ISLAMIC UNIVERSITY OF TECHNOLOGY (IUT)
BOARD BAZAR, GAZIPUR.
THE ORGANIZATION OF ISLAMIC COOPERATION (OIC)
DEPARTMENT OF COMPUTER SCIENCE & ENGINEERING (CSE)

Course No: CHEM- 4242 **Title: Chemistry Lab**

MARKS: 75 **CREDIT: 3 hours per alternative week.**

Distribution of marks:
Attendance & Report on Experiment …….15
Examination……15
Viva Voice ……...……..15
Quiz ……...….15 x 2 = 30

TOTAL MARKS ……. .75

CONCISE SYLLABUS: Quantitate Inorganic Analysis,

ADVICE TO THE STUDENT:
Chemistry Laboratory is a place where seriousness is very much essential. Any time a serious accident may happen due to carelessness of the student. If there is an accident it will never be reversible. Student will not get a second chance. Understanding of what to do, what will not to do, will serve well in minimizing the danger for the student. Advance preparation for the sessional class is very much important. For student, to get good results of the sessional course, there are several things a student should try to do.

Firstly, Student should understand this laboratory manual and how to use it effectively. It is his duty to learn it carefully.

Secondly, he must try to understand both the purpose and the principle the experiments he will do.

Thirdly, he will need to organize his time effectively in advance of each laboratory period. As in the chemistry laboratory a student has to work with various hazardous chemicals, he should wear an APRON during his practical work. *WITHOUT APRON NO STUDENT* will be advised to work in the laboratory. To avoid unnecessary hazards of explosion a student should never pour any reagent back into a stock bottle. By this a student may add impurities which could add spoil the experiment for the person using the stock reagent after him. So, pouring chemicals back into bottle is a dangerous practice.

A student should not undertake any unauthorized experiment. The chances of an accident are high particularly with an unauthorized experiment which has not been completely checked to reduce the hazard. In chemistry laboratory, there are many inflammable substances which are always danger of fire. So, student is advised not to smoker or put inflamed stick here and there. Most of the chemicals are toxic to some extent, so a student should never eat nor drink in the laboratory. There is always the possibility that whatever a student eats or drinks it may become contaminated with the hazardous material.

NOTE BOOK:

For recording data obtained during experiments: ``***A BOUND NOTE BOOK*`` should be used. The note book should have consecutive numbered pages. If it does not, student should number the pages immediately. The note book is the place where all primary data must be recorded. It is a bad laboratory practice to record primary data on loose sheet, or perishable paper. Data must be recorded in permanent ink. Teacher may wish to check the students note book any time. So, a student should always have it up to date. The preparation of reports is accomplished by using the data or information recorded in the laboratory note book. When a student begins the actual experiment, his note book should always be kept nearby so that he will be able to record in it those result which he obtains when working in the laboratory. Nothing should be kept on the table, except instruction manual, note book and calculator.

LABORATORY GLASSWARE:

Since laboratory glassware is very expensive and the person who is working with this glassware is responsible for it. Student should give proper attention and take special care of it. Needless maltreatment of laboratory equipment's may cost student in big amount of money.

Mistreatment of equipment's can also cause of time loss in the laboratory. A student should be familiar with the equipment of his experiment.

CLEANING OF GLASSWARE:

Glassware can be cleaned most easily if it is done immediately after use. It is good practice to wash the equipment right way. With time the materials left in the apparatus begin to attack the surface of the glass. Various washing powder and cleaning agents may be used in washing glassware. Synthetic detergent may also be used.

REPORT WRITING:

Students are advised to follow the following sequences in order to write their report on the experiment done in the class.

l. Introduction:

Theory of the process (students should mention the name and objective of the experiment) the methods, chemical reaction involved in the experiment and the types of indicators used.

2. Chemicals and apparatus used:

The name of the chemicals / reagents used in the experiment should be mentioned.

3. Experimental Procedure:

4. Experimental data:

Each table will have a title for which it is meant. Student should mention accurately the units of the parameters and experimental conditions like, whether the experiment is carried out in acidic or basic media, experimental temperature etc.

5. Calculations:

The students must have a proper clear concept of units, specially of concentration units used in the calculation of results.

6. Results:

Briefly mention the result / results obtained.

7. Discussion

Course Objectives:

1. To impart a scientific approach and to familiarize the applications of chemistry in the field of technology.
2. To develop the experimental skill of the students

Course Outcome:

1. An ability to gain knowledge about different types of qualitative and quantitative estimation.
2. An ability to understand, explain and use instrumental techniques for chemical analysis and to analyze the quality of water by determining its chemical parameters.
3. To acquire the skill for the preparation of engineering materials.

EXPT. NO: 1

Outcomes

At the end of this lab exercise you should be able to:

- Prepare different types of standard solution.
- Perform the experimental procedure without any difficulty, become familiar. with acid base titration.
- Calculate the concentration of unknown solution.

(a) STANDARDIZATION OF NAOH SOLUTION WITH STANDARD OXALIC ACID SOLUTION

Preparation of N/10 oxalic acid solution (MW = 126):

Transfer 0.63 g. of pure oxalic acid (HOOC-COOH).$2H_2O$) in a 100 mL volumetric flask and dissolved in distilled water and make up to the mark. Normality of the prepared acid solution will be calculated as:

$$N_{acid} = \frac{\text{Weight taken in g} \times 0.1\ \text{N}}{0.63\ \text{g}} \ldots\ldots\ldots\ldots\ldots (2)$$

PROCEDURE:

Take 10 mL of supplied NaOH solution in a conical flask by means of a pipette and dilute it to about 50 mL. Add 1-2 drops of phenolphthaline indicator to the solution. Then add standard oxalic acid solution drop by drop from a burette. Shake the flask frequently while adding the acid solution. Stop the addition of oxalic acid solution as soon as the pink colour of the solution just disappears. Record the burette reading. The burette reading should be taken carefully at the lower meniscus of the liquid. Difference of the initial and final burette reading gives the volume of the acid added. The process should be repeated at least four times, and these should agree within $\pm$0.1 mL.

Reaction: a HOOC−COOH (aq) + bNaOH (aq) → p NaOOC−COONa (aq) + qH_2O (l)

Calculation:
Formula method:

(a) Calculate the normality of the supplied NaOH solution by using the following relation.

$$C_{acid} \times V_{acid} = C_{base} \times V_{base} \ldots\ldots\ldots\ldots\ldots (2)$$

(b) Unitary method: Calculate the results using unitary methods.

(b) STANDARDIZATION OF HYDROCHLORIC ACID WITH STANDARD NaOH SOLUTION

Dilution of Concentrated HCl acid to approximately 0.1 N solution:

The strength of commercially supplied hydrochloric acid is about 9.4 N. According to dilution formula, we know

$$C_{dilute} \times V_{dilute} = C_{conc.} \times V_{conc.}$$

$$V_{conc} = \frac{C_{dilute} \times V_{dilute}}{C_{Conc}} \ldots\ldots\ldots\ldots\ldots (1)$$

$$= \frac{0.1\ N \times 100\ mL}{9.34}$$

$$= 1.1\ mL$$

Take about 1.2-1.3 mL concentrated HCl in a 100 mL volumetric flask and add distilled water to make up to the mark.

PROCEDURE:

Take 10 mL of supplied NaOH solution in a conical flask by means of a pipette and dilute it to about 50 mL. Add 2-3 drops of phenolphthaline indicator to the solution. Then add previously prepared HCl acid (approx. 0.1N) solution drop wise from a burette. Shake the flask frequently during the addition of HCl acid solution. Stop the addition of HCl solution as soon as the yellow colour of the solution just changes to orangevv. Record the burette reading. The burette reading should be taken carefully at the lower meniscus of the liquid. Difference of the initial and final burette reading gives the volume of the acid added. The process should be repeated at least four times and these should agree within $\pm$0.1 mL. Calculate the normality of the dilute HCl and then calculate the strength of commercially supplied HCl solution by using the equation 2 and 3, respectively. Calculate the percentage of error for the strength of commercial HCl acid.

Calculation:

(a) Formula method:

$$C_{dil.HCl} \times V_{dil.HCl} = C_{NaOH} \times V_{NaoH} \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

$$C_{dil.HCl} \times V_{dil.HCl} = C_{Conc.HCl} \times V_{Conc.HCl} \ldots\ldots\ldots (3)$$

(b) Unitary method: Calculate the results using unitary methods.

Question:

1. Which one of NaOH and Oxalic acid is the primary standard substance in this experiment?
2. Explain giving definition of primary standard and secondary standard substance.
3. Can you explain why indicator changes its color?
4. Why is Phenolphthalein chosen as the suitable indicator for this experiment? Use titration curve for explanation.
5. Write the balanced reaction. Mention the type of reaction with definition.
6. Calculate the strength of NaOH and HCl solution in normality (M).

EXPT. NO : 2

Outcomes

At the end of this lab exercise you should be able to:

- Know about iodometry, iodometric titration, and redox titration.
- Perform the experiment based on redox titration.
- Calculate the concentration of unknown secondary standard substance.

(a) STANDARDIZATION OF SODIUM THIOSULPHATE SOLUTION WITH A STANDARD POTASSIUM DICHROMATE SOLUTION

PREPARATION OF 100 ML 0.1N POTASSIU DIDHROMATE SOLUTION (MW = 294):

Transfer 0.49 g pure $K_2Cr_2O_2$ into a 100 mL volumetric flask and then dissolve with distilled water and make upto the mark.

PROCEDURE:

Take 4 mL of 10% potassium iodide (KI) solution in a conical flask and dilute to about 50 ml. Add about 0.3 g of $NaHCO_3$ and shake the flask until the salt dissolves. Add about 4 mL of concentrated HCl acid and then add 10 mL standard $K_2Cr_2O_2$ solution by means of a pipette in the same flask. Shake the flask and cover it with a watch glass, allow the solution to stand for about five minutes in the dark. Rinse the watch glass and dilute the solution about 100 mL. Titrate the liberated iodine with sodium thiosulphate solution from a burette until the brown colour fades (light yellow). Then add about 1 mL of starch solution and continue titration by adding sodium thiosulfate solution from the burette until one drop of the sodium thiosulphate solution changes the colour of the solution from deep blue to light green. This is the end point. Repeat the titration at least four times.

Reaction:

$K_2Cr_2O_7(aq) + 14HCl(aq) + 6KI(s) = 2CrCl_3(aq) + 3I_2(g) + 8KCl(aq) + 7H_2O(l)$ (in dark)

$6\ Na_2S_2O_3\ (aq) + 3I_2\ (g) = 6NaI\ (aq) + 3Na_2S_4O_6\ (aq)$ (during titration)

Therefore, 1 mol $K_2Cr_2O_7 \equiv 3$ mol I2 $\equiv 6$ mol $Na_2S_2O_3$

And finally, 1 mol $K_2Cr_2O_7 \equiv 6$ mol $Na_2S_2O_3$

Calculation:

(a) **Formula method:** Calculate the strength of sodium thiosulphate solution using the following equation:

$$C_{Na_2S_2O_3} \times V_{Na_2S_2O_3} = C_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7} \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

(b) **Unitary method:** Calculate the results using unitary methods.

(b) ESTIMATION OF COPPER CONTAINED IN A SUPPLIED SOLUTION BY IODOMETRIC METHOD

PROCEDURE:

Pipette out 10 mL. of supplied copper salt solution into a conical flask. Add a few drop of dil. Na_2CO_3. A pale greenish precipitate should appear. Dissolve the precipitate by adding few drop of acetic acid (CH_3COOH). Add about 10 mL of 10% potassium iodide (KI) solution and titrate the liberated iodine against the standard thiosulphate solution (Standardized previously) until the brown colour of iodine changes to light yellow. Add 1 or 2 mL of starch solution and continue titration till the blue colour begins to fade. Now add few drops of 10% Ammonium thiocyanate solution and continue titration until the blue color is just discharged. Calculate the amount of copper present in one liter of the supplied solution.

Reactions:

$2CuSO_4\ (aq) + 4KI(s) \rightarrow Cu_2I_2\ (aq) + I_2\ (g) + 2K_2SO_4\ (aq)$ (in dark)

$2\ Na_2S_2O_3\ (aq) + I_2\ (g) = 2NaI\ (aq) + Na_2S_4O_6\ (aq)$ (during titration)

So, 2 mol $Na_2S_2O_3 \equiv 1$ mol $I_2 \equiv 2$ mol $CuSO_4$

(a) **Formula method:** Calculate the strength of sodium thiosulphate solution using the following equation:

$$C_{Na_2S_2O_3} \times V_{Na_2S_2O_3} = C_{Cu^{2+}} \times V_{Cu^{2+}} \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

(b) **Unitary method:** Calculate the results using unitary methods.

1 mL 1N $Na_2S_2O_3 \equiv$ 0.06354 g of Cu^{2+}

Question:

1. Define oxidation and reduction reactions in terms of electron transferred and in terms of oxidation number?
2. Give half-reactions for $K_2Cr_2O_7$, KI, $Na_2S_2O_3$ and I_2 and then level them as oxidizing and reducing agents with explanation?
3. What are Iodometric and Iodimetric titrations? Give an example.
4. What is the function of $NaHCO_3$ used in this experiment? Why is the solution kept in a dark box/place and for at least 5 minutes?
5. Write the structure of starch. Mention the advantages of using starch as an indicator in this experiment. Indicator is used in this experiment at the last stage – why?
6. What is the function of acetic acid and Na_2CO_3?
7. What is the function of NH_4CNS in this titration?

EXPT. NO: 3

Outcomes

At the end of this lab exercise you should be able to:

- Apply Beer Lambert law for the determination of any species quantitively using UV-spectrometer.
- Know about calibration curve and details calculation for the estimation of different organic, inorganic, and other materials.

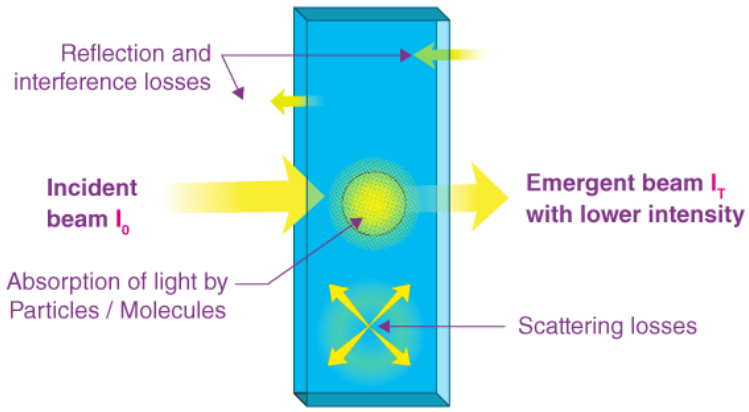
Determination of wavelength of absorption maximum and colorimetric estimation of Fe^{3+} in solution.

Theory:

Beer Lambert law $A = \varepsilon cL$

Where $A = \frac{I_0}{I_t} = \frac{1}{T}$

- A is the amount of light absorbed for a particular wavelength by the sample.
- ε is the molar extinction coefficient
- L is the distance covered by the light through the solution
- c is the concentration of the absorbing species
- I is the intensity of emergent beam
- I_0 is the intensity of incident beam.



Method:

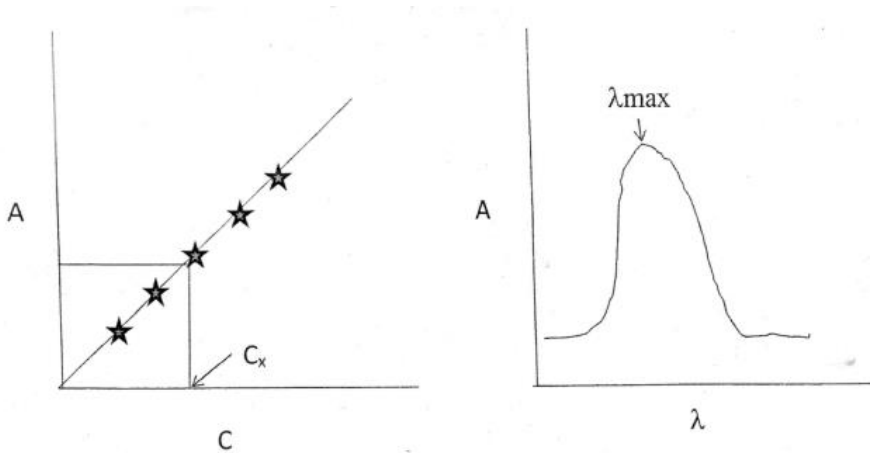
1. Prepare 100 ppm Fe^{3+} stock solution.
2. Transfer 1 mL, 2 mL, 3 mL, 4 mL and 5 mL stock solution to the 50 mL individual volumetric flasks to prepare different concentrated solution.
3. Add 1 ml 6N HNO_3 solution into 5 known concentrated volumetric flasks and one unknown sample 50 mL volumetric flask and one for blank 50 mL volumetric flask.
4. Add 5 mL NH_4SCN (20%) in the above 7 mentioned flasks.
5. Wait for 10 minutes.
6. Add distilled water upto 50 mL mark.

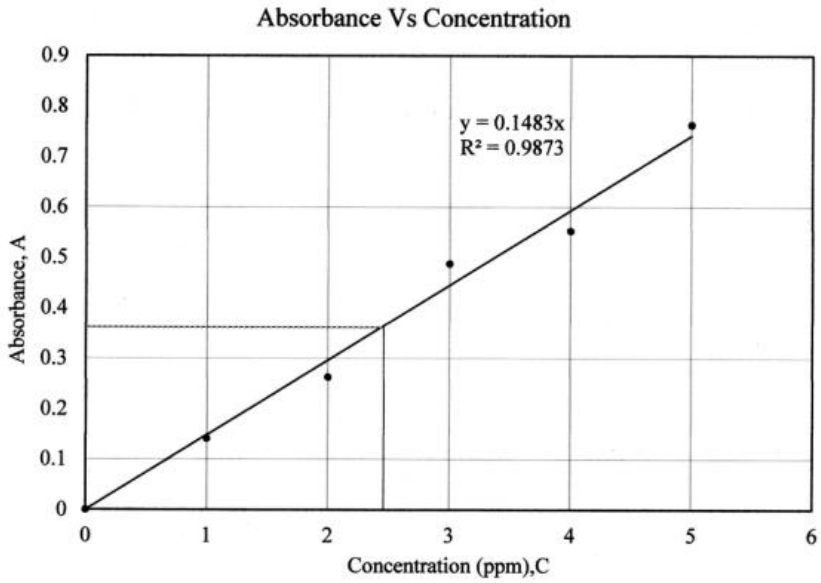
$Fe^{3+} + 6SCN^- = [Fe(SCN)_6]^{3-}$

Here NH_4SCN is added in excess amount so that the complex formed had the formula of $[Fe(SCN)_6]^{3-}$.

Measurement of absorbance:

1. Open the UV-spectrophotometer software in the PC and wait for starting the windows.
2. Take blank solution in a quartz cell or cuvette and place it in the spectrometer (left and right side would be clear face).
3. Press base line and auto zero.
4. Then take experimental solutions in the quartz cell (starting from low concentration to high concentration) and measure the absorbance of the corresponding samples.
5. Draw calibration curve and find out the molar extinction coefficient from the calibration curve.
6. Use the calibration curve to find out the concentration of the unknown solution.





Why HNO_3 is added but H_2SO_4 is not used?

HNO_3 was added to prevent hydrolysis and formation of ferric hydroxide. In absence of HNO_3 following reaction may occur

$Fe^{3+} + H_2O = Fe(H_2O)_3 + 3H^+$

H_2SO_4 can also be used to suppress hydrolysis but not recommended. Because the sulphate ion has a certain tendency to form complexes with Fe^{3+} ions.

Why does absorbance have no unit?

Absorbance doesn't have any unit because it is the ratio of the amount of light that passes through a solution compared to the amount of light that is passed into it. Sometimes you may see absorbance expressed in "absorbance units", which is abbreviated as AU and has no dimension.

What are the limitations of Beer-Lambert law?

Following are the limitations of Beer-Lambert law:

- A diluted solution is used.
- There shouldn't be a scattering of the light beam.
- Monochromatic electromagnetic radiation should be used.

Why does Beer-Lambert law fail at higher concentrations?

Beer-Lambert law fails at higher concentrations because the linearity of the law is limited to chemical and instrumental factors. When the solution has higher concentrations, the proximity between the molecules of the solution is so close that there are deviations in the absorptivity. Also, when the concentration is high, the refractive index changes.

What is Beer-Lambert's law for absorption spectroscopy?

Beer-Lambert's law for absorption spectroscopy is a linear relationship between the absorbance and the concentration of an absorbing species. The states imply that type, as well as the concentration of the molecules, are necessary.

State the situations when Beer's law is not obeyed.

Following are the situations when Beer's law is not obeyed:

- When different types of molecules are in equilibrium with each other.
- An association complex is formed by the solute and the solvent.
- When fluorescent compounds are used.
- When thermal equilibrium is attained between the excited state and the ground state.

Experiment 4

Drawing the pH-neutralization curves from the titration of a strong acid with a strong base and calculating the concentration of the strong acid.

Theory:

When alkali solution is gradually added to an acid solution, the pH of the solution increases due to neutralization of H^+ ions. At the end point there is a sharp increase in the value of pH. The curve showing the variation of pH with the amount of alkali added is called the neutralization curve. The end point is marked by point of inflection in the curve. Further, knowledge of this curve allows selection of appropriate acid-base indicators for titration. For this purpose, a pH meter will be used.

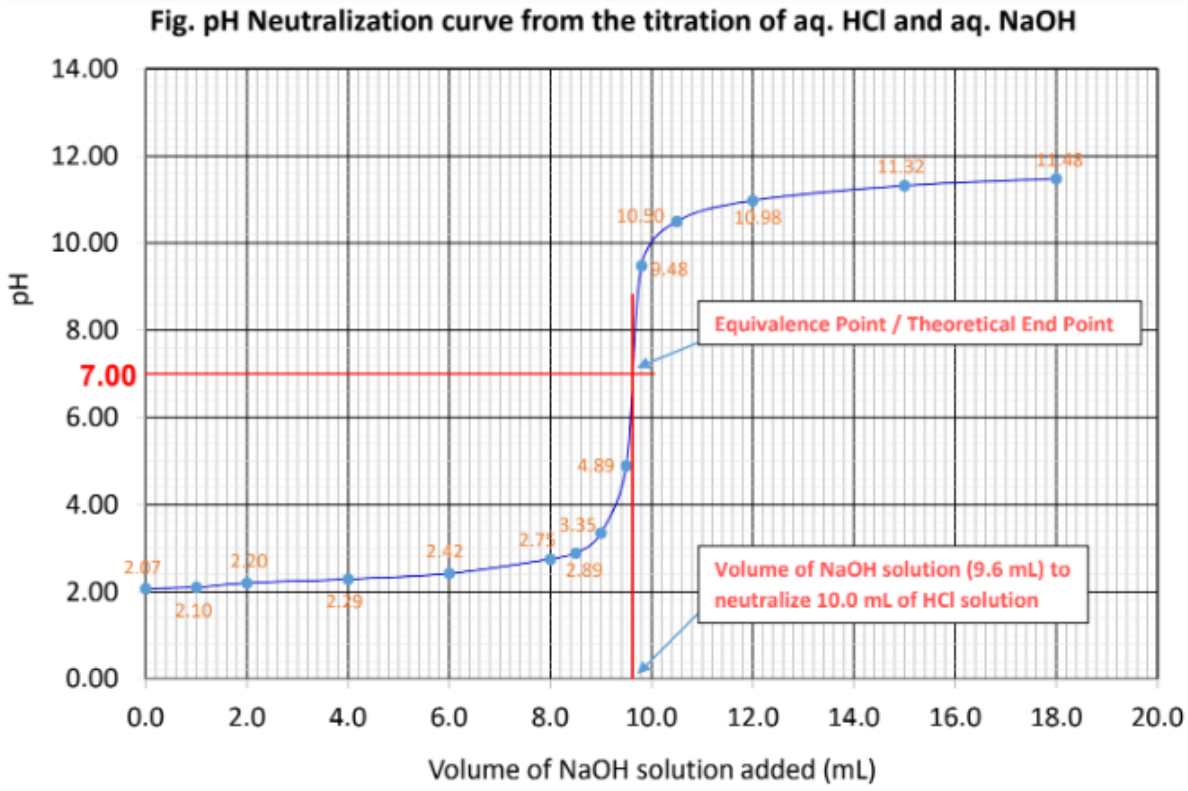
Procedure:

1. Take 10.0 ml of the supplied HCl solution in a 250 ml beaker, dilute to about 100 ml (90 ml water +10 ml HCl).
2. Place the electrode of the pH meter in the above solution.
3. Fill the burette with the supplied 0.1 M NaOH solution.
4. Add about 0.5 ml aliquot from the burette to the solution in the beaker. Stir the solution and measure it's pH. After adding 9 ml NaOH solution then gradually add NaOH solution with 0.2 ml interval up to 11 ml. Add NaOH solution 1ml to 15 ml.
5. Tabulate burette readings, volume of alkali added, and pH of the solution.

Volume of HCl	Volume of NaOH solution added from Burette (mL)	pH
10.0	0.0	
	1.0	
	2.0	
	3.0	
	4.0	
	5.0	
	6.0	
	7.0	
	8.0	
	8.5	
	9.0	
	9.5	
	10.0	
	10.5	
	11	
	12	
	13	
	14	
	15	
	16	
	17	
	18	
	19	
	20	

6. Plot pH vs volume of NaOH solution added.

7. Draw a smooth curve; locate the equivalent point (end point). This is the volume of alkali required to neutralize the acid.
8. Calculate the concentration of HCl solution in terms of Molarity and Normality.



Questions:
1. What is meant by pH? Calculate the pH values of 0.1 M H_2SO_4 and 0.1 M NaOH.
2. Draw pH – Neutralization curves for the titration of different types of acids and bases (ex. Strong acid and strong base) and comment on the suitable indicators for those titrations.

Experiment No 5

Determination of the order of reaction from the Kinetic studies of thiosulphate ions and hydrogen ions in aqueous solution.

Outcomes

At the end of this lab exercise you should be able to:

- Describe the rate of reaction, order of reaction, and molecularity of the reaction.
- Perform the experimental procedure without any difficulty, determine order of the reaction.
- Describe the reaction mechanism

Theory:

Thiosulphate undergoes disproportionation to sulphur and sulphur dioxide in presence of acid.

$S_2O_3^{2-}(aq) + 2\ H^+(aq) \rightarrow S(s) + SO_2(g) + H_2O(l)$

The rate law for this reaction is given below

$C = k\ [S_2O_3^{2-}(aq)]^m\ [H+(aq)]^n$ ……..(1)

Where *m* and *n* are the orders of the reaction with respect to $S_2O_3^{2-}(aq)$ and $H^+(aq)$ respectively and k is specific reaction rate constant which depends on the temperature at which reaction is carried out. The effect of concentration of the reactants on the rate of the reaction can be studied by measuring the specific reaction rate.

The reaction between thiosulphate ions and hydrogen ions is very easy to study. The products sulphur is produced in the colloidal form and causes turbidity in the solution. As the reaction proceeds, the amount of sulphur increases, and the solution becomes more and more turbid with time. The progress of the reaction can be followed by direct observation of the extent of turbidity in the solution with naked eye. If the concentration of Sulphur is *x* mol/L at any time *t* then rate of the reaction is given by

$C = x/t$ …….. (2)

If the time t, taken for the formation of this particular amount of sulphur (*x* mol/L) can be monitored by observing any physical changes at a certain condition. Then *x* can be taken as a constant for each of the experiment with different initial concentrations of $S_2O_3^{2-}(aq)$ and $H^+(aq)$ can be realized as the rate of the reaction. Now eqn-1 can be written

$x/t = k\ [S_2O_3^{2-}(aq]^m\ [H^+]^n$……..(3)

If a set of experiment is carried out with different concentration of [$S_2O_3^{2-}(aq)$] but leaving the same concentration of [H^+ *(aq)]* in each experiment. Then the above equation (3) can be written as

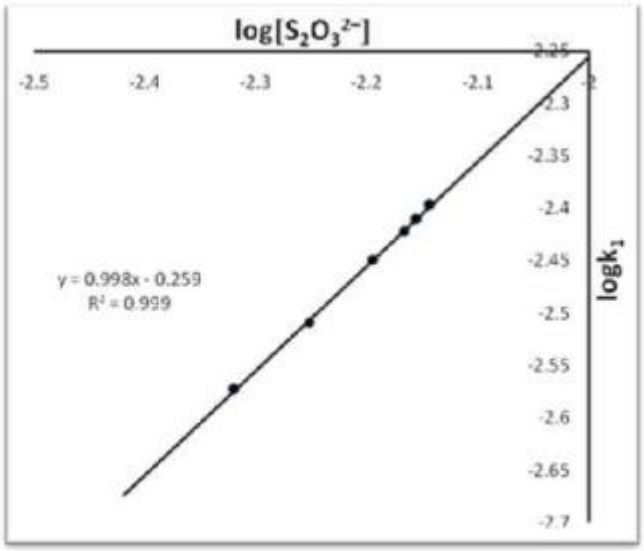
$1/t = k'\ [S_2O_3^{2-}(aq]^m$ ……..(4) { let $x=1,\ k\ [H^+]n = k'$ }

Taking logarithm on both side of the equation-4

$\log (1/t) = \log k' + m \log [[S_2O_3^{2-}(aq]]$

$\Rightarrow \log (1/t) = m \log [S_2O_3^{2-}(aq] + \log k'$ ……..(5)

Now plot of log (1/t) vs. log $[S_2O_3^{2-}(aq]$ will be a straight line and the slope *m* is the order of the reaction with respect to $S_2O_3^{2-}$.



Here, log (1/t)=logK_1

Apparatus: Beaker 250ml (2), glass rod, stopwatch.

Procedure:

(1) Take 5.0 ml of 0.1M sodium thiosulphate solution in a beaker and dilute it with 40.0 ml. of water.

(2) Then, add 5.0 ml. of a dil. HNO_3 solution to it and start recording time as soon as the acid is added.

(3) Gently shake the solution for a very short time to mix up all the components and place the beaker over a cross drawn on a piece of paper.

(4) Observe any physical changes occurring in the solution. Gradually the amount of sulphur in the solution increases. Eventually there is so much sulphur that it is no longer possible to see the cross through the solution. Note down the time taken for the cross to disappear.

(5) Repeat the experiment but using sodium thiosulphate solutions of different concentrations according to the Table-1. In each run, record how long it take for the same amount of sulphur to be formed in each experiment.

(6) Plot a graph of log (1/t) (vertically) against log *[$S_2O_3^{2-}$] (*horizontally*).* Interpret this plot and determination of the order (m) of the reaction.

Exp. No	$S_2O_3^{2-}$ *(mL)*	H_2O *(mL)*	Dil. HNO_3 (mL)	T (sec)	1/t (sec ^{1})	log (1/t)	New conc $S_2O_3^{2-}$*(molL^{-1})*	log ($S_2O_3^{2-}$*)*
1								
2								
3								
4								
5								
6								
7								

Question:

1. What is meant by rate of a chemical reaction? What are the factors influencing the rate of the reaction?
2. Define order and molecularity of reaction. Define first order and Zero order reaction.
3. What are the methods used to determine the order of a reaction? Write the principle of any method.
4. What is half life? Discuss how the half-life is related to the initial reaction of reactant for different order reactions.