DETERMINATION OF STRENGTH OF $KMnO_4$ SOLUTION BY TITRATING IT WITH STANDARD FERROUS AMMONIUM SULPHATE SOLUTION

PRINCIPLE

Potassium permanganate in H_2SO_4 medium oxidises Fe^{2+} to Fe^{3+}.

$$MnO_4^- + 8H^+ + 5e \rightleftharpoons Mn^{2+} + 4H_2O \quad \text{---- [1]}$$

$$Fe^{2+} - e \rightleftharpoons Fe^{3+} \quad \text{---- [2]}$$

Equation (2) is multiplied by 5 and then added to equation (1),

$$MnO_4^- + 5Fe^{2+} + 8H^+ \longrightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O \quad \text{---- [3]}$$

Molecular equation (taking Mohr's Salt)

$$2KMnO_4 + 10[FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O] + 8H_2SO_4 \downarrow$$

$$K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 10(NH_4)_2SO_4 + 68H_2O \quad \text{---- [4]}$$

$FeSO_4$ of Mohr's Salt takes part as a reducing agent in this reaction.

So, with help of standard Mohr's salt solution in 2 (N) H_2SO_4 medium in the presence of phosphoric acid, $KMnO_4$ solution can be titrated at ordinary temperature to determine the strength, g/L and molar strength of $KMnO_4$ solution.

If V_1 mL Mohr's salt of strength S_1 (N) requires V_2 mL $KMnO_4$ solution of strength S_2 (N) to reach the equivalence point, then $V_1S_1 = V_2S_2$.

or $S_2 = \frac{V_1S_1}{V_2}$.

Indicator: $KMnO_4$ solution is the self-indicator in this titration.

NECESSARY APPARATUS & CHEMICALS

Apparatus: 1) Burrette (50 mL), 2) Pipette (25 mL), 3) Stand and burette clamp, 4) Conical flask, 5) Wash bottle, 6) Measuring cylinder.

Chemicals: 1) Standard (N/10) Mohr's salt solution, 2) (~N/10) $KMnO_4$ solution of unknown strength, 3) 2 (N) H_2SO_4 solution, 4) Syrupy H_3PO_4 (or NH_4HF_2).

PROCEDURE

1) Burette, pipette and conical flask are washed first with first tap water and then with distilled water.

2) Burette is rinsed twice with 2-3 mL supplied $KMnO_4$ solution each time. The burette is filled with this $KMnO_4$ solution upto the '0' mark.

3) The pipette is rinsed with standard Mohr's salt solution. 25 mL Mohr's salt solution is transferred to 500 ml conical flask with a pipette. 15 ml of 2 (N) H_2SO_4 solution and 3ml syrupy H_3PO_4

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are added to the solution and the mixture is shaken well.

4) The conical flask containing Mohr's salt solution is placed on a piece of white paper below the burette. $KMnO_4$ solution from the burrete is added slowly with gentle shaking of the solution. The pink colour of $KMnO_4$ disappears as soon as it comes in contact with the solution. Titration is continued till one drop of $KMnO_4$ solution brings a faint pink colour that persists for 30 seconds. Addition of $KMnO_4$ is stopped. Burette reading is noted. This process is repeated twice.

Strength of standard Mohr's salt solution = 0.972 (N/10)

EXPERIMENTAL RESULT

No. of titration	Volume of Mohr's salt (ml)	Burrette reading for $KMnO_4$ solution		Volume of $KMnO_4$ solution required (ml)	Mean volume of $KMnO_4$ (ml)
		Initial (ml)	Final (ml)		
1	25	0	25.2	25.2	
2	25	0	25.1	25.1	25.1
3	25	0	25.1	25.1	

CALCULATION

$V_1 S_1 = V_2 S_2$ [V_1 = Vol. of mohr's salt = 25ml, S_1 = 0.972, V_2 = Vol of $KMnO_4$ = 25.1ml]

$\therefore S_2 = \frac{V_1 S_1}{V_2} = \frac{25 \times 0.972}{25.1 \times 10}$ (N) = 0.0968 (N)

Amount of $KMnO_4$ in $g.L^{-1}$ = $0.0968 \times 31.6 = 3.0588$

Molar strength = $\frac{3.0588}{158}$ (M) = 0.0193 (M)

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TEST FOR FUNCTIONAL GROUPS PRESENT IN ORGANIC COMPOUNDS

• Test for ALDEHYDE (-CHO) and KETO (>C=O) Groups :

EXPERIMENT	OBSERVATION	INFERENCE
• Preliminary test :-		
1) 2,4 -DNP test ⇒ 2-3 ml of 2,4- DNP. (Brady's reagent) is added to 2ml of alcoholic solution of the sample and shaken.	1) Red (or yellow) precipitate	1) Aldehyde (-CHO) or keto (>C=O) group may be present
• Distinctive tests for alhedydes and Ketones : * If the test (1) is positive the given tests are performed.		
a) In a test tube, about 0.1g (0.2ml) sample is added to a mixture of equal volumes of Fehling A and Fehling B solutions. The	a) Brick red precipitate (of Cu_2O).	a) Presence of aldehyde group is confirmed.

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EXPERIMENT	OBSERVATION	INFERENCE
reaction mixture is heated for 4-5 minutes in a boiling water bath.		
b) In a test tube, about 3 ml Tollen's reagent is shaken with a small amount of the sample. If the reaction mixture does not turn grey or black, the mixture is placed in a hot water bath for few minutes.	b) A shining mirror is formed on the inner surface of the test tube or a grey precipitate is formed.	b) Confirms the presence of aldehyde group (-CHO).

All compounds having aldehyde group (particularly aromatic aldehydes) do not respond to Fehling's test. Hence, Tollen's reagent is used in the detection of -CHO group.

- Test for Carboxylic Acid (-COOH) Group :

EXPERIMENT	OBSERVATION	INFERENCE
1) Test with $NaHCO_3$: A small amount of the sample is added to 5 ml saturated solutions of $NaHCO_3$, taken in a	1) Effervescence (due to liberation of CO_2).	1) Carboxylic acid group (-COOH) may be present.

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EXPERIMENT	OBSERVATION	INFERENCE
test tube.		
2) Esterification Test: About 0.5 g (or 0.5 ml) of the sample is taken in a test tube to which 2ml ethanol and 1 ml concentrated H_2SO_4 are added. The mixture is then heated in a boiling water bath. The mixture is poured into 10 ml Na_2CO_3 solution, taken in a beaker.	2) Sweet fruity smell.	2) Presence of carboxylic acid (-COOH) group is confirmed.

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VARIATION OF CELL POTENTIAL IN $Zn|Zn^{2+}||Cu^{2+}|Cu$ WITH CHANGE IN CONCENTRATION OF ELECTROLYTES ($CuSO_4/ZnSO_4$)

• Theory :

With increase in concentration of the electrolyte, the reduction potential of an electrode increases —

$$M^{n+}(aq) + ne \longrightarrow M(s)$$

In case of a Zn-Cu electrochemical cell, zinc electrode acts as the anode and copper electrode acts as the cathode.

EMF of the cell is - $E_{cell} = E_{cathode} - E_{anode}$

In this equation, it is found that E_{cell} increases with increase in $E_{cathode}$ and decrease in E_{anode}. So for the cell $Zn|Zn^{2+}||Cu^{2+}|Cu$, E_{cell} can be increased by using a higher concentration of Cu^{2+} and a lower concentration of Zn^{2+}.

The relation between the concentration of electrolytes and the standard electrode potential can be found from Nernst Equation :

$$E_{cell} = E^{\circ}_{cell} - \frac{0.059}{n} \log \frac{[M]}{[M^{n+}]} = E^{\circ}_{cell} + \frac{0.059}{n} \log [M^{n+}]$$

(Active mass of solid, $[M] = 1$; E° = standard electrode potential)

• Chemicals : i) 1 (M) $ZnSO_4$ solution, ii) 1 (M) $CuSO_4$ solution, iii) 0.1 (M) $ZnSO_4$ solution,

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iv) 0.1 (M) $CuSO_4$ solution, v) Clean Zn and Cu-rods.

⇒ Preparation of 0.1(M) solution: 25 ml of 1(M) solution is pipetted out and taken in a 250 ml volumetric flask. Remaining volume is made upto the mark with distilled water. Volumetric flask is shaken well to get a homogeneous solution.

● Procedures:

1) A beaker (500 ml) is half-filled with 1(M) $CuSO_4$ solution.

2) The Cu-rod is cleaned with a sand paper and suspended in the copper sulphate ($CuSO_4$) solution (partially dipped) from a wooden slab.

3) A porous pot is half-filled with 1(M) $ZnSO_4$ solution. A Zn rod is cleaned with sand paper and partially dipped in the $ZnSO_4$ solution. The pourous pot along with the Zn-rod is suspended from the wooden slab in the $CuSO_4$ solution in beaker.

4) The +ve end of the voltmeter with high internal resistance, is connected to the Cu-rod and the -ve end is connected to the Zn-rod by a conducting wire, with the help of a screw.

5) The readings of the voltmeter is taken when the fluctuation of its pointer becomes stable.

6) After this, the experiment is repeated taking 0.1(M) $ZnSO_4$ and 1(M) $CuSO_4$ solution; 1(M) $ZnSO_4$ and 0.1(M) $CuSO_4$; 0.1(M) $ZnSO_4$ and 0.1(M) $CuSO_4$ solutions respectively.

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Observations :-

No. of Observations	Concentration of $ZnSO_4$ solution	Concentration of $CuSO_4$ solution	EMF of the cell
1	1(M)	1(M)	
2	0.1(M)	1(M)	
3	1(M)	0.1(M)	
4	0.1(M)	0.1(M)	

Conclusion :

On decreasing the concentration of $ZnSO_4$ solution around the Zn-electrode, EMF of the cell increases. On the other hand, on increasing the concentration of $CuSO_4$ solution around the Cu-electrode, EMF of the cell increases.

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WET TEST OF BASIC RADICALS

EXPERIMENT	Observation	INFERENCE
LEAD (Pb^{2+}) radical		
In a test tube, a small amount of the solution is heated with potassium iodide solution.	Golden-yellow precipitate dissolves on heating. Golden sprangles (needle-shaped precipitate) appears on cooling.	$Pb(NO_3)_2 + 2KI \rightarrow$ $PbI_2 \downarrow$ (yellow) $+ 2KNO_3$ PbI_2 is soluble in hot water but insoluble in cold water.
Ca^{2+} ion		
About 2ml of the solution is taken in a test tube and made alkaline with NH_4OH to which ammonium oxalate solution is added.	Heavy white precipitate soluble in HCl but insoluble in acetic acid.	Precipitate of insoluble calcium oxalate, soluble in HCl. $CaCl_2 + (NH_4)_2C_2O_4 \rightarrow$ $CaC_2O_4 \downarrow + 2NH_4Cl$ $CaC_2O_4 + 2HCl \rightarrow$ $CaCl_2 + H_2C_2O_4$

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EXPERIMENT	OBSERVATION	INFERENCE
STRONTIUM (Sr^{2+}) radical		
About 0.2g $SrCl_2$ is dissolved in 10 ml. distilled water and the following tests are perform-ed with the solution. 2ml solution is taken in a test tube and make alkaline with NH_4OH solution. The solution is then heated with $(NH_4)_2SO_4$ solution.	White precipitate.	White precipitate is of strontium sulphate, which is solution in dilute HCl. $SrCl_2 + (NH_4)_2SO_4 \rightarrow SrSO_4 \downarrow + 2NH_4Cl$ $SrSO_4 + 2HCl \rightarrow SrCl_2 + H_2SO_4$
BARIUM (Ba^{2+}) radical		
About 0.2g $BaCl_2$ is dissolved in 10 ml distilled water. $(NH_4)_2SO_4$ solution is added to 2ml solution taken in a test tube.	White precipitate insoluble in HCl	White precipitate is of barium sulphate. $BaCl_2 + (NH_4)_2SO_4 \rightarrow BaSO_4 \downarrow + 2NH_4Cl$
MAGNESIUM (Mg^{2+}) radical		
In a 100 ml beaker, 0.2g of magnesium sulphate ($MgSO_4.7H_2O$)		

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EXPERIMENT	OBSERVATION	INFERENCE
is dissolved in 10 ml distilled water.		
a) 2ml of the solution is taken and made alkaline with a solution of NaOH or NH_4OH.	a) White precipitate	a) White precipitate is of insoluble magnesium hydroxide. $MgSO_4 + 2NaOH \rightarrow Mg(OH)_2 + Na_2SO_4$
b) The ppt obtained shaken with NH_4Cl solution.	b) The precipitate dissolves in NH_4Cl solution.	b) The precipitate dissolves due to the formation of soluble $MgCl_2$. $Mg(OH)_2 + 2NH_4Cl \rightarrow MgCl_2 + 2NH_4OH$

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WET TEST OF ACID RADICALS

EXPERIMENT	OBSERVATION	INFERENCE
	CO_3^{2-} radical	
a) About 2ml of Na_2CO_3 solution is taken in a test tube and $BaCl_2$ solution is added.	a) White precipitate	a) The white precipitate formed is of insoluble $BaCO_3$. $Na_2CO_3 + BaCl_2 \rightarrow BaCO_3\downarrow + 2NaCl$
b) Dilute HCl is added to the precipitate.	b) The precipitate dissolves in dilute HCl.	b) $BaCO_3$ reacts with HCl to give soluble $BaCl_2$. $BaCO_3 + 2HCl \rightarrow BaCl_2 + CO_2\uparrow + H_2O$
	S^{2-} radical	
a) $AgNO_3$ solution is added to 2ml aqueous ammonium sulphide solution.	a) Black precipitate	a) The black ppt. is of silver sulphide. $Na_2S + 2AgNO_3 \rightarrow Ag_2S\downarrow + 2NaNO_3$
b) The ppt. is heated with dilute HNO_3.	b) The ppt dissolves in HNO_3.	b) The ppt. gives soluble silver nitrate. $Ag_2S + 2HNO_3 \rightarrow 2AgNO_3 + H_2S\uparrow$

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EXPERIMENT	OBSERVATION	INFERENCE
Cl^- radical		
1) To about 2ml sample solution, dilute HNO_3 and $AgNO_3$ solution are added. The sample is aq. NaCl.	Curdy white ppt. is obtained.	The curdy white ppt is of insoluble AgCl. $NaCl + AgNO_3 \rightarrow AgCl\downarrow + NaNO_3$
a) A part of the ppt. is shaken with HNO_3	a) Ppt. does not dissolve.	a) AgCl is insoluble in HNO_3.
b) The other part of the ppt. is taken in a test tube and shaken with NH_4OH solution.	b) Precipitate dissolves.	b) Due to formation of soluble complex of argento-ammonium chloride. $AgCl + 2NH_4OH \rightarrow [Ag(NH_3)_2]Cl + 2H_2O$
NO_3^- radicals		
In a test tube, 2ml of the zinc nitrate $[Zn(NO_3)_2]$ solution is mixed with equal volume of freshly prepared acidified ferrous sulphate solution. The test tube is held inclined to which, conc. H_2SO_4	Brown ring formed at the junction of two liquids.	HNO_3 is reduced by ferrous sulphate to nitric oxide (NO). NO combines with $FeSO_4$ to give a brown complex of nitrosoferrous sulphate-pentahydrate. $Zn(NO_3)_2 + H_2SO_4 \rightarrow 2HNO_3 + ZnSO_4$

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EXPERIMENT	OBSERVATION	INFERENCE
is poured carefully from the side of test tube. The mix. is cooled under tap water.		$6FeSO_4 + 2HNO_3 + 3H_2SO_4 \rightarrow 3Fe_2(SO_4)_3 + 2NO + 4H_2O$ $FeSO_4 + NO + 5H_2O \rightarrow [Fe(H_2O)_5NO]SO_4$ (Brown coloured complex)
	SO_4^{2-} radical	
About 2ml aq. $MgSO_4.7H_2O$ solution is taken in a test tube to which barium chloride solution is added.	White ppt. insoluble in conc. HCl.	The white ppt. is of insoluble $BaSO_4$. $MgSO_4 + BaCl_2 \rightarrow BaSO_4\downarrow + MgCl_2$ (white)

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EXPERIMENT	OBSERVATION	INFERENCE
is poured carefully from the side of test tube. The mix. is cooled under tap water.		$6FeSO_4 + 2HNO_3 + 3H_2SO_4 \rightarrow 3Fe_2(SO_4)_3 + 2NO + 4H_2O$ $FeSO_4 + NO + 5H_2O \rightarrow [Fe(H_2O)_5NO]SO_4$ (Brown coloured complex)
	SO_4^{2-} radical	
About 2ml aq. $MgSO_4.7H_2O$ solution is taken in a test tube to which barium chloride solution is added.	White ppt. insoluble in conc. HCl.	The white ppt. is of insoluble $BaSO_4$. $MgSO_4 + BaCl_2 \rightarrow BaSO_4 \downarrow + MgCl_2$ (white)

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DRY TEST FOR ACID RADICALS

EXPERIMENT	OBSERVATION	INFERENCE
	CO_3^{2-} radical	
a) 0.2g Na_2CO_3 sample is taken in a test tube and acidified with dilute HCl or H_2SO_4.	a) Colourless, odourless gas.	a) Carbonate salts react with dilute acids to liberate CO_2. $Na_2CO_3 + 2HCl \rightarrow 2NaCl + CO_2\uparrow + H_2O$
b) The issuing gas is passed into clear lime water.	b) The lime water initially turns turbid and then transparent.	b) Clear lime water turns turbid due to the formation of insoluble $CaCO_3$. $Ca(OH)_2 + CO_2 \rightarrow CaCO_3\downarrow + H_2O$ In presence of excess CO_2, $CaCO_3$ forms soluble $Ca(HCO_3)_2$ and the solution becomes transparent. $CaCO_3 + CO_2 + H_2O \rightarrow Ca(HCO_3)_2$

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EXPERIMENT	OBSERVATION	EXPLANATION
	S^{2-} radical	
About 0.2g Na_2S is taken in a test tube and dilute HCl or H_2SO_4 is added.	Gas with odour of rotten egg evolved.	Sulphite salts reacts with dilute acids to liberate H_2S gas with a rotten egg smell. $Na_2S + 2HCl \rightarrow 2NaCl + H_2S\uparrow$
• Filter paper soaked in lead acetate solution is held in the gas.	• Lead acetate paper turns black.	• Lead acetate reacts with H_2S to give black lead sulphide. $(CH_3COO)_2Pb + H_2S \rightarrow$ $(black)PbS\downarrow + 2CH_3COOH$
	Cl^- radical	
In a dry test tube, 0.2g NaCl sample is heated with conc. H_2SO_4.	Pungent smelling gas.	The pungent smelling gas is of HCl. $NaCl + H_2SO_4 \rightarrow NaHSO_4 + HCl\uparrow$
a) Moist blue litmus paper held on issuing gas.	a) The blue litmus paper turns red.	a) HCl (acidic gas) turns blue litmus paper red.
b) A glass rod soaked in NH_4OH held on issuing gas.	b) Dense white fumes.	b) HCl and NH_4OH react to give solid NH_4Cl producing dense white fumes. $HCl + NH_4OH \rightarrow NH_4Cl\downarrow + H_2O$

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EXPERIMENT	OBSERVATION	INFERENCE
	NO_3^- radical	
0.2g of the sample is taken in a test tube and heated with concentrated H_2SO_4.	Brown gas.	Nitrate salts react with concentrated H_2SO_4 to give nitric acid, which at high temperature is decomposed to brown coloured NO_2 gas. $Zn(NO_3)_2 + H_2SO_4 \rightarrow ZnSO_4 + 2HNO_3$ $4HNO_3 \rightarrow 2H_2O + 4NO_2\uparrow$ (brown) $+ O_2\uparrow$
	SO_4^{2-} radical	
1 part of the sample is mixed with 4 parts of Na_2CO_3 and heated in a reducing flame in a charcoal block.	Solid residue.	C reduces SO_4^{2-} salt to S^{2-} salt. $MgSO_4 + Na_2CO_3 = MgCO_3 + Na_2SO_4$ $Na_2SO_4 + 4C \rightarrow Na_2S + 4CO\uparrow$