Experiment: 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 will be titration.

Terms you will need to be familiar with in order to understand a discussion of titration are:

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 graduated glass tube about 40 cm long 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.

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:

$$HX_{(aq)} + NaOH_{(aq)} \longrightarrow H_2O_{(l)} + NaX_{(aq)}$$
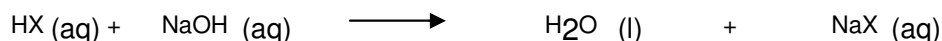
The indicator, phenolphthalein, is colorless in acidic solution but turns pink when there is an excess of base present, therefore you will know that the reaction is complete when you see the first faint hint of pink color. Because the pink color occurs when there is an excess of base present, the lighter the pink color is at the end point, the better. Also, the pink should last for 30 seconds after swirling the solution. After that time it may fade due to the presence of carbonic acid, H_2CO_3, in the solution. This is formed when CO_2 in the air reacts with water:

$$CO_2{}_{(g)} + H_2O_{(l)} \longrightarrow H_2CO_3{}_{(aq)}$$
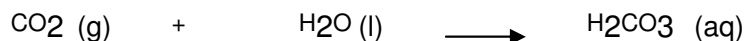
The H_2CO_3 reacts to neutralize the NaOH that was causing the pink color, and the pink color goes away.

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 mass of acid that would require 20.00 ml of standard base solution.

PROCEDURE

A. Obtain an unknown acid sample from your instructor. 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.

A reminder: Throughout the weighing process you should protect the Erlenmeyer flasks from your hands with a paper towel.

B. Weigh by difference three samples of unknown acid as follows:

1) Take the items listed below with you to the weighing room:
   - The Unknown acid sample.
   - A clean 125 ml Erlenmeyer flask labeled #1. **Make sure the external wall of the Erlenmeyer flask is completely dry.**
   - Your report sheet.
   - A pen (not pencil).

2) You need to weigh **accurately** about 0.15 g of the Unknown acid as follows:
   a) Turn the analytical balance **ON**.

   b) After you see a display of 0.0000 g, place the Erlenmeyer flask **labeled #1** on the pan of the analytical balance.

   c) Close the balance windows and touch the **TARE** button. A " 0.0000 g " will be displayed regardless of the mass of the Erlenmeyer flask.

   d)) Carefully pour about 0.15 g of the unknown acid (without spilling) into Erlenmeyer flask #1. Close the balance glass case windows. Record this reading to the one-tenth of a milligram on the report sheet in column 1.

   e) When you are finished, remove the Erlenmeyer flask, close the balance windows, touch the **TARE** bar, and press the **OFF** button.

3) To dispense a **second** sample into another Erlenmeyer flask **labeled #2**, repeat steps 1 and 2 given above. Record your data in column 2 on the report sheet.

4) To dispense a **third** sample into another Erlenmeyer flask **labeled #3**, repeat steps 1 and 2 given above. Record your data in column 3 on the report sheet.

5) Return to your desk and place the samples out of the way while you prepare the buret for titration. Ask your instructor to check and initial your weighing data before you begin to titrate.

C. Check out a buret from the stockroom. Clean the tube by first rinsing it three times with tap water. (Do not contaminate the stopcock with tap water.) Then rinse the tube with distilled water, letting the water drain through the stopcock. While draining through the stopcock, be sure to turn the stopcock completely around once. Obtain a buret clamp from the drawer under the hood, attach it to a ring stand, and then attach your buret to the clamp.

D. Take a clean, **dry** 50 or 100 ml beaker to the reagent bench and get approximately **40 ml** 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 wall of the tube, and finally draining it through the stopcock. Repeat the rinsing process once more to be certain all rinse water from step C has been replaced by standard NaOH solution.   -2-

E.   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. Read the fluid level in the buret to the nearest 0.03 ml 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. At this point you will **ask your instructor** to verify your buret reading and initial it.

F.   Prepare your acid sample in flask #1 by adding **approximately** 25 ml of distilled water to it and swirling gently to **completely** dissolve the acid. Then add 1 drop (and no more) of phenolphthalein indicator (on the shelf above your bench) to the acid solution.

G.   Titrate the solution in flask #1 by adding the NaOH solution from the buret gently and continuously swirling the unknown acid solution. Be sure the buret tip is down inside the Erlenmeyer flask so that no titrant is lost. During 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. It may help you to see this color when it first appears if you put a piece of white paper under the Erlenmeyer flask. Near the end point of the titration rinse down the inside walls of the Erlenmeyer flask with a little distilled water to return any splashed titrant of acid solution. You have reached the end point of the titration if the faint pink color lasts for at least 30 seconds after 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.

H.   Refill the buret. Read the fluid level in the buret and record this as the *initial* buret reading in column 2 on your report sheet. Prepare the acid sample in flask #2 for titration as you did sample #1 in step F above. Then titrate the solution with the standard NaOH solution as you did sample 1 in step G. Don't forget to add indicator to your acid solution before titrating and don't forget to make and record the final buret reading in column 2.

I.   Prepare and titrate the sample in flask #3 as you did the first two acid samples, recording titration data in column 3 on your report sheet. *Ask your instructor to approve and initial your buret readings.*

CALCULATIONS
Write your answers on your report sheet:

A.   Using your calculated mass and volume for sample 1, calculate the mass of your acid that would be required to neutralize 20.00 ml of the standard base. Show this calculation as your sample calculation on the report sheet. Repeat the calculation for samples 2 and 3.

B.   Calculate the average of these three answers.

C.   Report you average mass that would require 20.00 ml of standard base (part A of calculations) to your instructor and he/she 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 as indicated on your report sheet.   (See Measurements if you need help.)

NAME_____
        Last   First
Instructor's initial_____

A) Acid Samples

	1	2	3
Mass of Acid	g	g	g

Data Approval _____

B) Standard Base

	1	2	3
Initial Buret Reading	ml verification_____	ml	ml
Final Buret Reading	ml	ml	ml
Calculated Volume of Base	ml	ml	ml

Data Approval _____

CALCULATIONS:
A) Mass of acid sample that would require 20.00 ml of standard base:

	Sample #1	Sample #2	Sample #3
Setup:			
Answer:	g/20ml	g/20ml	g/20ml

B) Average mass of acid that would require 20.00 ml of standard base.
Setup:

_____ **g/20ml**

Accuracy (from Instructor):

_____ **% error**

C) Precision:

	Sample #1	Sample #2	Sample #3
Deviation Setup:			

Standard Deviation:
Setup:

_____ **g/20 ml**

Percent Deviation:
Setup:

_____ **%**

Use this page, if you need to repeat the experiment
A) Acid Samples

	4	5	6
Mass of Acid	g	g	g

<div align="center">Data Approval _____</div>

B) Standard Base

	4	5	6
Initial Buret Reading	ml verification_____	ml	ml
Final Buret Reading	ml	ml	ml
Calculated Volume of Base	ml	ml	ml

<div align="center">Data Approval _____</div>

CALCULATIONS:
A) Mass of acid sample that would require 20.00 ml of standard base:

	Sample #4	Sample #5	Sample #6
Setup:			
Answer:	g/20ml	g/20ml	g/20ml

B) Average mass of acid that would require 20.00 ml of standard base.
Setup:

<div align="right">_____ g/20ml</div>

Accuracy (from Instructor):

<div align="right">_____ % error</div>

C)   Precision:

	Sample #4	Sample #5	Sample #6
Deviation Setup:			
Answer:	= g/20ml	= g/20ml	= g/20ml

Standard Deviation:

Setup:

<div align="right">± _____g/20ml</div>

Percent deviation:
Setup:

<div align="right">_____ %</div>

QUESTIONS:

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

1.   A student weighed an unknown acid sample by difference and found its mass to be 0.3064g. The uncertainty in each reading made on the balance he used was $\pm$ 0.0003 g. Calculate the **percent uncertainty** in the mass of the sample.
Setup:

Answer_____%

2.   The unknown acid sample (refer to question #1) required 25.86 ml of standard base solution to neutralize it. The uncertainty in each buret reading was $\pm$ 0.02 ml. Calculate the **percent uncertainty** in the volume of standard base.
Setup:

Answer_____%

3.   Calculate the grams of the student's unknown acid sample that would neutralize 30.00 ml of the standard solution.
Setup:

Answer_____g

4.   Calculate the **percent uncertainty** to the answer to question #3.
Setup:

Answer_____%

5.   What would be the **absolute uncertainty** in the mass of the acid that is required to neutralize 30 ml base?
Setup:

Answer_____g

6.   On the basis of your answer to question #5, to how many significant figures should the answer to question #3(the grams acid to neutralize 30.00 ml of base) be reported? (No setup is required here.)

Answer_____

B.   What are two possible sources of error in a titration besides the unavoidable errors in reading a buret or balance?

1.   _____

_____

2.   _____

_____