**STANDARDIZATION OF SODIUM HYDROXIDE (NaOH) SOLUTION WITH STANDARD OXALIC ACID ($C_2H_2O_4.2H_2O$) SOLUTION.**

PURPOSE
The strength of supplied NaOH solution (NaOH, being a secondary standard substance) will be measured with standard oxalic acid solution ($C_2H_2O_4.2H_2O$, being a primary standard substance) by acid-base titration.

INTRODUCTION
NaOH is a secondary standard substance, a strong base, white solid (mol. wt. 40, gram-equiv. wt. 40) and colorless in water solution. $C_2H_2O_4.2H_2O$ is a primary standard substance, a weak acid, white solid (mol. wt. 126, gram-equiv. wt. 63) and colorless in water solution. The balanced reaction between NaOH and $C_2H_2O_4.2H_2O$ is as follows:

$$HO_2C\text{-}CO_2H + 2NaOH = NaO_2C\text{-}CO_2Na + 2H_2O$$
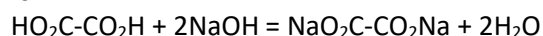
Since this reaction is a combination of weak acid and strong base, the choice of indicator is phenolphthalein ($C_{20}H_{14}O_4$). It is a white solid and is dissolved in 50% aqueous ethanol for use during titration.

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

REQUIRED CHEMICALS
Sodium hydroxide (NaOH), Oxalic acid ($C_2H_2O_4.2H_2O$) and Phenolphthalein ($C_{20}H_{14}O_4$).

PROCEDURE
1. **Preparation of standard solution**: Transfer approx. 0.63 gram of pure oxalic acid (HOOC-COOH.$2H_2O$) in a 100 ml measuring/volumetric flask and then dissolve it with distilled water up to the mark. Normality of the standard acid solution will be calculated as follows:

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

2. Take 10 mL of NaOH solution in a conical flask by means of a pipette and dilute it to about 50 ml.
3. Add 1-2 drops of phenolphthalein indicator to the solution.
4. Then add standard oxalic acid solution drop by drop from a burette.
5. Shake the flask frequently while adding the acid solution.
6. Stop the addition of oxalic acid solution as soon as the pink color of the solution just disappears.
7. 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.
8. The process should be repeated at least thrice. Take the mean of the readings.
9. Calculate the strength of the supplied NaOH solution using the formulae:

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

where V_{NaOH} = 10 mL, $V_{Oxalic\ acid}$ = the mean of burette reading, $N_{Oxalic\ acid}$ = strength of oxalic acid and N_{NaOH} = strength of NaOH to be calculated.

----<End of Exp.1>----

EXPERIMENT #2
STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM HYDROXIDE (NaOH) SOLUTION.

PURPOSE
The strength of supplied HCl solution (HCl, being a secondary standard substance) will be measured with standard NaOH solution (NaOH, being a secondary standard substance and standardized by standard $C_2H_2O_4.2H_2O$ solution, as Exp.1) by acid-base titration.

INTRODUCTION
HCl is a secondary standard substance, a strong acid, colorless gas (mol. wt. 36.5, gram-equiv. wt. 36.5) and available in water solution. NaOH is a secondary standard substance, a strong base, white solid (mol. wt. 40, gram-equiv. wt. 40) and colorless in water solution. $C_2H_2O_4.2H_2O$ is a primary standard substance, a weak acid, white solid (mol. wt. 126, gram-equiv. wt. 63) and colorless in water solution. The balanced reactions between NaOH and $C_2H_2O_4.2H_2O$; NaOH and HCl are as follows:
$$HO_2C\text{-}CO_2H + 2NaOH = NaO_2C\text{-}CO_2Na + 2H_2O$$
$$NaOH + HCl = NaCl + H_2O$$
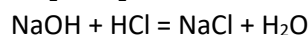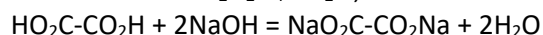
Since first reaction is a combination of weak acid and strong base, the choice of indicator is phenolphthalein ($C_{20}H_{14}O_4$). It is a white solid and is dissolved in 50% aqueous ethanol for use during titration. Second reaction is a combination of strong acid and strong base, so the choice of indicator is methyl orange ($C_{20}H_{14}O_4$). It is a red solid and is dissolved in water for use during titration.

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

REQUIRED CHEMICALS
Sodium hydroxide (NaOH), Oxalic acid ($C_2H_2O_4.2H_2O$), Hydrochloric acid (HCl), Phenolphthalein ($C_{20}H_{14}O_4$) and Methyl orange ($C_{20}H_{14}O_4$).

PROCEDURE
1. **Preparation of standard solution**: Transfer approx. 0.63 gram of pure oxalic acid (HOOC-COOH.2H$_2$O) in a 100 ml measuring/volumetric flask and then dissolve it with distilled water up to the mark. Normality of the standard acid solution will be calculated as follows:

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

2. Calculate the strength of the supplied NaOH solution using the formulae as Exp.1:

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

where V_{NaOH} = 10 mL, $V_{Oxalic\ acid}$ = the mean of burette reading, $N_{Oxalic\ acid}$ = strength of oxalic acid and N_{NaOH} = strength of NaOH to be calculated.

3. **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.
4. Take 10 mL of NaOH solution in a conical flask by means of a pipette and dilute it to about 50 mL.
5. Add 2-3 drops of methyl orange indicator to the solution and solution just changes to yellow.
6. Then add previously prepared (approx. 0.1N) HCl acid solution drop wise from a burette.
7. Shake the flask frequently during addition of HCl acid.
8. Stop the addition of HCl acid solution as soon as the yellow color of the solution just changes to pink.
9. Note the burette reading. Repeat the process at least twice and take the mean of the readings.
10. Calculate the strength of the dilute HCl solution and from there calculate the strength of commercial HCl using the following formulae:
    (A) *The strength of supplied dil. HCl solution*:

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

    (B) *The strength of conc. HCl solution*:

$$V_{dil.\ HCl} \times N_{dil.\ HCl\ determined} = V_{conc.\ HCl\ taken} \times N_{conc.\ HCl\ to\ be\ determined}$$
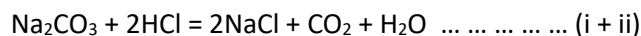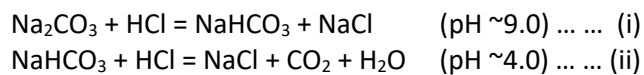
----<End of Exp.2>----

EXPERIMENT #3
STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM CARBONATE (Na$_2$CO$_3$) SOLUTION.

PURPOSE
The strength of supplied HCl solution (HCl, being a secondary standard substance) will be measured with standard Na_2CO_3 solution (Na_2CO_3, being a primary standard substance) by acid-base titration.

INTRODUCTION
HCl is a secondary standard substance, a strong acid, colorless gas (mol. wt. 36.5, gram-equiv. wt. 36.5) and available in water solution. Na_2CO_3 is a primary standard substance, a weak base, white solid (mol. wt. 106, gram-equiv. wt. 53) and colorless in water solution. The balanced reaction between Na_2CO_3 and HCl is as follows:

$$Na_2CO_3 + HCl = NaHCO_3 + NaCl \qquad (pH\ \text{~}9.0) \dots \dots\ (i)$$
$$NaHCO_3 + HCl = NaCl + CO_2 + H_2O \quad (pH\ \text{~}4.0) \dots \dots (ii)$$

$$Na_2CO_3 + 2HCl = 2NaCl + CO_2 + H_2O \ \dots \dots \dots \dots \dots (i + ii)$$

In the first reaction one mole Na_2CO_3 is reacted with one mole of HCl to produce $NaHCO_3$ and NaCl with a pH range ~9.0. So, the choice of indicator is phenolphthalein ($C_{20}H_{14}O_4$) since it works within pH 8.3-10. In the second reaction one mole $NaHCO_3$ is reacted with another mole of HCl to produce NaCl, CO_2 and H_2O with a pH range ~4.0. So, the choice of indicator is methyl orange ($C_{20}H_{14}O_4$) since it works within pH

3.1-4.4. Phenolphthalein is a white solid and is dissolved in 50% aqueous ethanol for use during titration. Methyl orange is a red solid and is dissolved in water for use during titration.

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

REQUIRED CHEMICALS
Sodium carbonate (Na_2CO_3), Hydrochloric acid (HCl), Phenolphthalein ($C_{20}H_{14}O_4$) and Methyl orange ($C_{20}H_{14}O_4$).

PROCEDURE

1. **Preparation of standard 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.

   The strength of sodium carbonate solution = $\dfrac{Weight\ taken\,(in\ gm) \times 0.1}{0.53}$ (N)

2. **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.
3. Take 10 mL of Na_2CO_3 solution in a conical flask and dilute it to about 50 mL.
4. Add 1-2 drops of phenolphthalein and titrate against dilute HCl solution contained in a burette.
5. Now note the burette reading when just one drop of HCl discharges the pink color of the solution. This is the first end point.
6. 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.
7. The difference of the burette reading from initial to second end point will be the volume of the acid required for titration.
8. Repeat the whole experiment 2-3 times and take the mean reading initial to second end point.
9. *Take last reading without using phenolphthalein*.
10. Calculate the strength of the dilute HCl solution and from there calculate the strength of commercial HCl using the following formulae:

    (A) *The 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) *The strength of conc. HCl solution*:
    $$V_{dil.\ HCl}\ \text{x}\ N_{dil.\ HCl\ determined}\ =\ V_{conc.\ HCl\ taken}\ \text{x}\ N_{conc.\ HCl\ \text{to be determined}}$$

----<End of Exp.3>----

EXPERIMENT #4
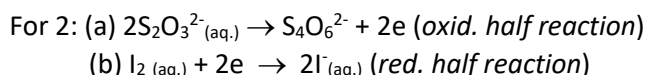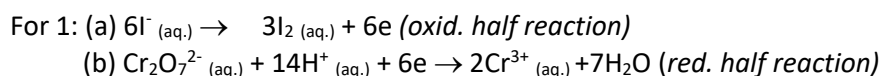**STANDARDIZATION OF SODIUM THIOSULPHATE ($Na_2S_2O_3$) SOLUTION WITH STANDARD POTASSIUM DICHROMATE ($K_2Cr_2O_7$) SOLUTION.**

PURPOSE

The strength of $Na_2S_2O_3$ solution (being a solution made from secondary standard substance) will be measured against standard $K_2Cr_2O_7$ solution by iodometric oxidation-reduction titration.

INTRODUCTION

$Na_2S_2O_3.5H_2O$ is a secondary standard substance, a reducing agent, white solid (mol. wt. 248, gram-equiv. wt. 248) and colorless in water solution. $K_2Cr_2O_7$ is a primary standard substance, an oxidizing agent, orange solid (mol. wt. 294, gram-equiv. wt. 49) and orange color in water solution. The balanced reactions of $K_2Cr_2O_7$ in presence of HCl and KI (white solid, a reducing agent) and of $Na_2S_2O_3$ with I_2 are as follows:

1. $K_2Cr_2O_7 + 14HCl + 6KI = 8KCl + 2CrCl_3 + 7H_2O + 3I_2$
2. $2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI$

Redox Half Reactions

For 1: (a) $6I^-_{(aq.)} \rightarrow 3I_{2\ (aq.)} + 6e$ (*oxid. half reaction*)
(b) $Cr_2O_7^{2-}{}_{(aq.)} + 14H^+_{(aq.)} + 6e \rightarrow 2Cr^{3+}_{(aq.)} + 7H_2O$ (*red. half reaction*)

For 2: (a) $2S_2O_3^{2-}{}_{(aq.)} \rightarrow S_4O_6^{2-} + 2e$ (*oxid. half reaction*)
(b) $I_{2\ (aq.)} + 2e \rightarrow 2I^-_{(aq.)}$ (*red. half reaction*)

The **Iodometric titration** is a general method to determine the concentration of an oxidizing agent in solution. In an iodometric titration, a starch solution is used as an indicator since it can absorb the I_2 that is released.

Starch solution is used in this titration involving iodine because it forms an intense blue complex with even a trace of iodine. But starch is not a redox indicator; it responds specifically to the presence of I_2, not to a change in redox potential. The active fraction of starch is amylose, a polymer of the sugar α-d-glucose. In the presence of starch, iodine forms I_5^- chains inside the amylose helix and the color turns dark blue.

__NaHCO₃__ (white solid) is used to remove excess of HCl acid in the solution and to create a layer of CO₂ in the conical flask to reduce loss of volatile I₂.

Solution turns deep brown when I_2 (brown solid) is produced during first reaction above. With addition of $Na_2S_2O_3$ solution it turns light yellow due to presence of trace of I_2. Starch forms intense blue color complexes with trace of iodine. With further addition of $Na_2S_2O_3$ solution iodine is completely consumed, end point is achieved, and solution turns light green at the end point due to presence of chromic ions (Cr^{3+}).

APPARATUS

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

REQUIRED CHEMICALS

Potassium iodide (KI), Sodium bicarbonate ($NaHCO_3$), Conc. Hydrochloric acid (HCl), Potassium dichromate ($K_2Cr_2O_7$), Sodium thiosulphate ($Na_2S_2O_3$) and Starch ($C_6H_{10}O_5$)$_n$.

PROCEDURE

1. **Preparation of approx. 0.1N potassium dichromate solution:** Transfer approx. 0.49 gram of pure $K_2Cr_2O_7$ into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

   The strength of $K_2Cr_2O_7$ solution = $\dfrac{Weight\ taken\,(in\ gm) \times 0.1}{0.49}$ *(N)*

2. Take 4 mL of 12% KI solution in a conical flask and dilute to about 50 mL.
3. Add about one gram of $NaHCO_3$ and shake the flask until the salt dissolves.
4. Add 4 mL conc. HCl acid and then add 10 ml standard $K_2Cr_2O_7$ 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).
5. Rinse the watch glass and dilute the solution about 100mL.
6. Titrate the liberated iodine with sodium thiosulphate solution from a burette until the brown color fades (light yellow).
7. 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.
8. Note the burette reading, repeat the whole experiment 2-3 times and take the mean.
9. Calculate the strength of sodium thiosulphate solution using the following formulae:

   The strength of supplied $Na_2S_2O_3$ solution:
   $$V_{Na2S2O3} \times N_{Na2S2O3} = V_{K2Cr2O7} \times N_{K2Cr2O7}$$

----<End of Exp.4>----

EXPERIMENT #5
ESTIMATION OF COPPER (Cu) CONTAINED IN A SUPPLIED SOLUTION OF COPPER SALT BY IODOMETRIC METHOD.

PURPOSE
The amount of copper ions in a supplied solution of copper salt will be measured by iodometric oxidation-reduction titration.
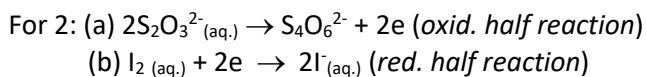
INTRODUCTION
$K_2Cr_2O_7$ is a primary standard substance, an oxidizing agent, orange solid (mol. wt. 294, gram-equiv. wt. 49) and orange color in water solution. $Na_2S_2O_3.5H_2O$ is a secondary standard substance, a reducing agent, white solid (mol. wt. 248, gram-equiv. wt. 248) and colorless in water solution. $CuSO_4.5H_2O$ is an inorganic compound (also called **blue vitriol**, an oxidizing agent, blue colored, mol. wt. 249.68) and blue in water solution. The balanced reactions of $CuSO_4$ in presence of NH_4OH, CH_3COOH and KI (white solid, a reducing agent) and of $Na_2S_2O_3$ with I_2 are as follows:
   1. $2CuSO_4 + 4KI = 2K_2SO_4 + 2CuI \downarrow + I_2$
   2. $2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI$

Redox Half Reactions
   For 1: (a) $2I^-_{(aq.)} \rightarrow I_{2\,(aq.)} + 2e$ *(oxid. half reaction)*
   (b) $2Cu^{2+}_{(aq.)} + 2e \rightarrow 2Cu^+_{(aq.)}$ *(red. half reaction)*

For 2: (a) $2S_2O_3^{2-}{}_{(aq.)} \rightarrow S_4O_6^{2-} + 2e$ (*oxid. half reaction*)

(b) $I_2{}_{(aq.)} + 2e \rightarrow 2I^-{}_{(aq.)}$ (*red. half reaction*)

- 6M NH_4OH: 4 drops for each reading; *to adjust the acidity or to keep high pH in the solution*.
- CH_3COOH: 4-5 drops for each reading; *to remove or dissolve the ppt by adjusting acidity*.
- 10% NH_4SCN: *to separate the adsorbed iodine from ppt (CuI solid).*

Starch solution is used in this titration involving iodine because it forms an intense blue complex with even a trace of iodine. But starch is not a redox indicator; it responds specifically to the presence of I_2, not to a change in redox potential. The active fraction of starch is amylose, a polymer of the sugar α-d-glucose. In the presence of starch, iodine forms I_5^- chains inside the amylose helix and the color turns dark blue.

Solution turns brown when I_2 (brown solid) is produced during first reaction above. With addition of $Na_2S_2O_3$ solution it turns light yellow due to presence of trace of I_2. Starch forms intense blue color complexes with trace of iodine. With further addition of $Na_2S_2O_3$ solution iodine is completely consumed, end point is achieved, and solution turns off white at the end point due to presence of insoluble CuI (white color solid).

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

REQUIRED CHEMICALS
Potassium iodide (KI), Sodium bicarbonate ($NaHCO_3$), Conc. Hydrochloric acid (HCl), Potassium dichromate ($K_2Cr_2O_7$), Sodium thiosulphate ($Na_2S_2O_3.5H_2O$), Starch ($C_6H_{10}O_5)_n$, Copper sulphate ($CuSO_4.5H_2O$), Ammonium hydroxide (NH_4OH, 6M), Conc. Acetic acid (CH_3COOH) and Ammonium thiocyanate (NH_4SCN).

PROCEDURE

1. **Preparation of approx. 0.1N potassium dichromate solution:** Transfer approx. 0.49 gram of pure $K_2Cr_2O_7$ into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

   The strength of $K_2Cr_2O_7$ solution = $\dfrac{Weight\,taken\,(in\ gm) \times 0.1}{0.49}$ (N)

2. **Standardization of supplied $Na_2S_2O_3$ solution:** Calculate the strength of sodium thiosulphate solution using the following formulae as Exp.4:

   The strength of supplied $Na_2S_2O_3$ solution:

   $$V_{Na2S2O3} \times N_{Na2S2O3} = V_{K2Cr2O7} \times N_{K2Cr2O7}$$

3. Pipette out 10 mL of copper salt solution (containing 4 gm blue vitriol in 500 mL) into a conical flask.

4. Add 3-4 drops of 6M NH_4OH until a faint permanent ppt remain and then add 6-8 drops of conc. CH_3COOH.
5. 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.
6. Add approx. 1 mL of starch solution, solution turns intense blue and continue titration till the blue color begins to fade.
7. Now add few drops of 10% ammonium thiocyanate solution and continue titration until the blue color is just discharged (off-white).
8. Note the burette reading. Repeat the process at least twice and take the mean of the readings.
9. Calculate the amount of copper present in 500 mL of copper salt solution using the following formula (also observed value of Cu in 500 mL solution):

$$2\ CuSO_4\ \equiv\ I_2\ \equiv\ 2\ Na_2S_2O_3$$
$$1\ ml\ 1N\ Na_2S_2O_3\ \equiv\ 0.06354\ gm\ of\ Cu^{2+}$$
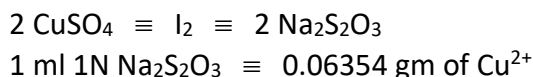
10. Also calculate percentage of error by calculating known value of Cu in the supplied Cu-salt solution.
   (a) Known value of Cu in 500 mL Cu-salt solution = (mass of Cu × amount Cu-salt taken) ÷ mol. wt. of Cu-salt = (63.54 × amount Cu-salt taken) ÷ 249.68

   (b) % of error =

$$\frac{known\ value - observed\ value}{known\ value} \times 100$$
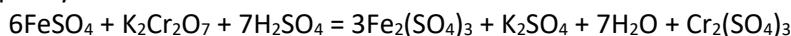
----<End of Exp.5>----

EXPERIMENT #6
DETERMINATION OF FERROUS ION (Fe^{2+}) IN A SUPPLIED SOLUTION OF IRON SALT BY STANDARD POTASSIUM DICHROMATE ($K_2Cr_2O_7$) SOLUTION.
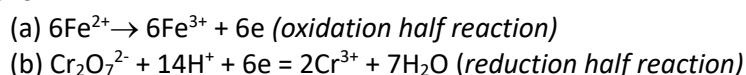
PURPOSE
The amount of ferrous ions (iron) in a supplied solution of Mohr's salt (iron salt) will be measured by standard $K_2Cr_2O_7$ solution.

INTRODUCTION
$K_2Cr_2O_7$ is a primary standard substance, an oxidizing agent, orange solid (mol. wt. 294, gram-equiv. wt. 49) and orange color in water solution. Iron salt, ferrous ammonium sulphate, $FeSO_4.(NH_4)_2SO_4.6H_2O$ is an inorganic compound (also called **Mohr's salt**, a reducing agent, blue-green solid, mol. wt. 392.14) and blue-green in water solution. The balanced reactions of $FeSO_4$ with $K_2Cr_2O_7$ in presence of 5% H_2SO_4 and conc. H_3PO_4 and diphenyl amine:

$$6FeSO_4 + K_2Cr_2O_7 + 7H_2SO_4 = 3Fe_2(SO_4)_3 + K_2SO_4 + 7H_2O + Cr_2(SO_4)_3$$

Redox Half Reactions

(a) $6Fe^{2+} \rightarrow 6Fe^{3+} + 6e$ *(oxidation half reaction)*
(b) $Cr_2O_7^{2-} + 14H^+ + 6e = 2Cr^{3+} + 7H_2O$ *(reduction half reaction)*

- Conc. H_3PO_4: It is used to reduce the oxidation potential by forming complexes with Fe^{+3} ions produced in the reaction.
- Diphenylamine: It is used as a redox indicator because it shows a very clear color change from green to violet when end point of the titration is reached, 1 gm solid in 100 mL conc. H_2SO_4
- 5% H_2SO_4: It is the main source of H_3O^+ which supply proton to remove oxygen, such as those in the dichromate ions.

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

REQUIRED CHEMICALS
Mohr's salt [$FeSO_4.(NH_4)_2SO_4.6H_2O$], Sulphuric acid ($H_2SO_4$, 5%), conc. Phosphoric acid (H_3PO_4), Potassium dichromate ($K_2Cr_2O_7$) and Diphenylamine [$(C_6H_5)_2NH$].

PROCEDURE

1. **Preparation of approx. 0.1N potassium dichromate solution:** Transfer approx. 0.49 gram of pure $K_2Cr_2O_7$ into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

   The strength of $K_2Cr_2O_7$ solution = $\dfrac{Weight\ taken\ (in\ gm) \times 0.1}{0.49}$ (N)

2. Take 10 mL of the supplied iron salt (Mohr's salt) solution in a conical flask.
3. Add 50 mL 5% sulfuric acid and 5 mL of conc. phosphoric acid.
4. 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.
5. Repeat the experiment at least thrice. Calculate the amount of iron per 500 mL of iron salt solution. Calculate the amount of iron present in 500 mL of iron salt solution using the following formula (also observed value of Fe in 500 mL solution):

   1 mL 1N $K_2Cr_2O_7$ ≡ 0.05584 gm of Fe^{2+}

6. Also calculate percentage of error by calculating known value of Fe in the supplied iron-salt solution.
   (a) Known value of Fe in 500 mL iron-salt solution = (mass of Fe × amount iron-salt taken) ÷ mol. wt. of iron-salt = (55.84 × amount iron-salt taken) ÷ 392.14

   (b) % of error =

   $$\dfrac{known\ value - observed\ value}{known\ value} \times 100$$

----<End of Exp.6>----

EXPERIMENT #7

DETERMINATION OF FERROUS ION (Fe^{2+}) IN A SUPPLIED SOLUTION OF IRON SALT BY STANDARD POTASSIUM PERMANGANATE (KMnO$_4$) SOLUTION.

PURPOSE

The amount of ferrous ions (iron) in a supplied solution of Mohr's salt (iron salt) will be measured by standard KMnO$_4$ solution.

INTRODUCTION

KMnO$_4$ is a secondary standard substance, a strong oxidizing agent, pink solid (mol. wt. 158, gram-equiv. wt. 31.6) and pink color in water solution. Na$_2$C$_2$O$_4$ is a primary standard substance, a reducing agent, white solid (mol. wt. 134, gram-equiv. wt. 67) and colorless in water solution. Iron salt, ferrous ammonium sulphate, FeSO$_4$.(NH$_4$)$_2$SO$_4$.6H$_2$O is an inorganic compound (also called **Mohr's salt**, a reducing agent, blue-green solid, mol. wt. 392.14) and blue-green in water solution. The balanced reactions of KMnO$_4$ with Na$_2$C$_2$O$_4$ and FeSO$_4$ separately in presence of H$_2$SO$_4$ are as follows:

      1.   2KMnO$_4$ + 5Na$_2$C$_2$O$_4$ + 8H$_2$SO$_4$ = 2MnSO$_4$ + 10CO$_2$ + 8H$_2$O (+ K$_2$SO$_4$ + 5Na$_2$SO$_4$)

      2.   2KMnO$_4$ + 10FeSO$_4$ + 8H$_2$SO$_4$ = 2MnSO$_4$ + 5Fe$_2$(SO$_4$)$_3$ + K$_2$SO$_4$ + 8H$_2$O

Redox Half Reactions

      For 1: (a) 5C$_2$O$_4^{2-}$ → 10CO$_2$ + 10e (Oxidation)

            (b) 2MnO$_4^-$ + 16H$^+$ + 10e = 2Mn^{++} + 8H$_2$O (Reduction)

      For 2: (a) 10Fe^{2+} → 10Fe^{3+} + 10e (Oxidation)

            (b) 2MnO$_4^-$ + 16H$^+$ + 10e = 2Mn^{++} + 8H$_2$O (Reduction)

APPARATUS

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

REQUIRED CHEMICALS

Potassium permanganate (KMnO$_4$), Sodium oxalate (Na$_2$C$_2$O$_4$), Sulphuric acid (H$_2$SO$_4$, 2N) and Mohr's salt [FeSO$_4$.(NH$_4$)$_2$SO$_4$.6H$_2$O].

PROCEDURE

1. **Preparation of approx. 0.1N sodium oxalate solution:** Transfer approx. 0.67 gram of pure Na$_2$C$_2$O$_4$ into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

    The strength of Na$_2$C$_2$O$_4$ solution = $\dfrac{Weight\,taken\,(in\ gm) \times 0.1}{0.67}$ *(N)*

2. **Standardization of supplied KMnO$_4$ 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. Calculate the strength of potassium permanganate solution using the following formulae:

The strength of supplied $KMnO_4$ solution: V_{KMnO4} x N_{KMnO4} **(S)**= $V_{Na-oxalate}$ x $N_{Na-oxalate}$

3. 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.
4. 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 (2).
5. Repeat the titration at least twice. Calculate the amount of iron per 500 mL of iron salt solution. Calculate the amount of iron present in 500 mL of iron salt solution using the following formula (also observed value of Fe in 500 mL solution):

    1 mL 1N $KMnO_4$ $\equiv$ 0.05584 gm of Fe^{2+}
6. Also calculate percentage of error by calculating known value of Fe in the supplied Fe-salt solution.
    (a) Known value of Fe in 500 mL iron-salt solution = (mass of Fe × amount iron-salt taken) ÷ mol. wt. of iron-salt = (55.84 × amount iron-salt taken) ÷ 392.14

    (b) % of error =

$$\frac{known\,value - observed\,value}{known\,value} \times 100$$

----<End of Exp.7>----

DETERMINATION OF STRENGTH OF A WEAK ACID (CH_3COOH) AGAINST A STRONG ALKALI (NaOH) SOLUTION BY MEASURING CONDUCTANCE.

PURPOSE
- 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 (acetic acid)

INTRODUCTION
NaOH is a secondary standard substance, a strong base, white solid (mol. wt. 40, gram-equiv. wt. 40) and colorless in water solution. CH_3COOH is also a secondary standard substance, a weak acid, colorless liquid (mol. wt. 60.05, gram-equiv. wt. 60.05) and colorless in water solution. The balanced reaction between NaOH and CH_3COOH is as follows:

$$CH_3COOH \quad \rightarrow CH_3COO^- + H^+$$
$$NaOH \qquad \rightarrow Na^+ + OH^-$$

$$CH_3COOH + NaOH = CH_3COONa + H_2O$$
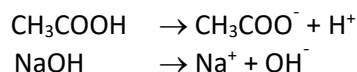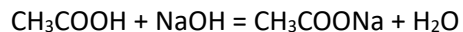
Conductometric Titration: The conductance of an acid solution varies with the amount of alkali solution added to it because of the change in the number and nature of the ions in the solution. The curve showing this variation in called the conductance titration curve. At the end-point (also called, neutralizing point) of acid-alkali reaction, there is a sharp change in the conductivity. Therefore, if the conductance titration curve between volume of alkali vs conductance 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), watch glass, pipette filler, dropper, stand and clamp etc.

REQUIRED CHEMICALS
Sodium hydroxide (NaOH, 0.1N) and Acetic acid (CH_3COOH, approx. 0.1N).

PROCEDURE
1. 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.
2. Measure the conductance of the acid solution (1st reading).
3. 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.
4. 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.
5. Now calculate the strength of CH_3COOH solution following the formulae:
$$V_{CH3COOH} \ x \ N_{CH3COOH} \ = \ V_{NaOH} \ x \ N_{NaOH}$$
where V_{NaOH} = from graph (ml), $V_{Oxalic \ acid}$ = 10 ml, $N_{Oxalic \ acid}$ = to be determined and N_{NaOH} = 0.1N.

----<End of Exp.8>----