

AMERICAN INTERNATIONAL UNIVERSITY – BANGLADESH (AIUB)

Department of Natural Science (Chemistry)

Faculty of Science & Technology

Program: B.Sc. Eng'g (EEE/CoE/CSE/IPE)



CHEMISTRY LABSHEETS

CHEM 1101: CHEMISTRY (EEE/CoE/CSE/IPE)

(Quantitative Inorganic Analysis)

**Volumetric titration: Acid-base titration, Redox titration, Estimation of Cu & Fe
Total hardness of water and Conductometric titration**

List of the Experiments :

1. STANDARDIZATION OF SODIUM HYDROXIDE (NaOH) SOLUTION WITH STANDARD OXALIC ACID ($C_2H_2O_4 \cdot 2H_2O$) SOLUTION.
2. STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM HYDROXIDE (NaOH) SOLUTION.
3. STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM CARBONATE (Na_2CO_3) SOLUTION.
4. DETERMINATION OF TOTAL HARDNESS OF WATER USING ERIOCHROME BLACK T (EBT) AS INDICATOR.
5. STANDARDIZATION OF SODIUM THIOSULPHATE ($Na_2S_2O_3 \cdot 5H_2O$) SOLUTION WITH STANDARD POTASSIUM DICHROMATE ($K_2Cr_2O_7$) SOLUTION.
6. ESTIMATION OF COPPER CONTAINED IN A SUPPLIED SOLUTION BY IODOMETRIC METHOD.
7. DETERMINATION OF FERROUS ION (Fe^{2+}) IN A SUPPLIED SOLUTION OF IRON SALT BY STANDARD POTASSIUM DICHROMATE ($K_2Cr_2O_7$) SOLUTION.
8. DETERMINATION OF STRENGTH OF A WEAK ACID (CH_3COOH) AGAINST A STRONG ALKALI (NaOH) SOLUTION BY MEASURING CONDUCTANCE.
9. STANDARDIZATION OF POTASSIUM PERMANGANATE ($KMnO_4$) SOLUTION WITH STANDARD SODIUM OXALATE ($Na_2C_2O_4$) SOLUTION.
10. DETERMINATION OF FERROUS IONS (Fe^{2+}) IN A SUPPLIED SOLUTION OF IRON SALT BY STANDARD POTASSIUM PERMANGANATE ($KMnO_4$) SOLUTION.

Text: 1. M. Mahbubul Huque and A. Jabber Mian, "Practical Chemistry", 2nd ed. (1972), Student Ways, ISBN: Not found. **References:** 1. J. Mendham, R. C. Denney, J. D. Barnes and M. Thomas, "Vogel's Text Book of Quantitative Chemical Analysis", 6th ed. (2000), Pearson Education Ltd, ISBN: 81-7808-538-0; 2. G. H. Jeffery, J. Bassett, J. Mendham, R. C. Denney, "Vogel's Text Book of Quantitative Chemical Analysis", 5th ed. (1989), Longman (ELBS), ISBN: 0-582-25167-2



CHEM 1101: CHEMISTRY (EEE/CoE/CSE/IPE)

EXPERIMENT NO. 1: STANDARDIZATION OF SODIUM HYDROXIDE (NaOH) SOLUTION WITH STANDARD OXALIC ACID (HO₂C-CO₂H, 2H₂O) SOLUTION.

OBJECTIVE: To know the strength of a secondary standard solution (for example, NaOH) against a primary standard solution by acid-base titration.

THEORY:

- (i) *Methods:* Acid-base titration,
- (ii) *Reactions:* HO₂C-CO₂H + 2NaOH = NaO₂C-CO₂Na + 2H₂O
- (iii) *Indicator:* Phenolphthalein

APPARATUS:

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, Stand and clamp etc.

REQUIRED CHEMICALS:

- 1. Supplied NaOH solution
- 2. Standard oxalic acid solution
- 3. Phenolphthalein indicator

PREPARATION OF APPROX. 0.1N OXALIC ACID SOLUTION. Transfer approx. 0.63 gram of pure oxalic acid (HOOC-COOH.2H₂O) in a 100 ml measuring flask and then dissolve it with distilled water up to the mark. Normality of the prepared acid solution will be calculated as follows:

$$\text{Strength of oxalic acid solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.63} \text{ (N)}$$

PROCEDURE: Take 10 mL of NaOH solution in a conical flask by means of a pipette and dilute it to about 50 ml. Add 1-2 drops of phenolphthalein 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 color of the solution just disappears. Note 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 thrice. Take the mean of the readings. Calculate the normality of the supplied NaOH solution.

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(Expt.1 contd.)

EXPERIMENTAL DATA:

Table: Standardization of supplied NaOH solution against standard oxalic acid solution by acid-base titration.

No. of reading	Vol. of NaOH (in mL.)	Vol. of Oxalic acid (burette reading) (in mL)			Mean (in mL)
		Initial	Final	Difference	
1	10				
2	10				
3	10				
4	10				

CALCULATIONS:

Strength of supplied NaOH solution:

$$V_{\text{NaOH}} \times N_{\text{NaOH}} = V_{\text{Oxalic acid}} \times N_{\text{Oxalic acid}}$$

RESULTS:

Students should know

- What are gram-equivalent weight, normality and molarity?
- Atomic weight, molecular weight of NaOH and HOOC-COOH, 2H₂O
- Why phenolphthalein is used?
- Reasons behind the change of colour.

Text: M. Mahbubul Huque and A. Jabber Mian, "Practical Chemistry", 2nd ed. (1972)



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EXPERIMENT NO. 2: STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM HYDROXIDE (NaOH) SOLUTION.

OBJECTIVE: To know the strength of HCl solution (being a solution made from secondary standard substance) against a previously standard solution by acid-base titration.

THEORY:

- (i) *Method:* Acid-base titration
- (ii) *Reactions:* 1. $\text{HO}_2\text{C}-\text{CO}_2\text{H} + 2\text{NaOH} = \text{NaO}_2\text{C}-\text{CO}_2\text{Na} + 2\text{H}_2\text{O}$
2. $\text{NaOH} + \text{HCl} = \text{NaCl} + \text{H}_2\text{O}$
- (iii) *Indicators:* Phenolphthalein, Methyl orange

APPARATUS:

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, Stand and clamp etc.

REQUIRED CHEMICALS:

1. Supplied NaOH solution
2. Standard oxalic acid solution
3. HCl acid solution
4. Phenolphthalein indicator
5. Methyl orange indicator

(A) Standardize the supplied NaOH solution as in Experiment No. 1

$$\text{Strength of oxalic acid solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.63} \text{ (N)}$$

Table-1: Standardization of supplied NaOH solution against standard oxalic acid solution by acid-base titration.

No. of reading	Vol. of NaOH (in mL)	Vol. of Oxalic acid (burette reading) (in mL)			Mean (in mL)
		Initial	Final	Difference	
1	10				
2	10				
3	10				

Strength of supplied NaOH solution:

$$V_{\text{NaOH}} \times N_{\text{NaOH}} = V_{\text{Oxalic acid}} \times N_{\text{Oxalic acid}}$$

Name: _____, ID No: _____, Section (Group): _____

(Expt.2 contd.)

(B) Preparation of approximately 0.1N hydrochloric acid solution:

Take 10 ml conc. HCl in a 1000 ml measuring flask and add distilled water up to the mark.

PROCEDURE: Take 10 mL of NaOH solution in a conical flask by means of a pipette and dilute it to about 50 mL. Add 2-3 drops of methyl orange indicator to the solution. Then add previously prepared (approx. 0.1N) HCl acid solution drop wise from a burette. Shake the flask frequently during addition of HCl acid. Stop the addition of HCl acid solution as soon as the yellow color of the solution just changes to orange or pink. Note the burette reading. Repeat the process at least three times and take the mean of the readings. Calculate the strength of the dilute HCl solution and from there calculate the strength of commercial HCl.

EXPERIMENTAL DATA:

Table-2: Standardization of supplied HCl solution against standard NaOH solution by acid-base titration.

No. of reading	Vol. of NaOH (in mL)	Vol. of HCl (burette reading) (in mL)			Mean (in mL)
		Initial	Final	Difference	
1	10				
2	10				
3	10				
4	10				

CALCULATIONS:

(A) Strength of supplied dil. HCl solution:

$$V_{\text{NaOH}} \times N_{\text{NaOH}} = V_{\text{dil. HCl}} \times N_{\text{dil. HCl to be determined}}$$

(B) Strength of conc. HCl solution:

$$V_{\text{dil. HCl}} \times N_{\text{dil. HCl determined}} = V_{\text{conc. HCl taken}} \times N_{\text{conc. HCl to be determined}}$$

RESULTS:

Students should know

- What is normality and molarity?
- Atomic weight, molecular weight and gram equivalent weight of NaOH, HCl and HOOC-COOH, 2H₂O
- Why phenolphthalein and/or methyl orange are used?
- Reason of using methyl orange instead of phenolphthalein.

Text: M. Mahbubul Huque and A. Jabber Mian, "Practical Chemistry", 2nd ed. (1972)



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EXPERIMENT NO. 3: STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM CARBONATE (Na₂CO₃) SOLUTION.

OBJECTIVE:

To know the strength of HCl solution (being a solution made from secondary standard substance) against a weak base like Na₂CO₃ by acid-base titration.

THEORY:

- (i) *Method:* Acid-base titration,
- (ii) *Reaction:* $\text{Na}_2\text{CO}_3 + \text{HCl} = \text{NaHCO}_3 + \text{NaCl}$ (pH ~9.0)
 $\text{NaHCO}_3 + \text{HCl} = \text{NaCl} + \text{CO}_2 + \text{H}_2\text{O}$ (pH ~4.0)
- (iii) *Indicator:* Phenolphthalein, Methyl orange

APPARATUS: Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, Stand and clamp etc.

REQUIRED CHEMICALS:

- 1. HCl acid solution,
- 2. Na₂CO₃ solution,
- 3. Phenolphthalein indicator
- 4. Methyl Orange indicator

PREPARATION OF APPROX. 0.1N Na₂CO₃ SOLUTION: Transfer approx. 0.53 gm of anhydrous Na₂CO₃ in a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{Strength of sodium carbonate solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.53} \text{ (N)}$$

PROCEDURE: Take 10 mL of Na₂CO₃ solution in a conical flask and dilute it to about 50 mL. Add 1-2 drops of phenolphthalein and titrate against dilute HCl solution (prepared as expt. no.2) contained in a burette. Now note the burette reading when just one drop of HCl discharges the pink color of the solution. This is the first end point. Then add 2–3 drops of methyl orange inside the same conical flask and continue titration against the same HCl solution. The end point reached when the yellow color of the solution just changes to faint pink (or orange). Note the burette reading. This is the second end point. The difference of the burette reading from initial to second end point will be the volume of the acid required for titration. Repeat the whole experiment 2-3 times and take the mean reading initial to second end point. *Take last reading without using phenolphthalein.* Calculate the strength of supplied dilute HCl solution and then find out the strength of concentrated HCl.

Name: _____, ID No: _____, Section (Group): _____

(Expt.3 contd.)

EXPERIMENTAL DATA:

Table: Standardization of supplied HCl solution against standard Na₂CO₃ solution by acid-base titration.

No. of reading	Vol. of Na ₂ CO ₃ (in mL)	Vol. of HCl (in mL)			Difference between (a) and (c) (in mL)	Mean (in mL)
		Initial (a)	1 st End-point (b)	2 nd End-point (c)		
1	10					
2	10					
3	10					
4*	10		---			

*4th reading with methyl orange only

CALCULATIONS:

(A) Strength of supplied dil. HCl solution:

$$V_{\text{Na}_2\text{CO}_3} \times N_{\text{Na}_2\text{CO}_3} = V_{\text{dil. HCl}} \times N_{\text{dil. HCl to be determined}}$$

(B) Strength of conc. HCl solution:

$$V_{\text{dil. HCl}} \times N_{\text{dil. HCl determined}} = V_{\text{conc. HCl taken}} \times N_{\text{conc. HCl to be determined}}$$

RESULTS:

Student should know:

- Is Na₂CO₃ a primary standard substance?
- Tell atomic weight, molecular weight and gram equivalent weight of HCl and Na₂CO₃.
- Can you use methyl orange first instead of phenolphthalein? If not why?
- Can you calculate the normality and molarity of HCl and Na₂CO₃?

Text: M. Mahbulul Huque and A. Jabber Mian, "Practical Chemistry", 2nd ed. (1972)



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EXPERIMENT NO. 4: DETERMINATION OF TOTAL HARDNESS OF WATER USING ERIOCHROME BLACK T (EBT) AS INDICATOR.

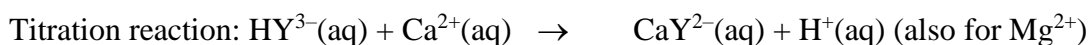
OBJECTIVE: To determination of total Hardness of water using Eriochrome Black T (EBT) as indicator.

THEORY:

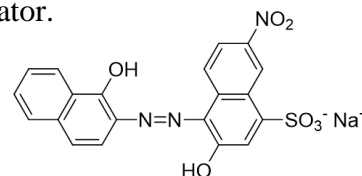
(i) *Method:* Complexometric titration. A complexometric titration is based on the formation of a complex between the analyte and the titrant. The chelating agent EDTA is very commonly used to titrate metal ions in solution. These titrations generally require specialized indicators that form weaker complexes with the analyte. A common example is Eriochrome Black T for the titration of calcium and magnesium ions to calculate hardness of water (see Lab Manual page at 21-23).

Ethylenediaminetetraacetic acid (EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of dye such as Eriochrome Black T (EBT) is added to an aqueous solution containing calcium and magnesium ions at a P^H of 10.0 ± 0.1 , the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all the magnesium and calcium has been complexed the solution turns from wine red to blue, making the end point of the titration.

(ii) *Reaction:* (Y= deprotonated part of EDTA, In=EBT Indicator)



(iii) *Indicator:* Eriochrome Black T (EBT), a complexometric indicator.

**APPARATUS:**

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, Litmus paper, dropper, Stand and clamp, Spirit lamp etc.

REQUIRED CHEMICALS:

- (1) 0.01M EDTA solution,
- (2) Conc. HCl acid,
- (3) NaOH solution
- (4) Buffer solution (pH 10)
- (5) EBT indicator

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(Expt. 4 contd.)

Preparation of 0.01M EDTA solution (100 ml): Weigh out accurately 0.372 g of di-sodium EDTA ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_8\text{Na}_2 \cdot 2\text{H}_2\text{O}$) powder into a 100ml. volumetric flask and add a little water to dissolve it. Make up to mark with distilled water.

$$\text{Strength of EDTA solution} = \frac{\text{Weight taken (ingm)} \times 0.01}{0.372} \text{ (M)}$$

PROCEDURE:

Take the standard EDTA solution into the burette. Take 50 ml. of sample water (tap water) by a volumetric pipette into a conical flask. Acidify the sample water with conc. hydrochloric acid (use a litmus paper) and boil for a minute to drive off carbon dioxide. Cool and neutralize it with sodium hydroxide (use litmus paper). Add about 2 ml of buffer solution (pH 10) and 2 drops of EBT indicator. Titrate the solution with a standard 0.01M EDTA solution until the color changes from wine red to blue. Repeat the titration at least two times and take the mean value. The mean of three readings of EDTA is the volume of EDTA required to calculate the total hardness.

EXPERIMENTAL DATA:

Table-2: Determination of total Hardness of water using Eriochrome Black T (EBT) as indicator.

No. of reading	Vol. of supplied water (in mL)	Vol. of EDTA (burette reading) (in mL)			Mean (in mL) (V)
		Initial	Final	Difference	
1	50				
2	50				
3	50				
4	50				

CALCULATIONS:

1 ml 0.01 M EDTA solution \equiv 1.00 mg of CaCO_3 (as shown below)

So, total hardness as mg CaCO_3 per liter of water = $(a \times b \times 1000) \div \text{ml of sample taken}$

[where, a = ml. of titrant required (mean volume of EDTA solution)

b = mg CaCO_3 equivalent to 1.00 ml EDTA titrant. = 1mg]

or, b = $1 \times \text{molarity of EDTA} \div 0.01\text{M}$

RESULTS:

Students should know

- What are hard water and soft water?
- What are temporary and permanent hardness of water?
- Define the terms Chelate and Polydentate ligand.
- What are household and industrial problems due to hard water?

Text: M. Mahbubul Huque and A. Jabber Mian, "Practical Chemistry", 2nd ed. (1972)



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EXPERIMENT NO. 5: STANDARDIZATION OF SODIUM THIOSULPHATE ($\text{Na}_2\text{S}_2\text{O}_3$) SOLUTION WITH STANDARD POTASSIUM DICHROMATE ($\text{K}_2\text{Cr}_2\text{O}_7$) SOLUTION.

OBJECTIVE: To know the strength of $\text{Na}_2\text{S}_2\text{O}_3$ solution (being a solution made from secondary standard substance) against standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution by oxidation-reduction titration.

THEORY:

- (i) *Method:* Redox titration
- (ii) *Reaction:*
 - 1. $\text{K}_2\text{Cr}_2\text{O}_7 + 14\text{HCl} + 6\text{KI} = 8\text{KCl} + 2\text{CrCl}_3 + 7\text{H}_2\text{O} + 3\text{I}_2$
 - 2. $2\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 = \text{Na}_2\text{S}_4\text{O}_6 + 2\text{NaI}$
- (iii) *Indicator:* Starch solution

APPARATUS:

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, Stand and clamp etc.

REQUIRED CHEMICALS:

- 1. 12% KI solution,
- 2. NaHCO_3 ,
- 3. Conc. HCl acid,
- 4. Standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution
- 5. $\text{Na}_2\text{S}_2\text{O}_3$ solution,
- 6. Starch solution

PREPARATION OF APPROX. 0.1N POTASSIUM DICHROMATE SOLUTION.

Transfer approx. 0.49 gram of pure $\text{K}_2\text{Cr}_2\text{O}_7$ into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{Strength of } \text{K}_2\text{Cr}_2\text{O}_7 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.49} \text{ (N)}$$

(Expt. 5 contd.)

PROCEDURE:

Take 4 mL of 12% KI solution in a conical flask and dilute to about 50 mL. Add about one gm of NaHCO_3 and shake the flask until the salt dissolves. Add 4 mL conc. HCl acid and then add 10 ml standard $\text{K}_2\text{Cr}_2\text{O}_7$ 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 (inside the desk or dark chamber). Rinse the watch glass and dilute the solution about 100mL. Titrate the liberated iodine with sodium thiosulphate solution from a burette until the brown color fades (light yellow). Add about 1 mL starch solution and continue titration by adding sodium thiosulphate solution from the burette until one drop of the sodium thiosulphate solution changes the color of the solution from deep blue to light green. This is the end point. Repeat the whole experiment 2-3 times. Calculate the strength of sodium thiosulphate solution.

EXPERIMENTAL DATA:

Table: Standardization of supplied $\text{Na}_2\text{S}_2\text{O}_3$ solution against standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution by oxidation-reduction titration.

No. of reading	Vol. of $\text{K}_2\text{Cr}_2\text{O}_7$ (in mL)	Vol. of $\text{Na}_2\text{S}_2\text{O}_3$ (burette reading) (in mL)			Mean (in mL)
		Initial	Final	Difference	
1	10				
2	10				
3	10				

CALCULATIONS:

Strength of supplied $\text{Na}_2\text{S}_2\text{O}_3$ solution:

$$V_{\text{Na}_2\text{S}_2\text{O}_3} \times N_{\text{Na}_2\text{S}_2\text{O}_3} = V_{\text{K}_2\text{Cr}_2\text{O}_7} \times N_{\text{K}_2\text{Cr}_2\text{O}_7}$$

RESULTS:Students should know

- What are redox reaction, oxidizing agent and reducing agent?
- What is the difference between acid-base and redox indicator?
- Why it is necessary to keep your experimental solution in the dark?
- Is it iodometric or iodimetric that you are performing?
- Tell molecular weight and gram equivalent weight of $\text{K}_2\text{Cr}_2\text{O}_7$ and $\text{Na}_2\text{S}_2\text{O}_3$.
- What is the function of starch?
- Can you calculate the normality and molarity of $\text{K}_2\text{Cr}_2\text{O}_7$ and $\text{Na}_2\text{S}_2\text{O}_3$?

Text: M. Mahbubul Huque and A. Jabber Mian, "Practical Chemistry", 2nd ed. (1972)



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EXPERIMENT NO. 6: ESTIMATION OF COPPER (Cu) CONTAINED IN A SUPPLIED SOLUTION OF COPPER SALT BY IODOMETRIC METHOD.

OBJECTIVE: To determine the amount of Cu^{+2} ions in a supplied solution of copper salt by iodometric method.

THEORY:

- (i) Method: Redox titration
- (ii) Reaction:
 1. $2\text{CuSO}_4 + 4\text{KI} = 2\text{K}_2\text{SO}_4 + 2\text{CuI} \downarrow + \text{I}_2$
 2. $2\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 = \text{Na}_2\text{S}_4\text{O}_6 + 2\text{NaI}$
- (iii) Indicator: Starch

APPARATUS:

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, Stand and clamp etc.

REQUIRED CHEMICALS:

- | | | |
|--|---|---|
| (1) 12% KI solution, | (2) Copper salt solution | (3) NaHCO_3 solid, |
| (4) 6M NH_4OH | (5) Conc. HCl acid, | (6) Conc. CH_3COOH |
| (7) Standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution, | (8) 10% NH_4SCN solution, | (9) $\text{Na}_2\text{S}_2\text{O}_3$ solution, |
| (10) Starch solution, | | |

(A) Standardize sodium thiosulphate solution as **Expt. No. 5**.

Table-1: Standardization of supplied $\text{Na}_2\text{S}_2\text{O}_3$ solution against standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution by oxidation-reduction titration.

No. of reading	Vol. of $\text{K}_2\text{Cr}_2\text{O}_7$ (in mL)	Vol. of $\text{Na}_2\text{S}_2\text{O}_3$ (burette reading) (in mL)			Mean (in mL)
		Initial	Final	Difference	
1	10				
2	10				

$$\text{Strength of } \text{K}_2\text{Cr}_2\text{O}_7 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.49} \text{ (N)}$$

$$\text{Strength of supplied } \text{Na}_2\text{S}_2\text{O}_3 \text{ solution (S): } V_{\text{thio}} \times N_{\text{thio}} = V_{\text{dichromate}} \times N_{\text{dichromate}}$$

(Expt. 6 contd.)

(B) Estimation of Cu ions:

PROCEDURE: Pipette out 10 mL of copper salt solution into a conical flask. Add 3-4 drops of 6M NH_4OH until a faint permanent ppt remain and then add 6-8 drops of conc. CH_3COOH . Now add about 10 ml of 12% potassium iodide (KI) solution and titrate the liberated iodine against the standard sodium thiosulphate solution (standardized previously) until the brown color of iodine changes to light yellow. Add approx. 1 mL of starch solution, solution turns intense blue and continue titration till the blue color begins to fade. Now add few drops of 10% ammonium thiocyanate solution and continue titration until the blue color is just discharged (off-white). Calculate the amount of copper present in 500 mL of copper salt solution.

EXPERIMENTAL DATA:**Table-2:** Determination of the amount of copper in a supplied solution of blue vitriol by iodometric method.

No. of reading	Vol. of Copper salt solution (in mL)	Vol. of $\text{Na}_2\text{S}_2\text{O}_3$ (burette reading) (in mL)			Mean (in mL) (V)
		Initial	Final	Difference	
1	10				
2	10				
3	10				
4	10				

CALCULATIONS: $2 \text{ CuSO}_4 \equiv \text{I}_2 \equiv 2 \text{ Na}_2\text{S}_2\text{O}_3$
 $1 \text{ ml } 1\text{N } \text{Na}_2\text{S}_2\text{O}_3 \equiv 0.06354 \text{ gm of Cu}^{2+}$

Amount of copper ions in 10 mL of copper salt solution $= 0.06354 \times V \times S \text{ gm}$

Amount of copper ions in 500 mL of copper salt solution $= 0.06354 \times V \times S \times 50 \text{ gm}$

Observe value of Cu^{2+} (in 500mL solution) $=$

Known value of Cu^{2+} (in 500mL solution) $=$

RESULTS:**PERCENTAGE OF ERROR:**

$$\frac{\text{Known value} - \text{Observed value}}{\text{Known value}} \times 100 =$$

Students should know

- Why it is necessary to keep your experimental solution in the dark?
- Is it iodometric or iodimetric that you are performing?
- Tell molecular weight and gram equivalent weight of $\text{K}_2\text{Cr}_2\text{O}_7$ and $\text{Na}_2\text{S}_2\text{O}_3$.
- What is the function of starch?
- What is the purpose of adding NH_4SCN solution?

Text: M. Mahbulul Huque and A. Jabber Mian, "Practical Chemistry", 2nd ed. (1972)



CHEM 1101: CHEMISTRY (EEE/CoE/CSE/IPE)

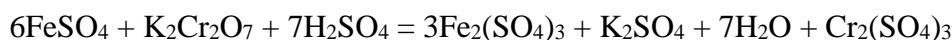
EXPERIMENT NO. 7: DETERMINATION OF FERROUS ION (Fe²⁺) IN A SUPPLIED SOLUTION OF IRON SALT BY STANDARD POTASSIUM DICHROMATE (K₂Cr₂O₇) SOLUTION.

OBJECTIVE: To know the amount of iron (Fe²⁺) in a supplied solution of iron salt by standard K₂Cr₂O₇ solution.

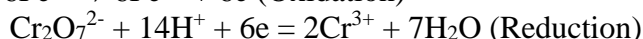
THEORY:

(i) Method: Redox titration

(ii) Reaction:



Redox half reactions: $6\text{Fe}^{2+} \rightarrow 6\text{Fe}^{3+} + 6\text{e}^-$ (Oxidation)



(iii) Indicator: Diphenyl amine, (C₆H₅)₂NH

APPARATUS:

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, Stand and clamp etc.

REQUIRED CHEMICALS:

1. Iron salt solution,
2. 5% Sulfuric acid,
3. Conc. Phosphoric acid,
4. Standard K₂Cr₂O₇ solution,
5. Diphenyl Amine indicator

PREPARATION OF APPROX. 0.1N POTASSIUM DICHROMATE SOLUTION.

Transfer approx. 0.49 gram of pure K₂Cr₂O₇ into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{Strength of K}_2\text{Cr}_2\text{O}_7 \text{ solution (S)} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.49} \text{ (N)}$$

(Expt. 7 contd.)

PROCEDURE: Take 10 mL of the supplied iron salt (Mohr's salt) solution in a conical flask. Add 50 mL 5% sulfuric acid and 5 mL of conc. phosphoric acid. Then add 4-5 drops of diphenyl amine indicator and titrate slowly against the standard potassium dichromate solution drop wise maintaining an interval of few seconds between each drop until the addition of one drop causes the formation of intense purple or violet blue coloration which remains permanent and is unaffected by further addition of dichromate solution. Repeat the experiment at least thrice. Calculate the amount of iron per 500 mL of iron salt solution.

EXPERIMENTAL DATA:

Table: Determination of the amount of iron in Mohr's salt solution using standard $K_2Cr_2O_7$ solution.

No. of reading	Vol. of Mohr's salt solution (in mL)	Vol. of $K_2Cr_2O_7$ (burette reading) (in mL)			Mean (in mL) (V)
		Initial	Final	Difference	
1	10				
2	10				
3	10				
4	10				

CALCULATIONS: 1 mL 1N $K_2Cr_2O_7$ \equiv 0.05584 gm of Fe^{2+}

Amount of iron in 10 mL of iron salt solution $= 0.05584 \times V \times S$ gm
=

Amount of iron in 500 mL of iron salt solution $= 0.05584 \times V \times S \times 50$ gm
=

Observe value of Fe^{2+} (in 500mL solution) =

Known value of Fe^{2+} (in 500mL solution) =

RESULTS:**PERCENTAGE OF ERROR:**

$$\frac{\text{Known value} - \text{Observed value}}{\text{Known value}} \times 100 =$$

Students should know

- Why it is necessary to use both the *sulfuric acid* as well as *phosphoric acid* in the reaction?
- Atomic weight, molecular weight of $K_2Cr_2O_7$ and $KMnO_4$.
- Could you use $KMnO_4$ instead of $K_2Cr_2O_7$?
- Why the solution shows light bottle green colour after addition of $K_2Cr_2O_7$.

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Attention: Please bring one graph paper

**AMERICAN
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Experiment 8

CHEM 1101: CHEMISTRY (EEE/CoE/CSE/IPE)

EXPERIMENT NO. 8: DETERMINATION OF STRENGTH OF A WEAK ACID (CH_3COOH) AGAINST A STRONG ALKALI (NaOH) SOLUTION BY MEASURING CONDUCTANCE.

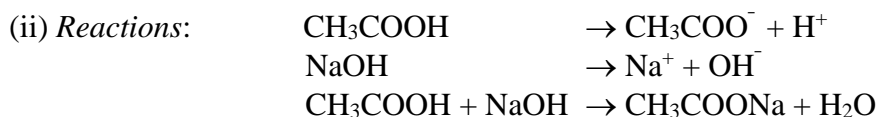
OBJECTIVE:

- To draw the titration curves by measuring the conductance
- To find out the end-point of an acid-base reaction
- To know the strength of supplied sample solution (acid or base)

THEORY:

(i) *Methods:* Conductometric titration,

The conductance of an acid solution varies with the amount of alkali solution added to it because of the change in the number and nature of the ions in the solution. The curve showing this variation is called the conductance titration curve. After complete neutralization, the conductance increases due to the addition of the conducting ions of the alkali. At the end-point there is a sharp change in the conductivity. Therefore, if the conductance titration curve is drawn graphically, the end-point of the titration can easily be determined. The conductance of a dilute acetic acid solution is due to the small amounts of H^+ ions and acetate ions resulting from the dissociation of the weak acid. When small amount of alkali is added and the H^+ ions are neutralized, an equal amount of H^+ ions are not generated by further dissociation of the weak acid because the acetate ions suppress the dissociation due to common ion effect. Therefore the conductance decreases. Upon further addition of alkali the conductance increases because of the addition of Na^+ ions and formation of acetate ions. After the end-point, the conductance increases at a sharper rate due to addition of the fast moving OH^- ions, in addition to the Na^+ ions.



APPARATUS:

Conductivity meter, Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), plastic beaker, watch glass, pipette filler, dropper, glass rod, stand and clamp etc.

REQUIRED CHEMICALS:

(1) Supplied 0.1N NaOH solution, (2) Supplied dil. CH_3COOH solution

(Expt. 8 contd.)

PROCEDURE:

Take 10 mL of the supplied CH_3COOH solution in a beaker. Place the previously washed (with hot water) conductance cell in it and add sufficient water (~200 mL) to keep the electrodes of the cell immersed. Measure the conductance of the acid solution (1st reading). Fill a burette with the supplied ~ 0.1 N NaOH solution. Add 2/1 mL NaOH solution from the burette, stir the solution and measure the conductance (2nd to 12th reading) after each addition. Get a graph paper ready for the plotting conductance data. Plot conductance data (in μs) against the final volume (in mL) of NaOH solution on graph paper and find the end-point. The end-point gives the required volume of NaOH equivalent to 10 ml of supplied dil. CH_3COOH solution. Now calculate the strength of CH_3COOH solution.

EXPERIMENTAL DATA:**Table:** Conductance-measurement of CH_3COOH and NaOH solutions using conductivity meter.

No. of reading	Vol. of CH_3COOH (in mL)	Vol. of NaOH (burette reading) (in mL)				Conductance (μs), Y
		Initial	Final	Difference	Total X	
1	10	0	0	0	0	
2		0	2	2	2	
3		2	4	2	4	
4		4	6	2	6	
5		6	8	2	8	
6		8	10	2	10	
7		10	11	1	11	
8		11	12	1	12	
9		12	13	1	13	
10		13	14	1	14	
11		14	15	1	15	
12		15	16	1	16	

CALCULATIONS:Strength of supplied CH_3COOH solution:

$$V_{\text{CH}_3\text{COOH}} \times N_{\text{CH}_3\text{COOH}} = V_{\text{NaOH}} \times N_{\text{NaOH}}$$

RESULTS:

(Attach the graph paper with this lab-sheet)

Students should know

- Define (a) electrolyte, (b) specific conductance, (c) molar conductance
- What is conductance cell?
- How does the molar conductance of a strong electrolyte vary with concentration?
- How is the molar conductance at infinite dilution determined for (a) a strong electrolyte and (b) a weak electrolyte?



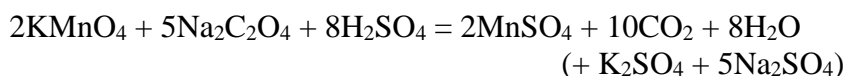
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EXPERIMENT NO. 9: STANDARDIZATION OF POTASSIUM PERMANGANATE (KMnO₄) SOLUTION WITH STANDARD SODIUM OXALATE (Na₂C₂O₄) SOLUTION.

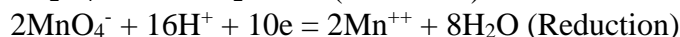
OBJECTIVE: To know the strength of supplied KMnO₄ solution (being made from a secondary standard substance) by standard Na₂C₂O₄ solution.

THEORY:

- Method:* Redox titration
- Reaction:*



Redox half reactions: $5\text{C}_2\text{O}_4^{2-} \rightarrow 10\text{CO}_2 + 10\text{e}^-$ (Oxidation)



- Indicator:* KMnO₄ act as its own indicator.

APPARATUS:

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, Stand and clamp etc.

REQUIRED CHEMICALS:

- Sodium Oxalate,
- 2N Sulfuric acid,
- Potassium Permanganate

PREPARATION OF APPROX. 0.1N SODIUM OXALATE SOLUTION.

Transfer approx. 0.67 gram of pure Na₂C₂O₄ into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{Strength of Na}_2\text{C}_2\text{O}_4 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.67} \text{ (N)}$$

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(Expt. 9 contd.)

PROCEDURE:

Pipette out 10 mL standard oxalate solution in a conical flask. Add 50 mL 2N sulfuric acid solution to it and then add 4-5 drops of KMnO_4 solution from a burette. Shake the solution until the color disappears. Continue to add permanganate solution from the burette at a moderate rate and shake the solution continuously. As the color disappears more slowly, slow down the addition of permanganate solution drop wise until one drop gives a light but definite pink color. This is the end point of the titration. Repeat the process at least thrice. Take at least last reading by heating the solution upto 60 to 70°C. Calculate the mean value of burette readings and find out the strength of supplied KMnO_4 solution.

EXPERIMENTAL DATA:

Table: Standardization of supplied KMnO_4 solution by standard $\text{Na}_2\text{C}_2\text{O}_4$ solution.

No. of reading	Vol. of Oxalate solution (in ml.)	Vol. of KMnO_4 (burette reading) (in ml.)			Mean (in ml.)
		Initial	Final	Difference	
1	10				
2	10				
3	10				
4*	10				

*4th reading by heating the solution 60-70°C

CALCULATIONS:

Strength of supplied KMnO_4 solution:

$$V_{\text{KMnO}_4} \times N_{\text{KMnO}_4} = V_{\text{Na-oxalate}} \times N_{\text{Na-oxalate}}$$

RESULTS:

Students should know

- Why you don't use any indicator in this experiment?
- Molecular weight, gram equivalent weight of $\text{Na}_2\text{C}_2\text{O}_4$ and KMnO_4 .
- Why you need to heat the experimental solution?
- What happens if you don't shake the solution properly?

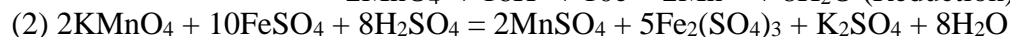
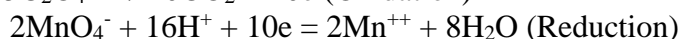
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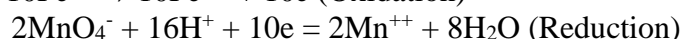
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EXPERIMENT NO. 10: DETERMINATION OF FERROUS ION (Fe^{2+}) IN A SUPPLIED SOLUTION OF IRON SALT BY STANDARD POTASSIUM PERMANGANATE (KMnO_4) SOLUTION.**OBJECTIVE:** To know the amount of iron (Fe^{2+}) in a supplied solution of iron salt by standard KMnO_4 solution.**THEORY:**(i) *Method:* Redox titration(ii) *Reaction:*

Redox half reactions: $5\text{C}_2\text{O}_4^{2-} \rightarrow 10\text{CO}_2 + 10\text{e}^-$ (Oxidation)



Redox half reactions: $10\text{Fe}^{2+} \rightarrow 10\text{Fe}^{3+} + 10\text{e}^-$ (Oxidation)

(iii) *Indicator:* KMnO_4 act as its own indicator**APPARATUS:**

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, Stand and clamp etc.

REQUIRED CHEMICALS:

(1) Iron salt solution, (2) 2N Sulfuric acid, (3) KMnO_4 solution and (4) $\text{Na}_2\text{C}_2\text{O}_4$ solution

PROCEDURE:

PREPARATION OF APPROX. 0.1N SODIUM OXALATE SOLUTION: Transfer approx. 0.67 gram of pure $\text{Na}_2\text{C}_2\text{O}_4$ into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{Strength of } \text{Na}_2\text{C}_2\text{O}_4 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.67} \text{ (N)}$$

(A) **Standardize the KMnO_4 solution by standard $\text{Na}_2\text{C}_2\text{O}_4$ solution:** Pipette out 10 mL standard oxalate solution in a conical flask. Add 50 mL 2N sulfuric acid solution to it and then add 4-5 drops of KMnO_4 solution from a burette. Shake the solution until the color disappears. Continue to add permanganate solution from the burette at a moderate rate and shake the solution continuously. As the color disappears more slowly, slow down the addition of permanganate solution drop wise until one drop gives a light but definite pink color. This is the end point of the titration. Repeat the process at least twice. Calculate the mean value of burette readings and find out the strength of supplied KMnO_4 solution.

Pipette out 10 mL of the iron (Mohr's salt) solution in a conical flask. Add about 6 mL of 2N sulfuric acid solution. Dilute to about 50 mL distilled water.

Name: _____, ID No: _____, Section (Group): _____

(Expt.10 contd.)

Titrate the resulting solution with standard potassium permanganate solution. The end point is indicated by the first appearance of permanent light pink color as procedure (A). Repeat the titration at least twice. Calculate the amount of iron per 500 mL of iron salt solution.

EXPERIMENTAL DATA:

Table-1: Standardization of supplied KMnO_4 solution by standard $\text{Na}_2\text{C}_2\text{O}_4$ solution.

No. of reading	Vol. of Oxalate solution (in mL)	Vol. of KMnO_4 (burette reading) (in mL)			Mean (in mL)
		Initial	Final	Difference	
1	10				
2	10				

Strength of supplied KMnO_4 solution: $V_{\text{KMnO}_4} \times N_{\text{KMnO}_4} (\text{S}) = V_{\text{Na-oxalate}} \times N_{\text{Na-oxalate}}$

Table-2: Determination of the amount of iron in Mohr's salt solution using standard KMnO_4 solution.

No. of reading	Vol. Of Mohr's salt solution (in mL)	Vol. Of KMnO_4 (burette reading) (in mL)			Mean (in mL) (V)
		Initial	Final	Difference	
1	10				
2	10				
3	10				
4	10				

CALCULATIONS: 1 mL 1N $\text{KMnO}_4 \equiv 0.05584$ gm of Fe^{2+}

Amount of iron in 10 mL of iron salt solution $= 0.05584 \times V \times S$ gm

=

Amount of iron in 500 mL of iron salt solution $= 0.05584 \times V \times S \times 50$ gm

=

Observe value of Fe^{2+} (in 500mL solution) =

Known value of Fe^{2+} (in 500mL solution) =

RESULTS:

PERCENTAGE OF ERROR:

$$\frac{\text{Known value} - \text{Observed value}}{\text{Known value}} \times 100 =$$

Students should know

- Why you don't have to use any indicator in this experiment?
- Molecular weight, gram equivalent weight of $\text{Na}_2\text{C}_2\text{O}_4$ and KMnO_4 .
- Why you need to heat the experimental solution?
- What happens if you don't shake the solution properly?

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