Ahsanullah University of Science and Technology

Department of Arts and Sciences

LABORATORY MANUAL
FOR
CHEMISTRY SESSIONAL COURSES

Manual I

[Quantitative Inorganic Analysis]

DOs and DON'Ts (Cautions):

- All students must wear an apron (long, full sleeve, white) before entering the lab.
- Students must not do anything without any given instruction.
- Students must not touch any chemical without any given instruction.
- Students must handle apparatus and chemicals more carefully.
- Put on Safety glass.
- Maintain cleanliness.
- Be disciplined.

Students Must Learn:

- Chemical analysis: Quantitative analysis and Qualitative analysis
- Titration, objective of titration, required apparatus
- Solution, solute, solvent, dilute solution, standard solution
- Primary standard substance, Secondary standard substance
- Units of concentartion: Molarity, Molality, Normality, %, mole fraction
- Neutralization reaction: Definition and examples
- Indicator, Function of indicator, pH range of indicators, End point
- Acids and Bases: Arrhenius, Brönsted, and Lewis concepts
- Salt: Definition, acidic, basis and neutral salts with example
- pH, Buffer solution
- Titration curves – suitable indicator, Theories of indicator
- Distilled water
- Name of the common apparatus

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- Redox reaction, oxidation and reduction half-reaction
- Oxidation number, calculation of oxidation number
- Oxidizing agent, Reducing agent
- Iodometry and Iodimetry titration
- Starch indicator, its structure
- Effect of temperature on the rate of reaction
- Auto indicator and auto catalyst
- Hardness of water, types and unit of hardness of water.
- Ligand, complex, coordinate covalent bond, chelate.

Experiment No. 01

Experiment Name: Standardization of sodium hydroxide solution with a standard solution of oxalic acid.

Preparation of standard 0.050 M oxalic acid solution:

Weigh out exactly **0.63 g** of pure crystalline oxalic acid dihydrate ($HOOC-COOH\cdot 2H_2O$) and take it in a **100 mL** volumetric flask. Dissolve it with little amount of distilled water and fill it up to the mark with distilled water.

Working Procedure:

i) Take the supplied sodium hydroxide solution in the burette.
ii) Pipette out exactly **10.0 mL** of the prepared oxalic acid solution into a conical flask.
iii) Add **1-2** drops of **phenolphthalein** as an indicator.
iv) Record the initial burette reading.
v) Add **sodium hydroxide** solution from the burette into the conical flask with constant shaking until the color changes from ***colourless to pink*** and record the burette reading again (final burette reading).
vi) Repeat the procedures **(ii-v)** at least three times and take the average (for *average, data which have differences within 0.3 should be considered)*. This value is the volume of sodium hydroxide solution required to neutralize the standard 10.0 mL of oxalic acid solution.
vii) Calculate the concentration in terms of Molarity by using both **formula** and **unitary method.**

Reaction:

$$a\ \mathbf{HOOC-COOH}\,(aq) + b\ \mathbf{NaOH}\,(aq) \rightarrow p\,\mathbf{NaOOC-COONa}\,(aq) + q\,\mathbf{H_2O}\ (l)$$

Calculation:

It is clear from the above reaction that ***a*** mol (**coefficient**) of oxalic acid reacts with ***b*** mol (**coefficient**) of sodium hydroxide. The concentration of sodium hydroxide solution (in terms of morality) can be determined using the following formula:

$$\frac{M_1V_1}{a} = \frac{M_2V_2}{b}$$

Where, M_1 = Molarity of oxalic acid solution = 0.050 M
V_1 = Volume of oxalic acid solution **taken** = 10.0 mL
M_2 = Molarity of sodium hydroxide solution **=?** M
V_2 = Volume of sodium hydroxide solution **required** = Avg. burette reading (**?** mL)

Questions:

1. Which one of NaOH and Oxalic acid is the primary standard substance in this expt. and why?
2. Write the balanced reaction. Mention the type of reaction with definition.
3. Can you explain why the indicator changes its color? **Hint:** theories of indicator action.
4. Why Phenolphthalein is chosen as the suitable indicator for this experiment? Sketch the titration curve for explanation.
5. Calculate the concentration of NaOH solution in normality (***N***).

Experiment No. 02

Experiment Name: **Standardization of HCl solution with a secondary standard solution of sodium hydroxide (NaOH)**.

Standardization of sodium hydroxide solution: The supplied sodium hydroxide solution was standardized as per the procedure described in **exp # 01.**

Working Procedure:

- **i)** Take the sodium hydroxide (NaOH) solution in the burette.**[a]**
- **ii)** Pipette out exactly **10.0 mL** of the supplied hydrochloric acid solution into a conical flask by a volumetric pipette.
- **iii)** Add **1-2** drops of **methyl orange** as an indicator.
- **iv)** Record the initial burette reading.
- **v)** Titrate the hydrochloric acid solution with the sodium hydroxide solution until the color changes from ***pink to yellow.***[b]
- **vi)** Note the final burette reading.
- **vii)** Repeat the procedure **(ii-vi)** at least three times and take the average. This value is the volume of sodium hydroxide solution required to neutralize the hydrochloric acid solution.
- **viii)** Calculate the concentration in terms of Molarity by using both **formula** and **unitary** method.

Reaction:

$$a\ \mathrm{HCl\ (aq)} + b\ \mathrm{NaOH\ (aq)} \rightarrow p\ \mathrm{NaCl\ (aq)} + q\ \mathrm{H_2O\ (l)}$$

Calculation:

Calculate the molarity of the HCl solution from the following formula:

$$M_1V_1 / a = M_2V_2 / b$$

Where, M_1 = Molarity of hydrochloric acid solution **(=?).**
V_1 = Volume of hydrochloric acid solution **taken**.
M_2 = Molarity of sodium hydroxide solution.
V_2 = Volume of sodium hydroxide solution **required** = Avg. burette reading (**?** mL)

Questions:

1. Show by pH–Neutralization curve that methyl orange is suitable indicator for this titration.
2. Do you think that phenolphthalein or methyl red can be used here? Justify your answer.
3. Calculate the concentration of HCl in terms of normality (*N*).

Note:

(a) If this experiment is not carried out just after finishing the experiment-**01** then HCl solution is better to take in the burette instead of NaOH solution.

(b) In some places colour change is written as red to orange but pink to yellow is usually observed.

Experiment No. 03

Name of the Experiment: Drawing the pH-neutralization curve from the titration of a strong acid (HCl) with a strong base (NaOH) and calculate 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 the neutralization of $\mathbf{H^+}$ 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. Furthermore, knowledge of this curve allows the 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. (before use pH meter, it should be calibrated, see page **6**)
3. Take supplied NaOH solution in the burette.
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.
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.

Questions:

1. What is meant by pH? Calculate the pH values of 0.1 M H_2SO_4 and 0.1 M NaOH solution.

2. Draw pH – Neutralization curves for the titration of different types of acids and bases and comment on the suitable indicator for those titrations.

Calibration of pH meter:

1. Wash the probe/electrode of pH meter with distilled water.
2. Immerge the probe and temperature probe approximately 1.5 inch into a buffer solution having **pH 7.0**
3. Stair the beaker gently.
4. Press ‘CAL’.
5. Not ready indication will blink on LCD untill the ready was stabilized.
6. When the ready is stable and close to the selected buffer (indicated in the right-top side of the monitor) ‘ready’ indication will appear on ‘CFM’ will blink then press CFM to confirm the calibration.(it may take **one or two minutes** to stabilized the pH meter).
7. The calibrated value is then displayed on the right-top side of the LCD will display the second expected buffer value.
8. Repeat the same procedure for the calibration of pH meter for **pH 4.0.**
9. Press CAL for quit calibration.

Experiment No. 04

Experiment Name: Standardization of hydrochloric acid (HCl) solution with a standard solution of sodium carbonate (Na_2CO_3).

Preparation of standard Na_2CO_3 solution:

Weigh out exactly **0.53 g** of pure crystalline sodium carbonate (Na_2CO_3) and take it in a **100 mL** volumetric flask. Dissolve it with a little amount of distilled water and fill it up to the mark with distilled water.

Working Procedure:

i) Take the supplied hydrochloric acid (precaution must be maintained) solution in the burette.
ii) Pipette out **exactly 10.0 mL** of the prepared sodium carbonate solution into a conical flask by a volumetric pipette.
iii) Add **1-2** drops of **methyl orange** as an indicator.
iv) Record the initial burette reading.
v) Titrate the sodium carbonate solution with the hydrochloric acid solution until the first drop changes its colour from ***yellow to pink*.**
vi) Note the final burette reading.
vii) Repeat the procedure **(ii-vi)** at least three times and take the average. This value is the volume of hydrochloric acid solution required to neutralize the 10.0 mL of Na_2CO_3 solution.
viii) Calculate the concentration in terms of Molarity by using both formula and unitary method.

Reaction:

$$a\ Na_2CO_3\,(aq) + b\ HCl\,(aq) \rightarrow p\ NaCl\,(aq) + q\ CO_2\,(g) + r\ H_2O\,(l)$$

Calculation: Calculate the molarity of the hydrochloric acid solution from the following formula:

$$M_1V_1 / a = M_2V_2 / b$$

Where, M_1 = Molarity of sodium carbonate solution (0.050 M).
V_1 = Volume of sodium carbonate solution **taken** (10.0 mL).
M_2 = Molarity of hydrochloric acid solution (?).
V_2 = Volume of hydrochloric acid solution **required**.

Questions:

1. The above reaction actually occurs in two steps. Show these steps.
2. What is salt? What type of salt sodium carbonate is: acidic or basic? Explain with reaction.
3. Sketch the titration curve and give the reason for selecting methyl orange as an indicator.
4. Why Phenolphthalein is not a suitable indicator for this titration?
5. Calculate the strength of HCl in normality (*N*).

Experiment No. 05

Experiment Name: **Standardization of sodium thiosulphate ($Na_2S_2O_3$) solution with a standard solution of potassium dichromate ($K_2Cr_2O_7$).**

Preparation of standard potassium dichromate *($K_2Cr_2O_7$)* solution:

Weigh out exactly **0.47 g** of potassium dichromate **($K_2Cr_2O_7$)** and take it in a **100 mL** volumetric flask. Dissolve it with a little volume of distilled water and make the solution up to the mark.

Working Procedure:

i) Take the supplied **sodium thiosulphate** ($Na_2S_2O_3$) solution in the burette.
ii) Take about **50 mL** of distilled water into a conical flask by using a measuring cylinder.
iii) Add about **4 mL of 10% potassium iodide (KI)** solution into this conical flask.
iv) Add about **1 g** of **sodium bicarbonate** and shake well to dissolve it.
v) Add about **3 mL of conc. hydrochloric acid** slowly while gently rotating the flask to mix the liquids. *(CO_2 produced here to drive out O_2 from solution and to blanket/cover the solution surface.)*
vi) Add exactly **10.0 mL** of **potassium dichromate** solution and mix the solution well.
vii) Cover the flask immediately with a watch glass and allow standing in a **dark box** (*to prevent the solution from light*) for **5 minutes.** (*Solution will be dark brown*).
viii) Take out the flask from the dark box and dilute the solution by adding 50 mL of distilled water. *Note the initial burette reading.*
ix) Titrate the liberated **iodine** with the sodium thiosulphate solution from the burette until the color **fades and turns pale yellow**. (compare with the colour of $K_2Cr_2O_7$ solution)
x) Now add **1 mL or 10 drops of starch** (*indicator; indicates iodine*) solution into it. The solution will become **deep violet**.
xi) Continue the addition of sodium thiosulphate solution until the solution changes its color to **light green** (*sometimes water colour*).
xii) Note the final burette reading. This is the volume of sodium thiosulphate solution required.
xiii) Repeat the procedure **(ii-xii)** at least three times and take the average value.
xiv) Calculate the concentration in terms of molarity by using both **formula** and **unitary method.**

Reactions:

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

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

Therefore, $1 \text{ mol } K_2Cr_2O_7 \equiv 3 \text{ moles } I_2 \equiv 6 \text{ moles } Na_2S_2O_3$

And finally, $\mathbf{1 \text{ mol } K_2Cr_2O_7 \equiv 6 \text{ moles } Na_2S_2O_3}$

Calculation:

Calculate the concentration of sodium thiosulphate solution from the following formula:

$$M_1V_1 / 1 = M_2V_2 / 6$$

Where, M_1 = Molarity of potassium dichromate solution
V_1 = Volume of potassium dichromate solution **taken**
M_2 = Molarity of sodium thiosulphate solution **(=?** M**)**
V_2 = Volume of sodium thiosulphate solution **required**.

Questions:

1. Define oxidation and reduction reactions in terms of electron transfer 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 as oxidizing and reducing agents with the explanation.
3. What are Iodometric and Iodimetric titrations? Give example.
4. What is the function of $NaHCO_3$ used in this experiment?
5. Why is the solution kept in the dark box/place and for at least 5 minutes?
6. Write the structure of starch. Mention the advantages of using starch as an indicator in this experiment.
7. Indicator is used in this experiment at the last stage – why?

Experiment No. 06

Experiment Name: **Estimation of the amount of copper (Cu^{2+}) present in a supplied $CuSO_4$ solution with a secondary standard solution of $Na_2S_2O_3$ by iodometric method.**

Standardization of the sodium thiosulphate solution

The supplied sodium thiosulphate solution was standardized as per procedure described in (**exp. 4).**

Working Procedure:

(i) Take the supplied sodium thiosulphate solution into the burette.
(ii) Take **exactly 10.0 mL** of the supplied $CuSO_4$ solution with a volumetric pipette into a conical flask.
(iii) Add a few drops of dilute Na_2CO_3 solution until a faint permanent precipitate appears. Dissolve the precipitate by adding CH_3COOH drop wise (make sure that the solution is acidic).
(iv) Add about **4 mL of 10% KI** solution into it and cover it with a watch glass.
(v) Keep the flask in a dark box for **5 minutes**. A **reddish-brown** color will be formed. Then add **20 mL** distilled water into a conical flask.
(vi) Note the initial burette reading.
(vii) Titrate the solution with secondary standard sodium thiosulphate solution until the color becomes **light yellow**. *(this is not the end point)*
(vii) Add **10** drops of **starch** solution (*as indicator*) into the conical flask.
(viii) Add about **10 mL of 10% NH_4SCN** solution and continue adding sodium thiosulphate solution until the color of the solution becomes **straw/pale (white)**. *(it is the end point)*
(ix) Note the final burette reading.
(x) Repeat the titration at least three times and take the average value.

Reactions:

$$2CuSO_4\,(aq) + 4KI\,(aq) \rightarrow Cu_2I_2\,(s) + I_2\,(g) + 2K_2SO_4\,(aq)\ (\textbf{\textit{in dark}})$$

$$2Na_2S_2O_3\,(aq) + I_2\,(g) = 2NaI\,(aq) + Na_2S_4O_6\,(aq)\ (\textbf{\textit{during titration}})$$

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

2 moles $Na_2S_2O_3 \equiv$ 2 moles $CuSO_4$

1 mol $Na_2S_2O_3 \equiv$ 1 mol $CuSO_4$

Calculation:

Method1: Calculate the concentration of $CuSO_4$ solution in molarity unit.

Now use the following relationship:

$CuSO_4 \rightarrow Cu^{2+} + SO_4^{2-}$

So, 1 mol $CuSO_4$ produce 1 mol Cu^{2+} ion.

Therefore, the concentration of $CuSO_4$ solution = the concentration of Cu^{2+} ion

Now, the amount of Cu^{2+} ion present in supplied $CuSO_4$ solution is:

(concentration of Cu^{2+} ion) × (ionic mass of Cu^{2+} ion)

(..…$molL^{-1}$ × 63.54 $gmol^{-1}$)

…….. gL^{-1}

Method 2: 1M thiosulphate solution ≡ 1M $CuSO_4$ =1mol/L $CuSO_4$

= 1 mol/L Cu^{2+} = 63.54 g/L Cu^{2+}

So, 1000 ml of 1M thiosulphate solution = 63.54 g of Cu^{2+}

And finally, 1ml of 1M thiosulphate solution = 0.06354 g Copper.

Questions:

1. Show the half-reactions involved here.
2. What is the function of NH_4SCN in this titration?

Experiment No. 07

Experiment name: **Standardization of potassium permanganate solution ($KMnO_4$) with a standard sodium oxalate ($Na_2C_2O_4$) solution.**

Preparation of standard sodium oxalate solution:

Weigh out accurately **0.67 g** of sodium oxalate salt in a **100 mL** volumetric flask. Dissolve it by adding a little amount of distilled water and make up to the mark with distilled water.

Working procedure:

- **(i)** Take the supplied **potassium permanganate ($KMnO_4$)** solution into the burette.
- **(ii)** Pipette out **exactly 10.0 mL** of **sodium oxalate ($Na_2C_2O_4$)** solution by a volumetric pipette into a conical flask.
- **(iii)** Add about **50 mL** of **1M Sulphuric** acid solution to the conical flask.
- **(iv)** Note the initial burette reading.
- **(v)** Carry out the titration (add 3–5 drops of $KMnO_4$ solution) at the ordinary temperature until the ***pink*** color appears throughout the solution. Allow to stand until the pink color disappears and the solution becomes **colorless**.
- **(vi)** Warm the solution to **50-60 ˚C** and continue titration to a ***permanent pink*** color.
- **(vii)** Repeat the titration at least three times and take the average value.
- **(viii)** Calculate the concentration in terms of molarity by using both formula and unitary method.

Reaction:

$$a\mathbf{KMnO_4}\ (aq) + b\mathbf{Na_2C_2O_4}\ (aq) + c\mathbf{H_2SO_4}\ (aq) = d\mathbf{K_2SO_4}\ (aq) + e\mathbf{MnSO_4}\ (aq) + f\mathbf{CO_2}\ (g) + g\mathbf{H_2O}\ (l) + h\mathbf{Na_2SO_4}\ (aq)$$

Calculation:

$$M_1V_1 / a = M_2V_2 / b$$

Where, M_1 = Molarity of sodium oxalate solution
V_1 = Volume of sodium oxalate solution **taken**
M_2 = Molarity of potassium permanganate solution **(=?)**
V_2 = Volume of potassium permanganate solution **required**

Questions:

1. Balance the above equation by the half-reaction method.
2. Label $Na_2C_2O_4$ and $KMnO_4$ as oxidizing and reducing agents with reaction.
3. Sulphuric acid is used here; hydrochloric acid or nitric acid is not used. Explain.
4. Find the normality of $KMnO_4$ and $Na_2C_2O_4$ solutions in this experiment.
5. No indicator was used in this experiment. Why?

Experiment No. 08

Experiment Name: **Estimation of ferrous ions (Fe^{2+}) present in a supplied solution of ferrous sulphate ($FeSO_4$) with a secondary standard potassium permanganate ($KMnO_4$) solution**.

Standardization of the supplied potassium permanganate solution:

The supplied potassium permanganate solution was standardized as per procedure described in the expt.#**07**.

Working Procedure:

(i) Take the secondary standard potassium permanganate solution into the burette.
(ii) Pipette out **exactly 10.0 mL** of supplied ferrous sulphate solution by a volumetric pipette into the conical flask.
(iii) Add about **10 mL of 1M sulphuric** acid solution into the solution.
(iv) Titrate the solution with $KMnO_4$ until a ***faint pink*** colour appears throughout the solution.
(v) Repeat the titration at least three times and take the average value.

Reaction:

$$2KMnO_4(aq)+10FeSO_4(aq)+8H_2SO_4(aq) = K_2SO_4(aq)+ 5Fe_2(SO_4)_3(aq)+2MnSO_4(aq) + 8H_2O(l)$$

$$\text{Therefore, 2 moles } KMnO_4 \equiv 10 \text{ moles } FeSO_4$$

$$\textbf{or, 1 mol } KMnO_4 = \textbf{5 moles } FeSO_4$$

Calculation:

Method-1:

Calculate the molarity of $FeSO_4$ solution. To find the amount of **Fe^{2+}** per litre solution, see the calculation of **Method 1** in expt. **# 06**.

$$\textbf{Here, 1 mol } Fe^{2+} = \textbf{55.85 g } Fe^{2+}$$

Method-2: Calculate the amount of iron per liter of solution from the following equations:

$$1M\ KMnO_4 \equiv 5 \times 1M\ FeSO_4$$

$$= 5 \times 55.85 \text{ g/L of } Fe^{2+}$$

So, 1 ml of 1M $KMnO_4$ = 5**x** 0.05585 g of Fe^{2+} (see Method 2 in expt.# **06**)

Questions:

1. Balance the above equation by using half-reactions.
2. What is auto indicator? Give two examples.

Experiment No. 09

Name of the Experiment: Determination of the strength of Hydrogen peroxide (H_2O_2) solution with secondary standard $KMnO_4$ solution.

Theory:

Hydrogen peroxide (H_2O_2), generally known as an oxidizer, is commonly used as a bleaching agent. It is the simplest peroxide (a compound with an oxygen-oxygen single bond). Hydrogen peroxide is a clear liquid, more viscous than water that appears colorless in dilute solution. It is also used as a disinfectant, antiseptic etc. The oxidizing capacity of hydrogen peroxide is so strong that it is considered a highly oxidizing species. Hydrogen peroxide is naturally produced in organisms as a - by product of oxidative metabolism.

Though H_2O_2 usually act as an oxidizing agent (oxidizer), in the presence of a stronger oxidizing agent like $KMnO_4$, it behaves as a reducing agent. When potassium permanganate solution ($KMnO_4$) is added to hydrogen peroxide (H_2O_2) solution in the presence of sulphuric acid the following reaction occurs. This principle is used to determine the strength of the H_2O_2.

$$2MnO^{-}_4\,(aq) + 5H_2O_2\,(aq) + 6H^+\,(aq) = 2Mn^{2+}\,(aq) + 5O_2\,(g) + 8H_2O\,(l)$$

(Note: Standardize the $KMnO_4$ solution according to the described procedure in exp: 07).

Procedure:

1. Take supplied $KMnO_4$ solution into the burette.
2. Transfer 10.0 mL of the supplied solution of H_2O_2 into a conical flask.
3. Dilute this solution with 25 mL of water and shake thoroughly.
4. Add 10 mL of 1M H_2SO_4.
5. Perform the titration with potassium permanganate solution until the ***faint pink*** colour appears throughout the solution.
6. Repeat the titration three times.

Calculation:

Method 1

Find out the concentration of H_2O_2. Then calculate the % as instructed by the course teacher.

Method 2

1. The weight of hydrogen peroxide per litre of the original solution.
2. The ‘volume strength’ i.e the number of ml of oxygen at N.T.P that can be obtained from 1mL of the original solution.

1mL 1M $KMnO_4$ solution = 0.01701× 5 g H_2O_2

= 0.08505 g H_2O_2

$$\text{Concentration of } H_2O_2 \text{ (\% by volume)} = \frac{0.08505 \times \text{vol. of } KMnO_4 \text{ in mL} \times \text{Molarity of } KMnO_4}{\mathbf{10}} \times \mathbf{100}$$

Questions:

1. Give half-reactions and explain how H_2O_2 acts as a reducing agent.

2. What is the bleaching agent?

Experiment No. 10

Experiment Name: Determination of total Hardness of water with a standard solution of EDTA using Eriochrome Black T (EBT) as an indicator.

Hardness

Water hardness was understood to be a measure of the capacity of water to precipitate soap. Soap is precipitated chiefly by calcium and magnesium ions present. Other polyvalent cations also may precipitate soap but they often are in complex forms, frequently with organic constituents, and their role in water hardness may be minimal and difficult to define. Total hardness is defined as the sum of the calcium and magnesium concentration, both expressed as calcium carbonate in mg/L (milligram/litre). The hardness may range from zero to hundreds of milligrams per litre, depending on the source and treatment to which has been subjected.

Basic Principle

Ethylenediaminetetraacetic acid (EDTA) from 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.

Preparation of 0.010 M EDTA disodium salt (EDTA) solution:

Weigh out accurately **0.372 g of EDTA ($C_{10}N_2O_8H_{14}Na_2.2H_2O$)** powder and take it into a **100 mL** volumetric flask and add a little water to dissolve it. Make up to mark with distilled water.

Working Procedure

(i) Take the **EDTA** solution in the burette.
(ii) Take **50 mL of sample water** (tap water) by a volumetric pipette into a conical flask.
(iii) **Acidify** the sample water with **conc. hydrochloric acid** (use a litmus paper) and boil for a minute to drive off carbon dioxide **(CO_2).**
(iv) **Cool** and neutralize it with sodium hydroxide solution. (Add NaOH solution dropwise until the pH of the solution reach to **10**).
(v) Check pH of the solution by using pH-strip and ensure that pH of the solution is **10**.
(vi) Add about **2 mL** of buffer solution (pH=10) and **2** drops of **EBT** indicator. The colour of solution will be ***wine red***.
(vii) Note the initial burette reading.
(viii) Titrate the sample water with a standard **0.01M EDTA** solution until the color changes from ***wine red to blue***
(ix) Repeat the titration at least three times and take the mean value. The mean of three readings of EDTA is the volume of EDTA solution required to calculate the total hardness.

Calculation

Method 1: Unitary method

Find the concentration of Ca^{2+} using 1:1 complex formation with EDTA.

Then use the relationship between Ca^{2+} and $CaCO_3$ to calculate the amount (mg) of $CaCO_3$ per litre of sample water. (*Be careful about the volume of water; it is not 10 mL*)

Method 2: (see hints below)

1 mL 0.01 M EDTA solution = 1.00 mg of $CaCO_3$ (show by calculation)

Therefore, 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. (100 mg)

Questions:

1. Define hardness of water and discuss the process to soften it.
2. What are the permanent and temporary hardness of water?
3. Define ligand and discuss the classification of ligand.
4. Define the terms: Chelating agent, Chelate, Chelation and polydendate ligand.
5. Write down the structure of EDTA. What type of reaction takes place between the Calcium and EDTA?
6. What is soap? What is meant by micelle? How soap is related with hard water or hardness of water?

Hints:

[Molar mass of $CaCO_3$ is 100 g/mol]

1 mol EDTA ≡ 1 mol Ca^{2+} = 1 mol $CaCO_3$ = 100 g $CaCO_3$

So 1M EDTA ≡ 1M $CaCO_3$ = 1mol/L $CaCO_3$ = 100 g/L $CaCO_3$

Hence, 1000 ml 0.01M EDTA = 0.01 x 100 g $CaCO_3$

= 0.01x 100 x 1000 mg $CaCO_3$

= 1000 mg $CaCO_3$.

Experiment No. 11

Name of the Experiment: Determination of the available Chlorine in bleaching Powder.

Theory:

Bleaching powder consists essentially of a mixture of calcium hypochlorite, $Ca(OCl)_2$ and the basic calcium chloride $CaCl_2.Ca(OH)_2.H_2O$. Some free slaked lime is usually present. The active constituent is the hypochlorite (OCl^-), which is responsible for bleaching action. Upon treating bleaching powder with hydrochloric acid, chlorine is liberated.

$$OCl^- (aq) + Cl^- (aq) + 2H^+ (aq) = Cl_2 (g) + H_2O (l)$$

The available chlorine refers to the chlorine liberated by the action of dilute acid, and is expressed as the percentage by weight of the bleaching powder.

The available chlorine in bleaching powder can be determined iodometrically. For this the bleaching powder solution or suspension is treated with an excess of a solution of potassium iodide, and strongly acidified with acetic acid.

$$OCl^- (aq) + 2I^- (aq) + 2H^+ (aq) = Cl^- (aq) + I_2 (g) + H_2O (l)$$

The liberated iodine is titrated with a standard solution of sodium thiosulphate.

Procedure:

1. Weigh out accurately about 2.5 g (weight taken = X) of the bleaching powder into a clean ceramic mortar. Add a little water, and rub the mixture to a smooth paste. Add a little more water, triturate with the **pastle,** allow the mixture to settle and pour off the milky liquid into a **250 mL** volumetric flask. Grind the residue with a little more water and repeat the operation until the whole of the sample has been transferred to the flask either in solution or in a state of very fine **suspension**, and the mortar washed quite clean.
2. **Add water** to the volumetric flask up to mark and shake well.
3. Transfer **25 mL** of the turbid liquid immediately with a volumetric pipette into a **250 mL** conical flask.
4. Add about **25 mL** of water into the conical flask.
5. Add about **2 g** of solid **Iodate-free potassium iodide** to the conical flask.
6. Add **10 mL** of glacial acetic acid and shake, **Iodine** will be liberated immediately.
7. Keep the flask closed with a watch glass and place in a dark box for **5 minutes**. Then add **25 mL** distilled water to the flask.

8. Titrate the liberated iodine with standard **sodium thiosulphate** solution **(standardized as per expt.04)** until the color fades to pale yellow.
9. Add 1mL of starch indicator, a deep violet color appears.
10. Continue the titration with sodium thiosulphate until the color just disappears.

Reactions:

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

$$I_3^- + 2S_2O_3 \longrightarrow 3\,I^- + S_4O_6$$

Note: Sodium thiosulphate should be standardized according to procedure (04) before/ after the experiment.

Calculation:

Available Chlorine content (%) in bleaching powder =

$$\frac{\mathbf{0.03546g \times a \times b \times V \times 100}}{\mathbf{P \times Q}}$$

Where **a =** volume of $Na_2S_2O_3$ required in titration.
b = Molarity of $Na_2S_2O_3$ solution.
v = Total volume of suspension (250ml).
P = Amount in gm of bleaching powder taken.
Q = Volume of suspension (25ml).taken for titration.

Another Calculation:

Amount of bleaching powder in 50 mL = $\frac{X \times 50}{500}$ g

1mL 1*M* – $Na_2S_2O_3$ = 0.03546 g of Cl

Experiment No. 12

Name of the Experiment: **Preparation of Iron (11) Sulphate heptahydrate $FeSO4.7H_2O$**

Theory:

When metallic iron (Fe) reacts with sulphuric acid, it replaces the acidic hydrogen (H^+) in H_2SO_4 and products iron (11) sulphate.

$$\mathbf{Fe(s) + H_2SO_4\,(aq) \longrightarrow FeSO_4\,(aq) + H_2\,(g)}$$

$FeSO_4$ crystallizes from the aqueous solution with seven water molecules.

Procedure:

1. Pour **10 mL** of concentrated sulphuric acid slowly into **200 mL** of distilled water in a 500 mL beaker and add **10 g** of iron turnings.
2. When the reaction **slows** down, heat the beaker until the acid is practically all neutralized as indicated by the fact that evolution of hydrogen gas ceases.
3. **Filter** the content of the beaker, using a filter paper to remove any undissolved residue.
4. The filtrate should give a **bottle green** color.
5. If the solution is oxidized, appreciable amount of sulphuric acid must be added until the color becomes green. Oxidation will be indicated by the change in color of the solution from bottle green to **yellowish shade** of green or by the formation of rusty precipitate.
6. **Slowly** evaporate the solution to crystallization and cool.
7. Then place the beaker in an ice bath to complete crystallization.
8. **Filter off** the crystals from the mother liquor.
9. **Wash** the crystals twice with small portions of mother liquor.
10. **Dry** the crystals using absorbent paper.
11. **Weigh** the product and determine the yield.
12. Store the crystal in sample bottle.

Calculation:

1. Calculate the no. of moles of iron you have taken in this experiment.
2. Calculate the no, of moles of iron (**ii**) sulphate that you have obtained in this experiment.

Questions:

3. What happens when iron (**ii**) sulphate is kept in contact with air?
4. What type of reaction takes place during the conversion of iron to ferrous sulphate?
5. Write down the electronic configuration of iron (**ii**) and iron (**iii**).

Experiment No. 13

Name of the Experiment: Determination of Na_2CO_3 content in commercial Soda.

Theory:

Sodium carbonate (Na_2CO_3) content in commercial caustic soda solution can be determined by titrimetric method. The alkali carbonate ($CO^{2-}{}_3$) of the supplied solution can be determined by titrating with a standard acid solution. Then the carbonate ion in the solution may be precipitated by a dilute solution of $BaCl_2$.

Reaction: $$Na_2CO_3\,(aq) + 2HCl\,(aq) \longrightarrow 2NaCl\,(aq) + CO_2\,(g) + H_2O\,(l)$$

Procedure:

1. Weigh out accurately **1.44** g of washing soda in a **100 mL** volumetric flask.
2. Dissolve with a little water and make up to mark.
3. Take **10 mL** of the solution with a pipette in a **250 mL** conical flask.
4. Add about **25 mL** of water.
5. Add **1-2 drops of methyl orange** indicator to the solution.
6. Titrate the solution with standard hydrochloric acid solution **(**standardized as per exp: 02**)** until the color changes from **yellow to orange**.
7. Repeat the procedure at three at least three times and note the mean value.

Calculation:

1 ml N HCl = 0.05300 g Na_2CO_3

$$\text{\% of } Na_2CO_3 \text{ in washing soda} = \frac{\mathbf{0.05300\ g \times a \times b \times c}}{\mathbf{P \times Q}} \times \mathbf{100}$$

Where **a =** volume of HCl (mL) solution.
b = Molarity of HCl solution.
c = Total volume of solution (100 mL).
P = Volume taken for titration (10 mL).
Q = Weight of soda in g.

Report writing

- ❖ Report on each experiment should be submitted on the next class.
- ❖ Report should be submitted separately for each experiment.
- ❖ A4 size off set paper should be used. Writing must be on one side.

Top sheet of the report must contain the following points

Ahsanullah University of Science and Technology
Chemistry sessional report
Course number

Experiment no, Name of the experiment, Date of Performance, Date of Submission, Performer's Name, Department, Program, ID, Group no.

The top sheet may be composed but the rest of the report must be handwritten.

►The report should contain the following:

- ❖ Name of the experiment
- ❖ Objective of the experiment
- ❖ Theory (related to the experiment, equation, half-reactions etc.)
- ❖ Apparatus required
- ❖ Chemicals required
- ❖ Preparation of standard solution (passive form to be used) with calculation.
- ❖ Chemical reaction
- ❖ Experimental data table with the title.
- ❖ Calculation (both in formula and unitary method)
- ❖ Result
- ❖ Answers to the questions.

References:

1. Essentials of Physical Chemistry – by Bahl & Tuli
2. General Chemistry – by Ebbing
3. Modern Inorganic Chemistry – by S. Z. Haider
4. Vogel's Text Book of Quantitative Chemical Analysis (Pearson Education Asia)
5. Analytical Chemistry – by Gary D. Christian (John Wiley & Sons Inc.)