Experiment 1

Calibration of Apparatus and Statistical Analysis

Introduction:

Before the analysis, the analyst must calibrate the apparatus used for measurements. Most fundamentals are measurements of mass and volume. This experiment requires a large number of measurements of mass and volume, a precision of 0.2 % and an accuracy of 0.3% can be expected from the better volumetric and gravimetric procedures. This lab is designed to help you become familiar with the analytical (electronic) balance, the volumetric pipette, and the burette, their use, and sources of errors. These three devices are the principal means you will be using all semester to determine the quantities of substances present in your laboratory experiments.

Analytical Balance:

Analytical balances are capable of weighing accurately to ± 0.1 mg. To use the analytical balance effectively, the student must have a thorough knowledge of the operation of the balance (with the help of your TA). The weighing measurements can be obtained either by difference or by direct measurements using a weighing boat.

Experimental Procedure

Part A

Prepare a table in your notebook using Table 1 as a template.

1. Determine the mass of a clean, dry weighing bottle (without its lid) to within ± 0.1 mg. (4-replicates)
2. Determine the mass of the weighing bottle lid separately to within ± 0.1 mg. (4-rplicates)
3. Determine the total mass of the weighing bottle plus the lid to within ± 0.1 mg.(4-replicates)
4. Compare this result with the sum of the separate weights of the bottle and the lid to see how close the weights are
5. Discuss the magnitudes of your standard deviations.

Table 1.

Object	Mass ± 0,1 mg	Mean	Std.Dev	RSD
Bottle	13.259	13.259	8.16×10^{-4}	6.154×10^{-5}
	13.259			
	13.258			
	13.260			
Bottle Lid	14.768	14.76925	9.57×10^{-4}	6.4797×10^{-5}
	14.769			
	14.770			
	14.770			
Bottle & Lid	28.028	28.02775	1.258×10^{-3}	4.488×10^{-5}
	28.028			
	28.029			
	28.026			
Bottle& Lid(calculated)	28.027	28.02825	$1,258 \times 10^{-3}$	4.488×10^{-5}
	28.028			
	28.028			
	28.03			

Volumetric Apparatus:

Volumes that enter into stoichiometric calculations (exact volumes needed), must be delivered by using appropriate glass wares (equipments) like burettes, volumetric flasks and pipettes. Graduated cylinders and beakers can not be used for such purpose.

Experimental Procedure Part

B1 pipette

Note 1. The tip must be below the surface of the liquid while withdrawing an aliquot, use a rubber bulb to draw the liquid.

Note 2. Never blow out the pipette; it may retain some liquid, just touch the tip to the wall of the receiving flask.

Note 3. Prepare a table in your notebook using Table 2 as a template and use it to fill your data.

Fill a large beaker (500 ml) with distilled water, let the water reach the thermal equilibrium (10 minutes), record the temperature of the lab and the water

1. Weigh a clean dry beaker
2. Using a pipette, transfer 25 ml of the equilibrated distilled water to the clean beaker.
3. Reweigh the beaker (with distilled water).
4. Repeat the procedure 3-times, calibrate the pipette.

Table #2: pipette calibration								
#	g of beaker	Mass of beaker and H_2O	mass H_2O	Density of water	Vol water	mean	ST D	RSD %
1	56.618	66.470	9.852	0.996226	0.1011	0.10143	4.103×10^{-4}	4.104×10^{-3}
2	56.618	66.391	9.773		0.1019			
3	56.615	66.448	9.833		0.1013			

Experimental Procedure Part

B2 Burette

Note 1. The reading of the burette must be taken at the bottom of the meniscus.

Note 2. Watch for air entering the stopcock (remove any bubbles), and use the left hand to operate the stopcock.

Note 3. Wipe the outside of the burette after filling. When delivering the liquid, no residues of the liquid must be (stuck) on the inside wall of the burette.

Note 4. . Prepare a table in your notebook using Table 3 as a template and use it to fill your data.

1. Fill the burette with the equilibrated distilled water, read the volume accurately to 2 decimal places(it is not necessary to adjust the volume to 0.00 reading)
2. Check for any leakage by allowing the burette to stand for 5 minutes. (Re-check your initial reading).
3. Weigh a clean dry beaker.
4. Transfer approximately 10 ml of water (take **accurate reading**) to the beaker.
5. Reweigh the beaker to determine the mass of water.
6. Repeat the procedure for the same volume (10 ml) and for 20, 30 and 40 ml so that for each volume you have 3-replicates.
7. Calculate that mass of the water delivered and the actual volume of water delivered (use table 3 for temperature correction).

Table #3: Burette calibration

#	g of beaker	Mass of beaker and H_2O	mass H_2O	Density of water	Vol water	mean	STD	RSD%
1	56.621	66.494	9.873	0.996226	0.1009	0.100383	0.000493	7.8997 *10-3
2	56.6187	66.632	10.013		0.09947			
3	56.618	66.503	9.885		0.10078			

Table 4. Volume occupied by 1gm of water weighed in air against stainless steel weight

Temperature, T, °C	Volume ,ml	
	At T	Corrected to 20 °C
10	1.0013	1.0016
11	1.0014	1.0016
12	1.0015	1.0017
13	1.0016	1.0018
14	1.0018	1.0019
15	1.0019	1.0020
16	1.0021	1.0022
17	1.0022	1.0023
18	1.0024	1.0025
19	1.0026	1.0026
20	1.0028	1.0028
21	1.0030	1.0030
22	1.0033	1.0032
23	1.0035	1.0034
24	1.0037	1.0036
25	1.0040	1.0037
26	1.0043	1.0041
27	1.0045	1.0043
28	1.0048	1.0046
29	1.0051	1.0048

Density(water) = 1.000131 + 1.233 × 10^{-5} T – 5.421 × 10^{-6} T^2

Table

POSTLAB QUESTIONS: (Experiment 1)

1. Define a) weighing by difference b) absolute weight.
2. Volumetric glassware should not be dried in an oven, explain why?
3. Explain why it is not correct (accurately) for weighing a substance that is still hot or warm (say > 35 °C) compared to the room temperature (20 °C).
4. A 25mL pipette was found to deliver 24.876g of H2O when calibrated against stainless steel weight at 25°C. use the data in table 4 to calculate the volume delivered by the pipette at this temperature, repeat the calculations at 90°C.
5. Suggest some sources of errors in this experiment.

Experiment 2

Statistical Handling of Data

Introduction

It is impossible to carry a chemical analysis in such a way that the results are totally free of errors. The errors which affect an experimental result can be either deteriminant errors (systematic) or indeterminant (random).

Determinant errors are errors which can be avoided or determined. These cause the mean value x to differ from the accepted one xi and are due to instrumental, method, or personal error.

Indeterminant errors which can not be controlled and result from different variables. It causes data to be scattered symmetrically around the mean value.

Unfortunately, there is no simple method for determining the reliability of data with absolute certainly. Statistical tests can be applied to the data which can help us to decide on its reliability to a certain extent.

In this experiment, statistical methods are applied on the data of acid-base titration analysis.

Procedure:

A) Titration of Nitric Acid.

1. Measure 10.00 mL of 0.10 M HNO3 acid into a 250 mL conical flask.
2. Add 10 mL of distilled water and three drops of phenolphthalein.
3. Titrate with 0.100 M NaOH until the pink color appears.
4. Repeat four times.

B) Titration of blank analysis in the absence of sample.

1. Measure 10.00 mL of distilled water and three drops of phenolphthalein.
2. Titrate with 0.10 M NaOH until the color becomes pink. Only one titration is required.

❖ The result is then applied a correction to the actual measurements Data

❖ Fill the data in the following table:

#	Volume of NaOH V_s (mL)	Volume of blank V_b (mL)	Actual volume of NaOH $V_s - V_b$ (mL)
1			
2			
3			
4			

Calculations

1. Apply the Q-test to decide whether to retain or to reject the readings:

$$Q_{calculated} = \frac{|x_q - x_n|}{W}$$

where Xq is the questionable result (the result that you feel is out of place), Xn is the nearest neighbour to Xq after the result had be organized in ascending or descending order and W is the spread between the minimum and the maximum values. The value of Q-calculated may be compared with the rejection depending on number of observations and degree of confidence.

2. Get the mean value which is given by:

$$\bar{x} = \frac{\sum_{i=1}^{N} x_i}{N}$$

where N is the number of trials attempted.

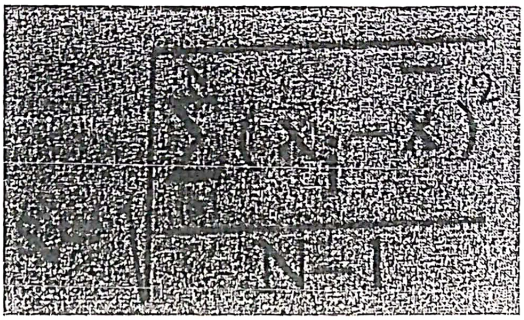
3. Obtain the median (middle value) of the results after arranging the data in order.
4. Obtain the average deviation from the mean:

$$\bar{d} = \frac{\sum_{i=1}^{N} |x_i - \bar{x}|}{N}$$

5. Relative average deviation from the mean:

$$\text{Relative average deviation} = \frac{\bar{d}}{\bar{x}} \times 100$$

6. Standard deviation (s):



7. Use the following table to decide whether the questionable is retained or rejected!.

No. of Observations	Q (90% confidence level)
4	0.76
5	0.64
6	0.56
7	0.51
8	0.47
9	0.44
10	0.41

- if Qcalculated > Q table, the value in question can be rejected with 90% confidence.

Questions:

1. Calculate the mean, standard deviation and relative standard deviation for the following set of data:
   10.1234, 9.9872, 10.2327, 10.2296, 10.1983, 10.2106

2. For the data set in question 2, determine whether if the second value of 9.9872 can be rejected or not using the Q test.

Experiment 3

Gravimetric Method: Determination of Sulfate ion (SO_4^{2-})

Introduction The concentration of sulfate ion will be determined gravimetrically by precipitation with barium chloride. When a solution containing sulfate ion is mixed with one containing barium ion, the following reaction is happened:

$$Ba^{2+}_{(aq)} + SO_4^{2-}{}_{(aq)} \rightarrow BaSO_{4(s)}$$

This reaction can be used for the gravimetric determination of sulfate or, less commonly, for barium. Barium sulfate usually precipitates as very fine particles. The high surface area of the particles facilitates contamination by adsorption. Larger particles can be obtained by heating the precipitate in the presence of its mother liquor. During this digestion process, recrystallization takes place resulting in a precipitate of larger particle size. Since barium sulfate is stable in air and is non-hygroscopic, the weighing can be performed in an open crucible.

Experimental Procedure:

1. Pipet 10.0 mL of unknown sulfate solution into a 400 mL beaker, then add 4 mL of 6 M HCl, and dilute to about 200 mL with distilled water.
1. Heat the solution to boiling, and quickly with vigorous stirring add 30 mL Barium chloride solution. (You should observe the formation of the white $BaSO_4$ precipitate).
2. Digest the precipitated $BaSO_4$ at just below boiling point for half hour on small flame. Then turn the burner off and allow the precipitate to settle in the beaker for about 20 minutes.
3. Filter the hot supernatant through weighed fritted glass or Gooch crucible.
4. Test the filtrate with few drops of $BaCl_2$ to ensure complete precipitation. (This can be tested by adding a drop of $AgNO_3$ solution to a test portion of the washing collected in a test tube. Absence of turbidity indicates chloride free precipitate)
5. Wash the precipitate with cooled water, Dry the precipitate in an oven at 110°C, after weighing, calculate the % of SO_4^{2-} in the unknown.

Questions

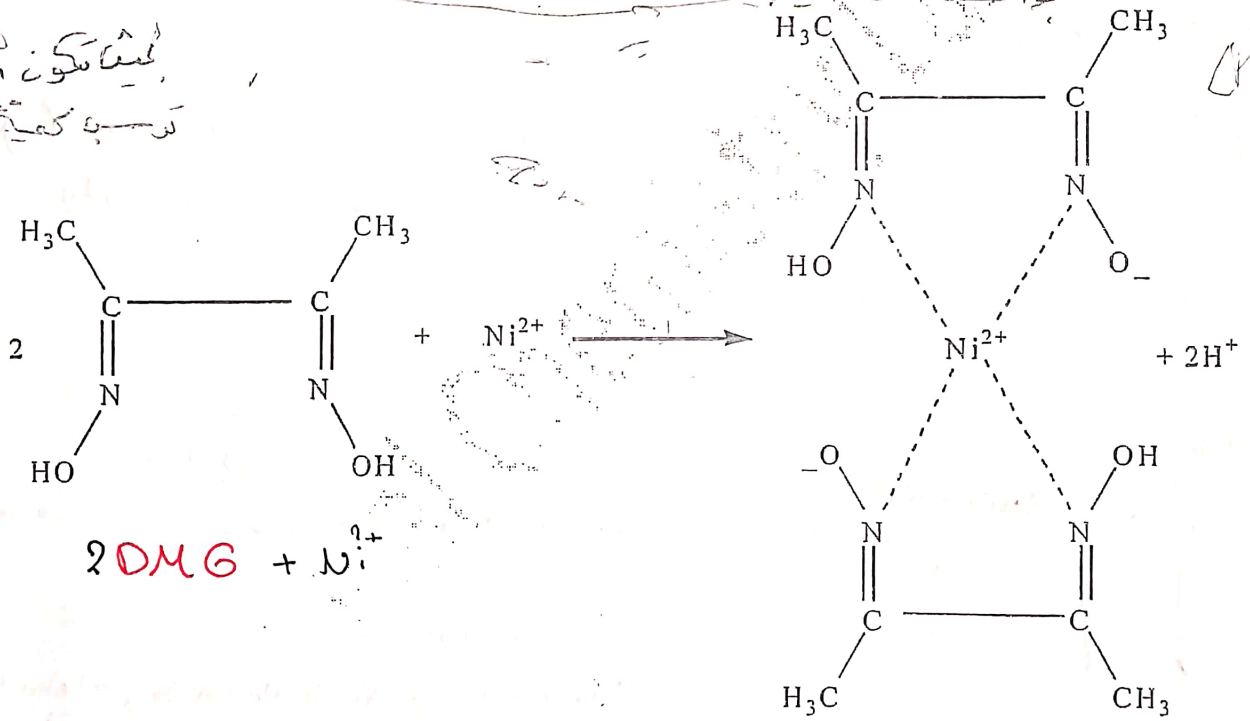
1. . From the following list, identify the interfering species for the sulfate determination method used in this experiment: Mg^{2+}, Pb^{2+}, Na^+, NO_3^-, Cl^-, PO_4^{3-}.
2. What is the importance of digestion step in precipitation?
3. The calcium in a 200.0 mL sample of natural water was determined by precipitating the cation as CaC_2O_4. The precipitate was filtered, washed, and ignited in a crucible with an empty mass of 26.6002 g. The mass of the crucible plus CaO (Mw 56.077 g/mol) was 26.7134 g. Calculate the concentration of Ca (Mw 40.078 g/mol) in the water in units of grams per 100 mL.

Experiment 4

Gravimetric Determination of Nickel in Steel

Introduction:

Many inorganic ions such as metal ions can be precipitated with certain organic reagents called "organic precipitants". Most of the organic precipitants complexes with the cation to form chelate ring, such complexes are often insoluble in water, course and bulky so that metal ions may be quantitatively precipitated. In this experiment Nickel in steel will be determined by precipitation with dimethylglyoxime in slightly alkaline solution:



This complex has the composition of $Ni(C_4H_7O_2N_2)_2$ with MW= 288.92 g/mol.

Interference from Fe(III) is eliminated by forming a soluble complex with tartrate ion.

Experimental Procedure

Note: pipette 10 ml of your unknown, add about 150 ml of water and continue the procedure as following:

1- Acidify the solution with 5 mL diluted HCl (use litmus paper for test)), heat the solution to ~ 70 °C.

2- Add 20 ml of 1% solution of dimethylglyoxime add ammonia until a slight excess exist (test by odor or litmus paper) and then add 1ml excess of ammonia.

3- Digest the precipitate at ~ 60 °C for about 30 min.

4- Cool the solution and filter through a weighed fritted glass or Gooch crucible, wash the precipitate with cooled water, followed by 10% alcohol, dry the precipitate in an oven at 110°C, after weighing ,calculate the % of Ni in steel as mg per liter.

POSTLAB QUESTIONS:

1- Name another (common) organic precipitant used for the determination of metals (such as aluminum) in aqueous solution.

2- Explain the effect(s) of adding too much of alcoholic dimethylglyoxime in this experiment?

3- What are the main advantages and disadvantages of using the organic precipitants in the determination of metal ions in aqueous solution?

4- Why ethanol is used for washing?

5- Name another (common) organic precipitant used for the determination of metals (such as aluminum) in aqueous solution.

6- Explain why the complexation of Ni^{2+} is carried out in ammonical medium?

EXPERIMENT 5

NEUTRAI IZATION TITRATION IN AQUEOUS MEDIUM

INTRODUCTION.

Neutralization titrations involve acid-base reactions. End point detection is based upon the abrupt change in pH at the equivalence point. In laboratory practice it is customary to prepare and standardize one solution of an acid and one of a base. These two solutions can then be used to analyze unknown samples of acids and bases. In this experiment a secondary solution of about 0.1 M HCl is going to be standardized against a primary standard solution of sodium carbonate. The standardized HCl solution will then be used for standardization of about. 0.1 M NaOH (secondary standard) solution. The standardized NaOH will be used for determination of phosphoric acid in commercial acid and for determination oldie equivalent weight of an unknown organic acid

EXPERIMENTAL PROCEDURE

A) Standardization of hydrochloric acid against sodium carbonate

Weigh accurately about 0.50g of previously dried primary standard sodium carbonate in a weighing bottle. Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Adjust the volume to the mark and mix well, calculate the .molarity of the Na_2CO_3 solution. Pipette 10 mL of the Na_2CO_3 solution into an Erlenmeyer flask, add 3 drops of bromophenol blue indicator and 20 ml distilled water then titrate with the HCl solution until the solution changes from blue to yellow. Repeat three times and calculate the exact molarity of the I HCl solution.

Na_2CO_3 is primery solution

blue ⟶ yellow

B) Standardization of the sodium hydroxide solution against HCl solution:

Pipette 10.0 mL of about 0.1 M NaOH solution into an Erlenmyer flask. Add 25 mL of distilled water and 3 drops of phenolphthalein indicator. Titrate with the standard HCl solution until the solution changes from pink to colorless. Repeat three times and calculate the exact molarity of the sodium hydroxide solution.

NaOH is primery solution.

pink ⟶ color less.

C) Determination of Phosphoric acid in commercial acid Phosphoric acid:

H_3PO_4 is a tribasic acid having three replicable hydrogen atoms. Neutralization of this acid leads to the production or the dihydrogen phosphate, the monohydrogen phosphate and the tribasic phosphate. The pH of the equivalence point at each stage is 4.6, 9.7 and 12.6 respectively. Thus in the titration of this acid an appropriate indicator with a transition pH range which matches the pH of the stage at the equivalence point should be selected. No satisfactory indicator is known for the third stage.

1) Titrate 10.0 mL from the provided phosphoric acid with the 0.1M NaOH solution using 3-5 drops of methyl orange or Bromophenol blue as indicator. Repeat 2 times. Calculate the concentration. of H_3PO_4 in the unknown phosphoric acid solution in grams per liter.

2) Titrate another 10.0 mL from the provided phosphoric acid with the 0.1M NaOH using 3-5 drops of phenolphthalein indicator. Repeat two times. Calculate the concentration of H_3PO_4 in the unknown phosphoric acid solution in grams per liter.

3) Compare the calculated concentration of H_3PO_4 using Bromophenol and phenolphthalein.

D) Titration of acetic acid in vinegar

Pipette 10 ml aliquot of this solution into Erlenmeyer flask, dilute with about 50 ml of distilled water. Add 3-4 drops of the phenolphthalein indicator. Titrate with 0.1M NaOH solution. Calculate the acid content (w/v)% as an acetic acid

Questions:

1. If vinegar contains 4% (w/v) of acetic acid ($K_a = 1.8 \times 10^{-5}$), calculate the pH of solution made by mixing 50 ml of this vinegar with 200 ml of distilled water.
2. What is the volume of standardized 0.1 M HCL needed to titrate 0.2010 g of Na_2CO_3?
3. What is the pH of a solution of 0.06 M of H_2CO_3, pKa1 = 6.4 and pKa2= 10.3
4. Why two indicators can be used for the determination of phosphoric acid?

EXPERIMENT 6

Determining a Solubility Product Constant

Introduction

In general, the solubility product constant, K_{sp}, is the equilibrium constant for the solubility equilibrium of a slightly soluble (or nearly insoluble) ionic compound. It equals the product of the equilibrium concentrations of the ions in the compound, each concentration raised to a power equal to the number of such ions in the formula of the compound. Lead(II) iodide is an example of a slightly soluble salt. The equilibrium in water is

$$PbI_{2(s)} \rightleftharpoons Pb^{2+}_{(aq)} + 2\,I^-_{(aq)}$$

The expression for the solubility product constant is

$$K_{sp} = [Pb^{2+}][I^-]^2$$

The equilibrium between solid $Ca(IO_3)_2$ and its ions in a saturated solution is

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2\,IO_3^-(aq)$$

If some analytical technique is used to determine the concentration of either the Ca^{2+} or IO_3^- ions in the saturated solution, the solubility of $Ca(IO_3)_2$ will be known and the solubility product constant can be calculated.

In this experiment the concentration of IO_3^- ions is determined through titration with a standardized solution of thiosulfate ion ($S_2O_3^{2-}$) in the presence of iodide ion (I^-), using starch as an indicator **near** the end of the titration. Iodate ion reacts with the iodide ions to give I_3 (triiodide ion) as the sole product containing iodine:

$$IO_3^-{}_{(aq)} + 8\,I^-_{(aq)} + 6\,H^+_{(aq)} \longrightarrow 3\,I_3^-{}_{(aq)} + 3\,H_2O_{(\ell)} \qquad (1)$$

The triiodide reacts with $S_2O_3^{2-}$ ions during the titration, according to

$$I_3^-{}_{(aq)} + 2\,S_2O_3^{2-}{}_{(aq)} \longrightarrow 3\,I^-_{(aq)} + S_4O_6^{2-}{}_{(aq)} \qquad (2)$$

$S_4O_6^{2-}$ is tetrathionate ion. Combining the two reactions gives a titration reaction of

$$IO_3^-{}_{(aq)} + 6\,S_2O_3^{2-}{}_{(aq)} + 6\,H^+_{(aq)} \longrightarrow I^-_{(aq)} + 3\,S_4O_6^{2-}{}_{(aq)} + 3\,H_2O_{(\ell)} \qquad (3)$$

Triiodide ion is a red-brown color and I^- is colorless, so no indicator is needed for most of the titration. Starch solution is added near the end of the titration (when most of the I_3^- ion has been consumed) in this titration because it reacts with I_3^- reversibly to form a more visible dark blue

color. As I_3^- is consumed in the titration, the color will fade as the titration progresses. If the starch is added too early, the I_3^- /starch compound precipitates out of solution and is slow to react.

Experimental procedure:

A) Preparation of Saturated $Ca(IO_3)_2$ Solution.

1. Using the markings on a 100-mL beaker, prepare $Ca(IO_3)_2$ by adding 50 mL of 0.2 M KIO_3 to 20 mL of 1 M $Ca(NO_3)_2$.
2. Stir vigorously with a stirring rod. A white precipitate of $Ca(IO_3)_2$ should form. Let the mixture settle for a few minutes.
3. Use gravity filtration to collect the precipitate after decanting off most of the solution.
4. Wash the precipitate with 3 small (~5 mL) portions of distilled water.
5. Place all of the wet precipitate in a 250-mL beaker and add 100.0 mL of distilled water.
6. Stir thoroughly with a stirring rod and let the mixture stand for at least 30 minutes, leaving the stirring rod in the beaker. You are attempting to make a saturated solution of $Ca(IO_3)_2$. Go on to Part B.

B) Determination of $[IO_3^-]$ and $[Ca^{2+}]$ in Saturated $Ca(IO_3)_2$ Solution.

- **Do not** add water during filtration or sampling or the molarity of IO_3^- ions will change.

1. Gravity filter the solution from part I into a clean, dry 125-mL Erlenmeyer flask. **Do not wash the precipitate on the filter paper.**
2. Rinse a 10-mL pipet with a small amount of the filtrate.
3. Pipet 10.00 mL of the filtrate into a clean 250-mL Erlenmeyer flask. Add 20.0 mL of distilled water to the flask.
4. Measure about 0.2 g of solid KI in a small beaker and dissolve in the solution from step 3 above. Add 20 drops of 2 M HCl to the flask and swirl to obtain a homogeneous redbrown solution. Begin titrating immediately.
5. Titrate with standardized $S_2O_3^{2-}$ until the solution becomes light orange to yellow. Add 3–4 drops of 2% starch solution. Continue titrating until the blue solution just turns colorless. Record the final volume of $S_2O_3^{2-}$ solution to 0.01 mL accuracy.
6. Titrate two more 10.00 mL samples.

Questions

1. Calculate the volume of 0.04000 M $S_2O_3^{2-}$ solution that would be needed to titrate 10.00 mL of 0.01000 M IO_3^-.

2. When 5.0 mL of 0.012M $Pb(NO_3)_2$ are mixed with 5.0 mL of 0.030M KI, a yellow precipitate of PbI_2 forms. Calculate the Ksp of PbI_2.

3. Assume that $CaCl_2$ was added to an aliquot of saturated $Ca(OH)_2$., will the Ksp of $Ca(OH)_2$ too high or too low? Explain.

Experiment 7

Iodimetric (direct) titration of Vitamin C tablets

Introduction:

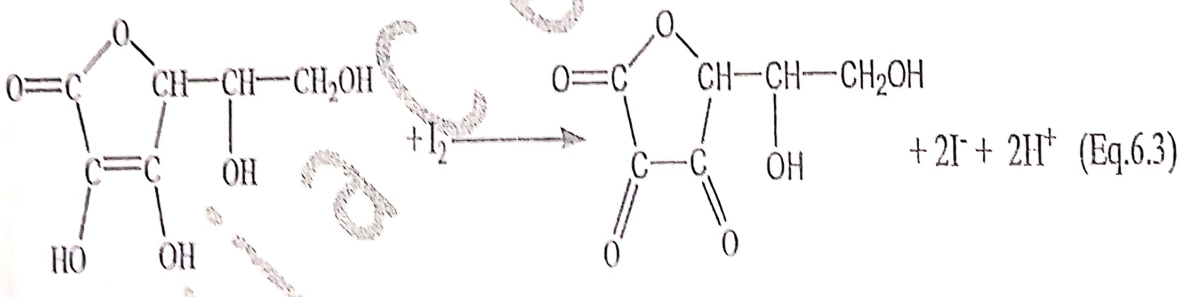
Iodine (I_2) is slightly soluble in water, but in presence of iodide ion(I^-) it forms the triiodide ion(I_3^-) which is very soluble.

$$I_2 + I^- \rightarrow I_3^-$$

An excess of iodide ion is added to decrease the volatility of I_2 by increasing the solubility (shift the equilibrium to the right). In chemical processes the iodine is used as an oxidizing agent (direct or iodimetry) while the iodide is used as a reducing agent (idometry or indirect). Iodine solution is standardized against standardized sodium thiosulfate solution (Na2S2O3)(Eq.6.2), the deep blue color of starch-iodine complex serves as an indicator.

$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

In this experiment, Vitamin C (ascorbic acid, C6H8O6) is determined by direct titration with standardized iodine solution, it is oxidized to dehydroascorbic acid by iodine.(Eq.6.3)



Experimental Procedure

A) Standardization of 0.05 M iodine solution.

1. Weigh accurately about 2g of anhydrous, pure sodium thiosulfate ($Na_2S_2O_3$). Transfer to 500 ml volumetric flask. Dissolve in previously boiled distilled water or deionized water (~250 ml), add about 0.03 g of Na_2CO_3, and stir until the solid has dissolved. Fill to the mark and mix well.

2. Wash and clean your burette, rinse it with the sodium thiosulfate solution (discard) and then fill it with $Na_2S_2O_3$ solution, record the exact volume (reading).

3. Pipette 25 ml of your iodine solution (Note 1) to a conical flask, dilute to about 50 ml with distilled water, add 1ml of 3M H_2SO_4 (for acidification) .

4. Immediately, titrate your iodine solution with the thiosulfate, when you get a faint yellow solution, add 2ml of starch and continue the titration, taking as the end point the change in color from blue to colorless.(repeat the procedure from step 3 so that you have 3 successful titrations)

5. Calculate the mean molarity of your iodine solution.

6. **NOTE 1:** If the iodine solution is not prepared you may prepare it:

Weigh about 3 g of iodine, place in 250 ml beaker, add ~ 10 g of KI, dissolve in 100 ml distilled water. Stire to dissolve all iodine, transfer to 250 ml reagent bottle, fill to about 250 ml, mix well.

B) Determination of ascorbic acid content of vitamin C tablets

1- Weigh accurately 15g of vitamin C (Tang Juice) , transfer to Erlenmeyer flask, and dissolve in about 150 ml of distilled water, add 3-5 ml of starch.

2- Titrate immediately (due to the fact that vitamin C is readily oxidized by oxygen) with standardized iodine solution to the appearance of dark blue color.

3- Calculate the % of ascorbic acid in the tablets.

POSTLAB QUESTIONS:

1-What is the daily recommended amount of vitamin C for healthy adult?

2- What is the purpose of adding Na_2CO_3, to thiosulfate solution?

3- If 1.0 g of vitamin C tablet treated as described in this experiment and titrated with 35ml of 0.2N of I_2 solution, calculate the % of ascorbic acid in the tablet?

Experiment 8

Argentometry (MOHR'S METHOD)

Introduction

In Mohr's method, a colored precipitate forms at the end point. Potassium chromate is used as an indicator. The titration reaction is :

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl \quad \text{(white precipitate)}$$

The end point reaction is:

$$2Ag^+ + CrO_4^{2-} \rightarrow Ag_2CrO_4 \quad \text{(red precipitate)}$$

The chromate ions react with silver ions after most of the chloride ions have been consumed. In other words, Ag_2CrO_4 is more soluble than AgCl. Chromate ion is yellow; the AgC1 precipitate is white while Ag_2CrO_4 precipitate is red. Thus to see a red color over the yellow color, the eye requires that the CrO_4^{2-} concentration is very small. Because of this the Mohr's method imparts a titration error.

Notice, however, that:

a. You should keep stirring the solution during the course of titration to get rid of the red precipitate of Ag_2CrO_4 that forms locally where the titrant is added to supersaturation with Ag^+. Only at the end point will the red color of Ag_2CrO_4 persist.

b. Operate at room temperature since is Ag_2CrO_4 much more soluble at high temperature.

Control the pH of the solution by using a buffer that has a range from 6.5 - 9 for the following reasons:

- In a strongly acidic solution, the end point appears rather late because any Ag_2CrO_4 formed will not precipitate but would rather he converted to the soluble dichromate ($Cr_2O_7^{2-}$) according to the following reaction:

$$\mathbf{CrO_4^{2} + 2H^+ \rightarrow Cr_2O_7^{2-} + H_2O}$$

- If the pH is greater than 9 (strongly basic), a brown precipitate of hydrous silver oxide is formed that would interfere with the required precipitate according to:

$$\mathbf{Ag^+ + OH^- \rightarrow AgOH \rightarrow Ag_2O_{(s)} + H_2O}$$

Experimental Procedure:

A) Standardization of silver nitrate by Mohr's method:

1. Dry sodium chloride (NaCl) in the oven for two hours at 105 °C.
2. Weigh accurately about 0.6000 g of dried NaCl.
3. Dissolve is distilled water and transfer quantitatively into a 100.0 mL volumetric flask. Dilute to the mark then shake the solution well.
4. Pipette 10.0 ml, of the standard chloride solution in a 250 mL conical flask and titrate with the silver nitrate solution using 1 mL chromate indicator until a faint but distinct change in color of the precipitate occurs (white precipitate changes to a reddish precipitate).
5. Repeat the titration with another 10.0 mL of the same standard chloride solution.
6. Calculate the average molarity of the silver nitrate solution and also report the concentration as g per liter.

B) Determination of a mixture of halids NaCl+ KCl according to Mohr's method:

1. Pipette 10.0 ml of the unknown solution into a 250 mL conical flask and add 1 mL of potassium chromate indicator.
2. Titrate against the standard silver nitrate solution.
3. Repeat the titration with another 10.0 mL of the unknown solution.
4. Record the average volume required for the titration.
5. Given that the total mass of both halides is 0.0650 g in 10.0 mL of the mixture, calculate the mass of NaCl and KCl in the 10.0 ml.
6. Calculate the mass of NaCl and KCl expressed as g per liter of sample solution.

Questions:

1. What is the role of chromate ions in chloride determination?
2. As potassium chromate is an oxidizing agent, what would happen to chloride determination if the sample were consists of organic matter (say 100 mg/L glucose) as well.
3. Find the **concentration** of chloride in a 25 mL solution to which few drops of K_2CrO_4 were added, if the end point required 20 mL of 0.10 M $AgNO_3$.

Experiment 9

Argentometry (Volhard method)

Introduction

Precipitate-forming titrations are widely used for determination of the halides, chloride (Cl^-), bromide (Br^-) and iodide (I^-), using silver nitrate ($AgNO_3$) as the titrant.

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl \qquad \text{(white precipitate)}$$

$$Ag^+(aq) + I^-(aq) \rightarrow AgI \qquad \text{(yellow ppt)}$$

In this experiment, chloride will be quantitatively precipitated from acidic solution by adding an excess of silver nitrate solution. The amount of excess $AgNO_3$ is determined by precipitating Ag^+ with thiocyanate (SCN^-) using Fe(III) as an indicator, when SCN^- is slightly excess the Fe(III) forms the red complex $FeSCN^{2+}$. This is commonly known as Volhard method.

$$Ag^+(aq) + SCN^-(aq) \rightarrow AgSCN \text{ (ppt)}$$

$$Fe^{3+} + SCN^- \rightarrow FeSCN^{2+} \qquad \text{(red)}$$

Experimental Procedure

A) Preparation of 0.02M $AgNO_3$ standard solution.

This solution is served as a primary standard, so be careful in your preparation

1- 1-Weigh **accurately** about 1.7g of standard grade $AgNO_3$ (previously dried).

2- 2- Using a powder funnel, carefully transfer the solid $AgNO_3$ to a 500 ml volumetric flask.

3- Wash the funnel with distilled water into the volumetric flask (be sure to transfer the entire solid from the funnel to the flask).

4- Half-fill the volumetric flask with distilled water and shake very well.

5- When the solid dissolved, fill to the mark (distilled water) and shake very well.

6- Transfer the solution to a brown storage bottle (previously washed, cleaned and rinsed with your $AgNO_3$ solution), label your bottle.

B) Preparation and standardization of 0.02M potassium thiocyantae (KSCN) solution.

1- Dissolve about 0.97 g of KSCN in 500 ml volumetric flask. Half-fill, when the solid dissolved, fill to the mark. Mix well.

2- Wash and clean your burette, rinse with KSCN solution (discard), and then fill it with KSCN, take the correct volume reading.

3- Pipette 25 ml of $AgNO_3$ solution into Erlenmeyer flask, dilute to ~ 50 ml with distilled water, add 2ml of concentrated HNO_3 and 2ml of ferric alum indicator.

4- Titrate with KSCN solution (vigorously shaking) to faint pale buff color; if your solution is orange, you have over-titrated.

5- Repeat the procedure (from step 3) for another 2- aliquots.

6- Calculate the molarity of your KSCN solution.

C) Determination of chloride in unknown sample.

1- Weigh accurately 0.35-0.4 g of your unknown sample, transfer to 250ml volumetric flask, dissolve in small portions of distilled water, when dissolved completely fill to the mark. Mix very well.

2- Pipette 25ml of your unknown sample into Erlenmeyer flask, add 2ml of concentrated HNO_3.

3- Add twice 25 ml (pipette) of your $AgNO_3$ solution (total of 50 ml).

4- Add 2 ml of ferric alum indicator and 5ml of diethyl ether (to prevent end-point fading)

5- Titrate with KSCN to the pale buff color (the color will fade slowly).

6- Repeat the procedure (from step C-2) so that you have a total of 4-replicates.

7- Calculate the % mass of chloride in your unknown sample.

POSTLAB QUESTIONS:

1- Why is it important to protect the $AgNO_3$ solution from light?

2- What is the effect of adding an excess of ammonium sulfate indicator?

3- Volhard method requires that the sample solution should be acidic during the titration, explain why?

4- Explain how the fading of end point takes place and why diethyl ether preventing it?

5- A solid sample weighing 0.50 g was dissolved in water and treated with 50.0 ml of 0.02M of $AgNO_3$, the excess Ag^+ was titrated with 32.0 ml of 0.015M KSCN, calculate the % of Cl in the sample.

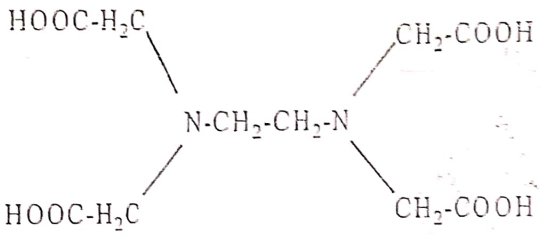
Experiment 10

Complex-formation titration

Determination of water hardness using EDTA

Introduction:

Ethylenediaminetetraacetic acid which is commonly abbreviated as EDTA is the most widely used complexometric titrant for variety of metals. Its structure is shown below:



The two nitrogen atoms and the four carboxyl groups give the ability of EDTA to be a multidentate (hexa) ligand, the fully protonated form of EDTA is abbreviated as H_4Y, other EDTA species are H_3Y^-, H_2Y^{2-}, HY^{3-} and Y^{4-} , the distribution of the five EDTA species is a pH dependent. In moderately acidic medium (pH 3-6) the H_2Y^{2-} species is predominant, while at pH 12 and higher the Y^{4-} is the predominant one.

EDTA forms stable, water soluble, 1:1 complexes with metal ions,

$$M^{n+} + Y^{4-} \rightleftharpoons MY^{-(4-n)} \qquad K_{abs} = \frac{[MY^{-(4-n)}]}{[M^{n+}][Y^{4-}]}$$

K_{nbs} is the absolute stability constant or the absolute formation constant. When M= Zn^{2+}, Ca^{2+} the log K_{obs} is 16.50 and 10.5 respectively. This refers to very high stability constant.

In the titration of metal ions with EDTA, Eriochrome Black T is atypical indicator, it behaves as a weak acid $(H_2In)^-$ as shown below (eq 9.2).

$$H_2In^- \longrightarrow HIn^{2-} + H^+ \quad K_1 = 5 \times 10^{-7}$$

red blue

In basic solutions at pH ≥ 7, the blue HIn^{2-} is the predominant species. However, in the presence of metal ions like Mg^{2+} it forms the red-purple complex. Because of the weak Ca^{2+} complex with the indicator, magnesium (Mg^{2+}) which forms stronger complex with indicator is added to the EDTA solution before the standardization of this solution. Upon titration of Ca^{2+} with EDTA, the Ca^{2+} replaces Mg^{2+} in MgY^{2-}. The liberated Mg^{2+} complexed with the indicator and forming the red $MgIn^-$, with the slight excess of EDTA the solution turns blue as a consequence of the reaction.

$$MgY^{2-} + Ca^{2+} \rightarrow CaY^{2-} + Mg^{2+}$$

$$In^{3-} + Mg^{2+} \rightarrow MgIn^- \quad \text{(red)}$$

$$Mg\text{-}In^- + HY^{3-} \rightarrow HIn^{2-} + Mg\text{-}Y^{2-}$$

Red Blue

Experimental Procedure

A) Preparation of EDTA standard

1- Weigh approximately about 1.0 g of previously dried (at 80 °C) disodium dihydrogen EDTA dihydrate ($Na_2H_2Y.2H_2O$).

2- Transfer the amount a long with about 0.05g of $MgCl_2.6H2O$ to a 500mL volumetric flask using a powder funnel. Add ~ 250 ml of deionised water.(dissolution may take 10-20 minutes), if the solution appears turbid add some drops of 0.1M of NaOH solution.

3- When the entire solid has dissolved, fill with deionised water to the mark.

B) Preparation of Ca^{2+} primary standard.

1- Weigh accurately about 0.1 g of previously dried $CaCO_3$.

2- Transfer to 250ml volumetric flask.

3- Add 1:1 HCl solution dropwise until the solution is clear.

4- Fill to the mark with deionised water. Mix well.