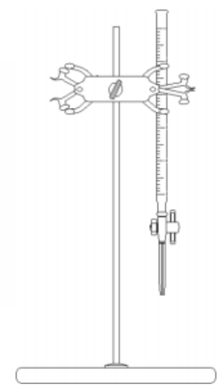
EXPERIMENT 4: Titration

INTRODUCTION

In this experiment you will be determining the volume of sodium hydroxide solution of known concentration required to neutralize a known mass of an unknown acid in solution. The technique used is called titration.

Terms with which you need to be familiar for this experiment:

TITRATION: a technique used to measure the volume of a solution of known concentration that is required to react with a measured amount (mass or volume) of an unknown substance in solution.



BURET: an instrument used to measure volume; a long graduated glass tube with a stopcock on one end. The volume measurement is made by reading the fluid level in the buret before and after the titrant, the fluid in the buret, is dispensed through the stopcock.

STANDARD SOLUTION: a solution of known concentration.

UNKNOWN: a substance or a mixture about which something is not known.

INDICATOR: a substance which is added to the reaction system in small amounts; it indicates that the reaction is complete (has reached the end point) by changing color.

EQUIVALENCE POINT: the stage in the titration at which stoichiometrically equivalent amounts of reactants have been combined and the reaction is complete

END POINT: the stage in the titration at which the indicator color change is observed, indicating that the reaction is complete.

EXPERIMENT SUMMARY:

Your unknown solid is a monoprotic acid. Let's call it HX. You will dissolve it in water, add one drop of indicator and then add just enough NaOH solution to neutralize the acid:

$$HX_{(aq)} + NaOH_{(aq)} \longrightarrow NaX_{(aq)} + H_2O_{(l)}$$

The indicator, phenolphthalein, is colorless in acidic solution but turns pink when there is an excess of base present, therefore you will know that the reaction is complete when you see the first faint hint of pink color. Because the pink color occurs when there is an excess of base present —that is, when you have passed the equivalence point—the lighter the pink color is at the end point, the better. Also, the pink should last for 30 seconds after swirling the solution. Beyond the thirty seconds it may fade due to the formation of carbonic acid, H_2CO_3, which is produced when CO_2 in the air reacts with the water in the solution:

$$CO_{2}(g) + H_2O(l) \longrightarrow H_2CO_{3}(aq)$$

The H_2CO_3 reacts to neutralize the excess NaOH that was causing the pink color, and the pink color goes away.

$$H_2CO_{3(aq)} + NaOH_{(aq)} \longrightarrow Na_2CO_{3(aq)} + H_2O_{(l)}$$

You will weigh your acid sample before you dissolve it in water and then you will measure the volume of NaOH solution required to neutralize the acid. Using these data you will calculate the ratio of the mass of acid to the volume of standard NaOH solution required to neutralize it.

PROCEDURE

A. Preparation of Acid Samples

1. Clean three 125 mL Erlenmeyer flasks. Make sure the external walls of the Erlenmeyer flasks are completely dry. Number them 1, 2, and 3 using pencil on the white spot on the flask. Throughout the weighing process you should protect the flasks from your hands with a paper towel.

2. Get an unknown acid sample from your instructor.

3. Weigh samples of approximately .15 g of your unknown into your flasks as follows: (Review Weighing Experiment for proper procedures for using the analytical balance.)

   a. Take the following items with you to the weighing room
      (1) The Unknown acid sample.
      (2) Erlenmeyer flask #1 (protect outside walls with paper towel)
      (3) Your report sheet.
      (4) A pen (not pencil).

   b. Weigh accurately (use the analytical balance) about 0.15 g of the unknown acid as follows:

      (1) Turn the analytical balance ON.

      (2) After you see a display of 0.0000 g, place Erlenmeyer flask #1 on the pan of the balance.

      (3) Close the balance windows and push the TARE bar so that the display reads 0.0000 g even though the Erlenmeyer flask is on the balance.

      (4) Carefully (you will not be able to have a "do-over" if you add too much) pour about 0.15 g of the unknown acid (without spilling) into Erlenmeyer flask #1. Close the balance windows. Read the mass, to 4 decimal places, and record it IN INK on your Report Sheet in column 1.

      (6) When you are finished, remove the Erlenmeyer flask, close the balance windows, and push the TARE bar and then the OFF button.

   c. To weigh a second sample, repeat steps a and b using Erlenmeyer flask #2 and recording the sample mass in column 3 of your Report Sheet.

   d. To weigh a third sample, repeat steps a and b using Erlenmeyer flask #3 and recording the sample mass in column 2 of your Report Sheet.

   e. Return to your desk and stopper the flasks. Ask your instructor to check and initial your weighing data. Store your flasks in your locker until the next lab meeting. Also save any leftover unknown acid.

STOP HERE

B. Preparation of Buret

1. Get a buret clamp and attach it to a ring stand.

2. Check out a 25 mL buret from the stockroom.

3. Clean the inside of the tube by rinsing it three times with tap water, but do not drain the tap water out through the stopcock (Do not contaminate the stopcock with tap water.) Then rinse

the inside of the tube with deionized water, letting the water drain through the stopcock. While draining through the stopcock, be sure to turn the stopcock completely around once. Attach the buret to the buret clamp.

4. Take a clean, dry 50 mL beaker to the reagent bench and get approximately 40 mL (do not use graduated cylinder to measure accurately!) of standard NaOH solution. Rinse the buret with the NaOH solution by first adding about 5 mL of the solution to the buret, and then rolling the tube so that the solution comes into contact with the entire inside w alls of the tube, and finally draining it through the stopcock. Repeat the process once more to be certain all rinse water from step C has been replaced by standard NaOH solution. Attach the buret to the buret clamp and place a waste beaker below the buret.

5. Fill the buret with the standard base solution. Drain a little of the titrant through the stopcock to remove any air bubbles that may be trapped there. Be sure the top of the column of the liquid is not above the top calibration mark (zero) on the buret . (Do not try to get the fluid level at the 0.00 mark.) Read the fluid level in the buret to 2 decimal places and record it in column 1 on your report sheet as the initial buret reading. (If your readings are not reported to two places after the decimal you will be asked to repeat the experiment.) There are buret-reading cards available in the lab to help you see the meniscus. Ask your instructor to verify your initial buret reading.

6. To your acid sample in flask #1 add approximately 25 mL of deionized wa ter and swirl gently to completely dissolve the acid. Then add 1 drop (and no more) of phenolphthalein indicator (which you will find in a dropper bottle at your bench) to the acid solution.

7. Place the acid solution under the buret. Be sure the buret tip is down inside the Erlenmeyer flask so that no titrant is lost. Place a piece of white paper under the flask.

8. Titrate the solution in flask #1 by adding the NaOH solution from the buret slowly while continuously swirling the solution in the flask. Dur ing the titration do not allow the fluid level in the buret to drop below the bottom calibration mark (25 mL mark). Continue adding the NaOH solution until you see a faint pink color. The piece of white paper under the Erlenmeyer flask should make this easier. Near the end point of the titration rinse down the inside walls of the Erlenmeyer flask with a little deionized water to return any solution splashed onto the flask walls. You have reached the end point of the titration if the faint pink color lasts f or at least 30 seconds after rinsing the flask walls and swirling the solution. When you have reached the end point, read the fluid level in the buret and record it in column 1 as the final buret reading.

9. Titrate the other two unknown acid samples, procee ding according to steps 5 through 8 above. Don't forget to refill your buret before each titration. Record your data in column 2 for the sample in flask #2 and in column 3 for the sample in flask #3.

10. Ask your instructor to initial your titration data, an d then proceed to the calculations to determine whether you will need to do additional titrations.

CALCULATIONS

A. Using your calculated mass and volume for sample 1, calculate the ratio of the mass (in grams) of your acid sample to the volume (in mL) of the standard base required to neutralize it. Repeat the calculation for samples 2 and 3. You may (and should) do your calculations in pencil.

B. Calculate the average of the three trials and report this answer to your instructor who will calcula te for you your percent error.

C. Report your average mass per mL of standard base (part A of calculations) to your instructor and s/he will tell you what your percent error is. If your percent error is greater than 5% you will be asked to repeat the experiment.

D. Calculate the precision of your determinations.

REPORT SHEET - EXPERIMENT 4

Name________________________________
last first

Instructor's initials ___________

		Trial 1	Trial 2	Trial 3	Trial 4 (if necessary)
DATA	Final Mass				
	Initial Mass				
CALCULATION	Mass of Acid				
DATA	Final Buret Reading				
	Initial Buret Reading				
CALCULATION	Volume base soln				
	Mass acid per volume base				

CALCULATIONS:

You may (and should) use pencil to do your calculations. Don't forget to show ALL UNITS in your setups.

1. Calculations of Mass Acid per Volume Base

   a. Trial 1:

   b. Trial 2

   c. Trial 3

   d. Trial 4 (if necessary)

2. Experimental Mass Acid per Volume Base (Average Mass Acid per Volume Base):

3. Accuracy (%Error) of Mass Acid per Volume Base ____________________________
(from instructor)

4. Precision (% Deviation) of Mass Acid per Volume Base

   a. Deviations

      Trial 1:

      Trial 2:

      Trial 3:

      Trial 4:

   b. Standard Deviation

   c. % Deviation

QUESTIONS:

For each of the following questions, give complete setups, including all units, and be sure your significant figures are correct.

1. How many grams acid would be required by 25.00 mL of the base? (Use your experimental mass acid per volume base to calculate this).

2. How many grams acid would be required by 40.00 mL of the base? (Use your experimental mass acid per volume base to calculate this).

3. A student weighed an unknown acid sample by difference and found its mass to be 0.5012 g. The uncertainty in each reading made on the balance he used was ±0.0002 g. The sample required 27.44 mL of the standard NaOH solution to neutralize it. The uncertainty in each buret reading was ±0.03 mL.

   a. Calculate the percent uncertainty in the mass of the sample.

   b. Calculate the percent uncertainty in the volume of standard base.

   c. Calculate the ratio of grams acid to mL NaOH solution.

   d. Calculate the percent uncertainty in the ratio of grams acid to mL NaOH solution.

   e. Calculate the uncertainty (absolute uncertainty) in the ratio of grams acid to mL NaOH solution

4. What are three possible sources of error in this experiment (do not include unavoidable errors in reading the buret or balance)?

   a. ____

   ____

   b. ____

   ____

   c. ____

   ____