark with sodium hydroxide solution, using the small funnel to help prevent spills. Adjust the level of liquid in the buret so the meniscus of the liquid falls exactly on the zero line by draining out extra solution using the buret's valve or adding solution with a Pasteur pipet if necessary. While filling the buret, place a waste beaker below the buret to catch any spilled liquid.

Write the concentration of the sodium hydroxide solution used on your data sheets.

Prepare your samples

Put 10.00 mL (using a 10 mL volumetric pipet) samples of your acetic acid unknown into five clean 125 mL or 250 mL Erlenmeyer flasks. Add 25 mL of deionized water and 2-3 drops of phenolphthalein indicator to each.

Analyze your samples

Using the buret, add NaOH to your first sample while swirling the flask. Add 1 mL portions of sodium hydroxide solution while swirling the flask. After each portion, check the color of the solution in the flask. If a pink color forms and persists after swirling, then stop the titration. Write the volume of sodium hydroxide necessary to produce a persistent pink color (the endpoint - see illustration 2) in the blank below.

- _____ mL of sodium hydroxide solution required.

Illustration 2 - Phenolphthalein endpoint. Lighter is better, but the color must fill the entire volume of the flask and remain after swirling.

If you're running short on Erlenmeyer flasks, you may clean the used flasks out and use them for other samples. (You do not need to save your samples after they have been analyzed.)

For the remaining four sample flasks, follow the procedure below. Read these steps completely before you start!

1. Refill your buret to the zero mark with sodium hydroxide solution, making sure to have a waste beaker underneath the buret tip to catch any spilled solution.
2. Place your sample flask underneath the buret tip, then open the buret's valve all

the way. Allow three milliliters less than the volume written in the blank for the first titration to flow into the flask. (For example, if the first sample required 25 mL for the pink color to appear, add 22 mL of sodium hydroxide in this step.)

3. Swirl the flask, and you should see any pink color disappear. If it does not disappear, you will need to prepare another sample flask and redo steps 1-3 adding less sodium hydroxide solution.
4. Now, adjust the buret to deliver sodium hydroxide drop by drop. Add sodium hydroxide drop-by-drop to the flask, while swirling the flask continuously. After each drop, check to see whether a pink color forms and persists after swirling. If a persistent pink color forms (the lighter, the better as long as it does not fade with swirling), stop the titration and record the volume of sodium hydroxide required on your data sheets Buret volumes should be recorded to the nearest 0.01 mL (example:s 23.24 mL, 13.21 mL)

Tip: If you are having trouble seeing the color change, place a sheet of white paper underneath the flask while titrating.

Repeat the above procedure (steps 1-4) for each remaining sample flask.

Cleanup

All chemicals in today's lab may be disposed of in the sink. Flush with plenty of water after disposal.

Calculations

You don't need to calculate anything for the first flask; it was just a trial run. For flasks 2, 3, 4, and 5, you will need to calculate the concentration of the acetic acid sample you used. The concentration unit for this experiment is *molarity*, which is defined as the moles of dissolved substance per liter of solution.

$$M_{HC_2H_3O_2} = \frac{\text{mol } HC_2H_3O_2}{\text{L acid solution}}$$

1. To calculate the concentration of acetic acid, you can follow this calculation procedure. Convert the volume of sodium hydroxide used to *milli*moles sodium hydroxide using the molar concentration of the sodium hydroxide as a conversion factor.
2. Convert millimoles of sodium hydroxide to millimoles acetic acid using the chemical equation (Remember, it's a 1:1 ratio!)
3. Calculate the molarity by dividing the millimoles of acetic acid and the volume of acetic acid solution originally put in each flask. (If you divide using the volume in milliliters, then the milli- prefixes will cancel out and the final answer will be in moles per liter. In other words, molarity.)

Here's a sample calculation for a case where 15.0 mL of acetic acid solution was titrated

with 31.3 mL of 0;350 M sodium hydroxide.

31.3 mL 0.350 M NaOH

15.0 mL acetic acid unknown

$HC_2H_3O_2 + NaOH \rightarrow NaC_2H_3O_2 + H_2O$

① $0.350 \text{ mol NaOH} = 1 \text{ L}$

$$31.3 \text{ mL} \times \frac{0.350 \text{ mol NaOH}}{1 \text{ L}} = 10.955 \text{ mmol NaOH}$$

② $1 \text{ mol } HC_2H_3O_2 = 1 \text{ mol NaOH}$

$$10.955 \text{ mmol NaOH} \times \frac{1 \text{ mol } HC_2H_3O_2}{1 \text{ mol NaOH}} = 10.955 \text{ mmol } HC_2H_3O_2$$

③ $$\underline{M} = \frac{10.955 \text{ mmol } HC_2H_3O_2}{15.0 \text{ mL}} = 0.730 \underline{M} \ HC_2H_3O_2$$

↑ this is the volume of the acid, not the volume of NaOH added.

Sample calculation: 15.0 mL of acetic acid solution titrated with 31.3 mL of 0.350 M sodium hydroxide

Finally, calculate the average molar concentration of samples 2, 3, 4, and 5. Use your calculator to calculate the sample standard deviation (Sx) of samples 2, 3, 4, and 5. Record the average and standard deviation along with the individual millimoles sodium hydroxide and concentration for each flask on your data sheets.

Complete the data sheets and answer all the questions, then turn in the sheets and questions to your instructor.

Datasheets and questions

Names of group members.

- Molar concentration of sodium hydroxide solution: _____ M
- Volume acetic acid solution used in each flask: ___**10.00**___ mL

Volumes and millimoles of sodium hydroxide solution required for each flask

Flask	Volume NaOH required (mL)	Millimoles NaOH required (mmol)
2		
3		
4		
5		

Calculated concentration of acetic acid in each flask

Flask	Concentration $HC_2H_3O_2$ (M)
2	
3	
4	
5	

Average concentration of acetic acid

- Average concentration of $HC_2H_3O_2$: _____M
- Standard deviation (Sx): _____M

Sample calculation

Show your complete calculations for the concentration of flask #2 in the space below.

Questions

1) Describe what would have happened in one of your titrations if you had forgotten to add phenolphthalein to the sample flask.

2) If, when preparing one of your sample flasks, you were to accidentally put in 10.00 mL of sodium hydroxide solution rather than 10.00 mL of acetic acid solution, how would you be able to discover your mistake?

3) Typical store-bought vinegar is 5% acetic acid by mass - or 0.83 M acetic acid solution. Within this experiment's margin of error, was your acetic acid solution sample the same concentration as a store-bought vinegar sample? Explain how you determined the answer to this question.

4) Based on the standard deviation you calculated, round the average concentration to the appropriate number of significant figures.

- _____ M $HC_2H_3O_2$

5) If, by mistake, you had put 12 mL of sample into your flask instead of 10.00 mL, what effect would that have on your calculated molar concentration? (Assume that you did *not* notice the mistake and thought you had placed 10.00 mL of sample into your flask.) Would the calculated acetic acid concentration be lower than the real concentration, higher than the real concentration, or the same as the real concentration of the sample?