AMERICAN INTERNATIONAL UNIVERSITY – BANGLADESH (AIUB)

Department of Natural Science (Chemistry)

Faculty of Science and 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 ($C_2H_2O_4.2H_2O$) 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 ($Na_2S_2O_3.5H_2O$) 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

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Experiment 1

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EXPERIMENT NO. 1: STANDARDIZATION OF SODIUM HYDROXIDE (NaOH) SOLUTION WITH STANDARD OXALIC ACID (HO_2C-CO_2H, $2H_2O$) 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*: $HO_2C\text{-}CO_2H + 2NaOH = NaO_2C\text{-}CO_2Na + 2H_2O$
(iii) *Indicator*: Phelophthalein

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.$2H_2O$) 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:

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

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:

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

RESULTS:

Students should know

- What are gram-equivalent weight, normality and molarity?
- Atomic weight, molecular weight of NaOH and HOOC-COOH, $2H_2O$
- 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 2

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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. $HO_2C\text{-}CO_2H + 2NaOH = NaO_2C\text{-}CO_2Na + 2H_2O$

2. $NaOH + HCl = NaCl + H_2O$

(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**

Strength of oxalic acid solution = $\dfrac{Weight\,taken(in\ gm) \times 0.1}{0.63}$ (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:

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

(*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*:

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

(B) *Strength of conc. HCl solution*:

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

RESULTS:

Students should know

- What is normality and molarity?
- Atomic weight, molecular weight and gram equivalent weight of NaOH, HCl and HOOC-COOH, $2H_2O$
- 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 3

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

EXPERIMENT NO. 3: STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM CARBONATE (Na_2CO_3) SOLUTION.

OBJECTIVE:

To know the strength of HCl solution (being a solution made from secondary standard substance) against a weak base like Na_2CO_3 by acid-base titration.

THEORY:

(i) *Method*: Acid-base titration,

(ii) *Reaction*: $Na_2CO_3 + HCl = NaHCO_3 + NaCl$ (pH ~9.0)

$NaHCO_3 + HCl = NaCl + CO_2 + H_2O$ (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. Na_2CO_3 solution,
3. Phenolphthalein indicator
4. Methyl Orange indicator

PREPARATION OF APPROX. 0.1N Na_2CO_3 SOLUTION: Transfer approx. 0.53 gm of anhydrous Na_2CO_3 in a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

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

PROCEDURE: Take 10 mL of Na_2CO_3 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.

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(Expt.3 contd.)

EXPERIMENTAL DATA:

Table: *Standardization of supplied HCl solution against standard Na_2CO_3 solution by acid-base titration.*

No. of reading	***Vol. of Na_2CO_3 (in mL)***	***Vol. of HCl (in mL)***			***Difference between (a) and (c) (in mL)***	***Mean (in mL)***
		Initial (a)	***1st End-point (b)***	***2nd 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*:

$$V_{Na2CO3} \text{ x } N_{Na2CO3} = V_{dil.\ HCl} \text{ x } N_{dil.\ HCl\ \text{to be determined}}$$

(B) *Strength of conc. HCl solution*:

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

RESULTS:

Student should know:

- Is Na_2CO_3 a primary standard substance?
- Tell atomic weight, molecular weight and gram equivalent weight of HCl and Na_2CO_3.
- Can you use methyl orange first instead of phenolphthalein? If not why?
- Can you calculate the normality and molarity of HCl and Na_2CO_3?

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

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Experiment 4

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

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.

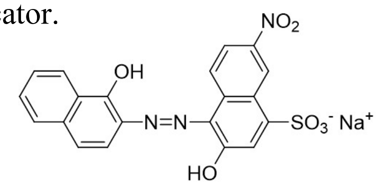
(ii) Reaction: (Y= deprotonated part of EDTA, In=EBT Indicator)

Titration reaction: $HY^{3-}(aq) + Ca^{2+}(aq) \rightarrow CaY^{2-}(aq) + H^{+}(aq)$ (also for Mg^{2+})

End point reaction: $HY^{3-}(aq) + MgIn^{-}(aq) \rightarrow MgY^{2-}(aq) + HIn^{2-}(aq)$

wine red **sky blue**

(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 ($C_{10}N_2O_8H_{14}Na_2.2H_2O$) powder into a 100ml. volumetric flask and add a little water to dissolve it. Make up to mark with distilled water.

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

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 ≡ 1.00 mg of $CaCO_3$ (as shown below)

So, total hardness as mg $CaCO_3$ per liter of water = (a x b x 1000) ÷ 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×molarity of EDTA÷0.01M

RESULTS:

Students should know

- What are hard water and soft water?
- What are temporary and permanent hardness of water?
- Define the terms Chelete and Polydendate legand.
- What are household and industrial problems due to hard water?

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