Experiment 1   Ref: University Hand Book (C.U)

Estimation of sodium carbonate and sodium hydrogen carbonate present in a mixture

Date:

Principle:

Neutralisation of Na_2CO_3 solution by strong acid (HCl) occurs in two steps:

$Na_2CO_3 + HCl = NaHCO_3 + NaCl$ ($P^H = 8.3$ at the equivalence point)

$NaHCO_3 + HCl = NaCl + CO_2 + H_2O$ ($P^H = \sim 4$ at the equivalence point)

The acid base indicator is to be so selected that its P^Hrange for the colour change coincides with the sudden sharp change of P^H at the equivalence point. So at the first neutralisation point (PH = 8.3), phenolphthalein (P^H range 3.1-10) shows its colour change from pink to colourless. At this stage Na_2CO_3consumes only half the amount of HCl required for complete neutralisation. If methyl orange (P^Hrange = 3.1 to 4.4) is added to this titrated solution and the titration with HCl is continued upto second equivalence point, then this titre value corresponds to the amount of HCl required to convert $NaHCO_3$ to NaCl (i.e. $NaHCO_3$ derived from Na_2CO_3 plus the amount of $NaHCO_3$ present in the original mixture.

1000ml 1(N) HCl $\equiv$ 53 g of $Na_2CO_3 \equiv$ 84 g of $NaHCO_3$

Experimental Data

Table-1: Standardization of supplied NaOH solution with the supplied standard (N/20) oxalic acid solution

No of titration	Volume of oxalic acid solution (ml)	Volume of NaOH solution required (ml)	Mean volume of NaOH solution (ml)	Strength of NaOH solution
1	25	22.1	22.1	1.1312 (N/20)
2	25	22.1		

Calculation:

Applying $V_1 \times S_1 = V_2 \times S_2$

Strength of NaOH solution (S_2) $= \dfrac{25 \times N/20}{22.1}$

$= \textbf{1.1312 (N/20)}$

V_1 = Vol. of oxalic acid solution
S_1 = Strength of oxalic acid solution
V_2 = Vol. of NaOH solution

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Table-2: Standardization of supplied HCl solution with the standardized (N/20) NaOH solution

No of titration	Volume of HCl solution (ml)	Volume of NaOH solution required (ml)	Mean volume of NaOH solution (ml)	Strength of HCl solution
1	25	19.5	19.5	0.8823 (N/20)
2	25	19.5		

Calculation: Applying $V_1 \times S_1 = V_2 \times S_2$

Strength of HCl solution (S_1) $= \dfrac{19.5 \times 1.1312 \ (N/20)}{25}$

$\qquad\qquad\qquad\qquad = 0.8823 \ (N/20)$

V_1 = Vol. of HCl solution
V_2 = Vol. of NaOH solution
S_2 = Strength of NaOH solution

Table-3:Titration using phenolphthalein as indicator

No of titration	Volume of supplied mixture solution (ml)	Volume of HCl solution required (ml)	Mean volume of HCl solution (ml)
1	25	9.5	9.5
2	25	9.4	
3	25	9.6	

Table-4:Titration using methyl orange as indicator

No of titration	Volume of supplied mixture solution (ml)	Volume of HCl solution required (ml)	Mean volume of HCl solution (ml)
1	25	23.5	23.5
2	25	23.4	
3	25	23.6	

Calculation

Estimation of Na_2CO_3 solution:

Volume of HCl required for 1/2 mole Na_2CO_3 solution= 9.5 ml

Volume of HCl required for 1mole Na_2CO_3 solution = 2× 9.5 ml = 19 ml

1000 ml 1 (N) HCl solution $\equiv$ 53 g of Na_2CO_3
19 ml   0.8823 (N/20) $= \dfrac{53 \times 19 \times 0.8823}{1000 \times 20}$

$\qquad\qquad\qquad\qquad = 0.0444$ g of Na_2CO_3

25 ml supplied mixture sample solution contains 0.0444 g of Na_2CO_3
1000   " $= 0.0444 \times 40$

$\qquad\qquad\qquad\qquad\qquad = \textbf{1.7769}$g of Na_2CO_3

∴ The amount of Na_2CO_3 in the supplied mixture sample = **1.7769**g/lt.

Estimation of $NaHCO_3$:

Volume of HCl required for $NaHCO_3$ solution present in the mixture = 23.5-2×9.5 =4.5ml

1000 ml 1(N) HCl solution $\equiv$ 84 g of $NaHCO_3$

4.5ml 0.8823 (N/20)   " $\equiv \dfrac{84 \times 4.5 \times 0.8823}{1000 \times 20}$ = 0.01667g of $NaHCO_3$

25 ml supplied solution contains 0.01667g $NaHCO_3$

1000 ml   " $= \dfrac{0.01667 \times 1000}{25}$ g $NaHCO_3$

$\qquad\qquad\qquad\qquad = 0.6668$ g $NaHCO_3$

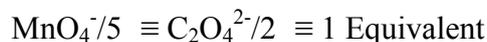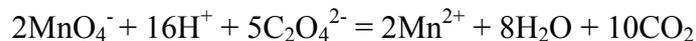∴ Therefore, $NaHCO_3$present in supplied solution = 0.6668g/L

Experiment 2

Estimation of oxalic acid by titrating it with KMnO₄

Date:

Principle:

Oxalic acid is estimated by titrating with standardised KMnO₄ solution in 2(N) H₂SO₄ medium at 70-80 ^{0}C. In dilute H₂SO₄ acid medium MnO_4^- quantitatively oxidises $C_2O_4^{2-}$ to CO_2 and itself reduced to Mn^{2+},

$$2MnO_4^- + 16H^+ + 5C_2O_4^{2-} = 2Mn^{2+} + 8H_2O + 10CO_2$$

$$MnO_4^-/5 \equiv C_2O_4^{2-}/2 \equiv 1 \text{ Equivalent}$$

$$1000 \text{ ml } 1(N) \text{ oxalate} \equiv 1000 \text{ ml } 1(N) \text{ permanganate}$$

KMnO₄ solution may be standardised against standard oxalic acid solution in 2(N) H₂SO₄ medium at 70-80 ^{0}C. Purple coloured KMnO₄ serves as self indicator. Its strengths may be calculated using the relation:

$$V_{MnO4}^- \times S_{MnO4}^- = V_{C2O4}^{2-} \times S_{C2O4}^{2-}$$

Experimental Data

(a)Table 1: Standardization of supplied ~(N/20) KMnO₄ solution

No of titration	Volume of standard oxalic acid taken (ml)	Volume of KMnO₄ solution required (ml)	Mean volume of KMnO₄ solution required (ml)	Strength of KMnO₄ solution
1	25	25.6		
2	25	25.7	25.6	0.9858(N/20)
3	25	25.5		

Applying $V_1 \times S_1 = V_2 \times S_2$

25 ×1.0095 (N/20)= 25.6× S₂   V₁ = Vol. of oxalic acid

S₂ (Strength of KMnO₄)= $\frac{25 \times 1.0095}{25.6}$ (N/20)   S₁ = Strength of oxalic acid

= 0.9858(N/20)   V₂ = Vol. of KMnO₄

(b) Table-2: Estimation of oxalic acid

No of titration	Volume of supplied solution (ml)	Volume of KMnO₄ solution required (ml)	Mean volume of KMnO₄ solution required (ml)
1	25	12.3	
2	25	12.4	12.3
3	25	12.2	

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Calculation:

1000 ml 1(N) KMnO₄ solution ≡ 63.03 g of oxalic acid

12.3 ml 0.9858(N/20) " $= \frac{63.03 \times 12.3 \times 0.9858}{1000 \times 20}$ = 0.0382 g of oxalic acid

25 ml supplied solution contains 0.0382 g of oxalic acid

1000 ml   " $= \dfrac{0.0382 \times 1000}{25}$ g of oxalic acid

$= 1.528$ g of oxalic acid

∴ Therefore, oxalic acid present in supplied solution = 1.528 g/L

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Experiment 3

Estimation of Fe (II) ions by titrating it with $K_2Cr_2O_7$ using internal indicator.

Date:

Principle:

In acid medium Fe^{II} in a solution may be estimated by direct titration with a standard solution of $K_2Cr_2O_7$ in presence of either phosphoric acid (H_3PO_4) or ammonium bifluoride (NH_4HF_2) using barium diphenylamine sulphonate (BDS) as indicator. Under this condition $K_2Cr_2O_7$ quantitively oxidises Fe^{2+} to Fe^{3+}:

$$Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} = 2Cr^{3+} + 7H_2O + 6Fe^{3+}$$
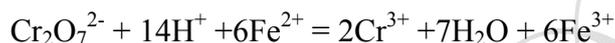
$$[Cr_2O_7^{2-}]/6 \equiv [Fe^{3+}]$$

$$1000ml\ 1(N)\ K_2Cr_2O_7\ solution \equiv 55.847\ g\ of\ Fe^{2+}$$

H_3PO_4 (or NH_4HF_2) stabilises Fe^{3+} by complex formation, which is essential for indicator action of BDS.

Experimental Data:

Determination of Fe^{2+}:

No. of titrations	Vol. of supplied Mohr's salt solution taken (ml)	Burette reading of standard $K_2Cr_2O_7$ solution (ml)			
		Initial	Final	Difference	Mean
1	25	0	25.2	25.2	
2	25	0	25.1	25.1	25.2
3	25	0	25.3	25.3	

Calculation:

$$1000ml\ 1(N)\ K_2Cr_2O_7\ solution \equiv 55.847\ g\ of\ Fe^{2+}$$

$$25.2ml\ 0.9915\ (N/20)\ " \equiv \frac{55.847 \times 25.2 \times 0.9915}{1000 \times 20} = 0.069977\ g\ of\ Fe^{2+}$$

25 ml supplied solution contains $0.069977\ g\ Fe^{2+}$

$$1000\ ml\ " = \frac{0.069977 \times 1000}{25}\ g\ Fe^{2+}$$

$$= 2.7988g\ Fe^{2+}$$

∴ Therefore, Fe^{2+} present in supplied solution = 2.7988g/L

Experiment 4
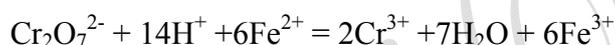# Estimation of Fe(II) and Fe(III) in a given mixture using $K_2Cr_2O_7$ solution

Date:

Principle:

In acid medium, direct titration of the Fe^{II}+ Fe^{III} mixture with standard $K_2Cr_2O_7$ solution gives the titre value (V_1) corresponding to the amount of Fe^{2+}. To estimate Fe^{3+} present in the mixture, it is first reduced to Fe^{2+} with highly pure Al-foil in 6(N) HCl medium.

$$3Fe^{3+} + Al = 3Fe^{2+} + Al^{3+}$$
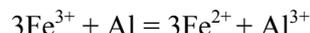
Acidity of the solution before titration is adjusted to 2(N) by diluting with distilled water. The resulting solution is then titrated with the same standard $K_2Cr_2O_7$ solution. This titre value (V_2) corresponds to the total iron [$Fe^{2+} + Fe^{3+}$]. The difference (V_2-V_1) corresponds to the amount of Fe^{3+}.

In acid medium $K_2Cr_2O_7$ quantitively oxidises Fe^{2+} to Fe^{3+}:

$$Cr_2O_7^{2-} + 14H^+ +6Fe^{2+} = 2Cr^{3+} +7H_2O + 6Fe^{3+}$$

$$[Cr_2O_7^{2-}]/6 \equiv [Fe^{2+}] \equiv [Fe^{3+}]$$

1000ml 1(N) $K_2Cr_2O_7$ solution $\equiv$ 55.847 g of Fe^{2+}

Experimental Data:

(i) Determination of Fe^{2+}:

No. of titrations	Vol. of supplied solution taken (ml)	Burette reading of standard K2Cr2O7 solution (ml)			
		Initial	Final	Difference	Mean
1	25	0	25.2	25.2	
2	25	0	25.1	25.1	25.2
3	25	0	25.3	25.3	

(ii) Determination of total iron (Fe^{2+} +Fe^{3+}):

No. of titrations	Volume of supplied solution (ml)	Burette reading of standard K2Cr2O7 solution (ml)			
		Initial	Final	Difference	Mean
1	25	0	49.2	49.2	
2	25	0	49.1	49.1	49.2
3	25	0	49.3	49.3	

Calculation:

1000ml 1(N) K2Cr2O7 solution $\equiv$ 55.847 g of Fe^{2+}

25.2 ml 0.9915 (N/20) " $\equiv \dfrac{55.847 \times 25.2 \times 0.9915}{1000 \times 20}$ = 0.069977 g of Fe^{2+}

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25 ml supplied solution contains 0.069977 g Fe^{2+}

1000 ml " $= \dfrac{0.069977 \times 1000}{25}$ g Fe^{2+}

$= 2.7988$g Fe^{2+}

∴ Therefore, Fe^{2+} present in supplied solution $= 2.7988$ g/L

The difference (49.2-25.2) $= 24$ ml 0.9915 $K_2Cr_2O_7$ solution gives the amount of Fe^{3+}.

1000ml 1(N) $K_2Cr_2O_7$ solution $\equiv 55.847$ g of Fe^{3+}

24 ml 0.9915 (N/20) " $\equiv \dfrac{55.847 \times 24 \times 0.9915}{1000 \times 20} = 0.06643$g of Fe^{3+}

25 ml supplied solution contains 0.066643 g Fe^{3+}

1000 ml " $= \dfrac{0.06643 \times 1000}{25}$ g Fe^{3+}

$= 2.6572$g Fe^{3+}

∴Therefore, Fe^{3+} present in supplied solution $= 2.6572$ g/L

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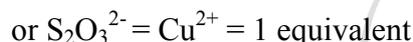Ph.D, Research Associate (C.U)
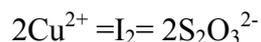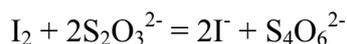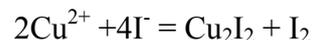Postdoc (South Korea & China)

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Experiment 5

Estimation of Cu (II) ions iodometrically using $Na_2S_2O_3$

Date:

Principle:

Copper (II) is iodometrically estimated by treating the solution containing Cu^{2+} with an excess of KI solution, when sparingly soluble coprous iodide, (Cu_2I_2) is precipitated with liberation of equivalent amount of iodine. Cu(II) is estimated by titrating the liberated iodine with a standardised solution of sodium thiosulphate.

$$2Cu^{2+} + 4I^- = Cu_2I_2 + I_2$$

$$I_2 + 2S_2O_3^{2-} = 2I^- + S_4O_6^{2-}$$

$$2Cu^{2+} = I_2 = 2S_2O_3^{2-}$$

$$\text{or } S_2O_3^{2-} = Cu^{2+} = 1 \text{ equivalent}$$

$$1000ml \ 1(N) \text{ thiosulfate solution} \equiv 63.546 \text{ g of } Cu^{2+}$$

Experimental Data:

(a) Standardization of supplied ~(N/20) thiosulfate solution:

No. of titrations	Vol. of $K_2Cr_2O_7$ solution taken (ml)	Burette reading of thiosulphate solution (ml)			
		Initial	Final	Difference	Mean
1	25	0	26.4	26.4	26.3
2	25	0	26.3	26.3	
3	25	0	26.4	26.2	

Applying $V_1 \times S_1 = V_2 \times S_2$

$25 \times 0.9915 \ (N/20) = 26.3 \times S_2$ V_1 = Vol. of $K_2Cr_2O_7$ solution

$S_2(\text{Strength of thiosulphate solution}) = \frac{25 \times 0.9915}{26.3} \ (N/20)$ S_1 = Strength of $K_2Cr_2O_7$ solution

$\qquad\qquad\qquad = 0.9425 \ (N/20)$ V_2 = Vol. of thiosulfate solution

(b) Estimation of Cu(II) by standardized thiosulfate solution:

No. of titrations	Volume of Cu^{2+} solution taken (ml)	Burette reading of thiosulphate solution (ml)			
		Initial	Final	Difference	Mean
1	25	0	30.1	30.1	30.1
2	25	0	30.1	30.1	
3	25	0	30.1	30.1	

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Calculation

1000 ml 1(N) thiosulphate solution $\equiv$ 63.546 g of Cu^{2+}

30.1 ml   0.9425 (N/20) $\equiv \dfrac{63.546 \times 30.1 \times 0.9425}{1000 \times 20} = 0.0901$ g Cu^{2+}

25 ml stock solution contains 0.0901 g Cu^{2+}

1000 ml $= \dfrac{0.0901 \times 1000}{25} = 3.604$ g Cu^{2+}

$\therefore$ Therefore, Cu^{2+} present in supplied solution $= 3.604$ g/L

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Experiment 6
Estimation of water of crystallization in Mohr's salt by titrating with KMnO4

Date:

Principle:

Normality of ferrous ammonium sulphate can be determined by directly titrating it against standard (N/20) $KMnO_4$ solution.

Equivalent weight = Strength/ Normality

∴ Equivalent weight of Mohr's salt $= \dfrac{Strength\ of\ Mohr's\ salt\ (g/l)}{Normality\ of\ Mohr;s\ salt}$

Substituting the value of strength and value of normality as calculated above, the equivalent weight of Mohr's salt can be calculated. Suppose it comes out to be E.
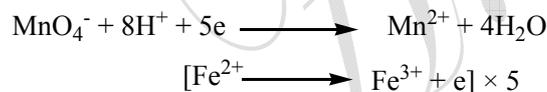
Since equivalent weight of Mohr's salt is equal to its molecular mass, therefore, molecular mass of Mohr's salt also equal to E.

Theoretical molecular mass of Mohr's salt, $FeSO_4.(NH_4)_2SO_4.nH_2O = 284+18n$

$$284 +18n = E \text{ where n can be calculated.}$$

∴ No of molecules of water of crystallisation, $n = \dfrac{E-284}{18}$

Ionic equation:

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

Experimental Data:

(a) Standardization of supplied ~(N/20) $KMnO_4$ solution:

No of titration	Volume of standard oxalic acid taken (ml)	Volume of KMnO₄ solution required (ml)	Mean volume of KMnO₄ solution required (ml)	Strength of KMnO₄ solution
1	25	23.2	23.2	
2	25	23.1		1.0830 (N/20)
3	25	23.3		

Applying $V_1 \times S_1 = V_2 \times S_2$

$25 \times 1.005\ (N/20) = 23.2 \times S_2$ V_1 = Vol. of oxalic acid

S_2 (Strength of KMnO₄)$= \dfrac{25 \times 1.005}{23.2}$ (N/20) S_1 = Strength of oxalic acid

           $= 1.0830$(N/20) V_2 = Vol. of KMnO₄

(b) Standardization of Mohr's salt solution by standardized KMnO$_4$ solution:

No. of titrations	Vol. of Mohr's salt solution taken (ml)	Burette reading of standardized KMnO$_4$ solution (ml)			
		Initial	Final	Difference	Mean
1	25	0	22.6	22.6	
2	25	0	22.6	22.6	22.6
3	25	0	22.6	22.6	

Applying V$_1$ × S$_1$ = V$_2$ × S$_2$

$25 \times S_1 = 22.6 \times 1.0830 \ (N/20)$ V$_1$ = Vol. of Mohr's salt solution

S_1 (Strength of Mohr's salt solution)$= \dfrac{22.6 \times 1.0830}{25}$ (N/20)   V$_2$ = Vol. of KMnO$_4$ solution

$\qquad\qquad\qquad = 0.9790 (N/20)$ S$_2$ = Strength of KMnO$_4$ solution)

$\qquad\qquad\qquad = 0.04895 \ (N)$

Calculation of equivalent weight of Mohr's salt:

Equivalent weight of Mohr's salt, $E = \dfrac{Strength \ of \ Mohr' salt \ (g/l)}{Normality \ of \ Mohr;s \ salt}$

$\qquad\qquad = \dfrac{19.6}{0.04895}$

No. of molecules of water of crystallisation, n:

$$\therefore 284 + 18n = \dfrac{19.6}{0.04895}$$
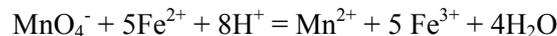
$$18n = 400.40858 - 284$$

$$n = 6.467$$

Experiment 7 (Not in the syllabus)
Estimation of Fe(II) using standardized KMnO₄ solution

Date:

Principle:

In dilute H_2SO_4 acid medium, $KMnO_4$ quantitively oxidises Fe^{2+} to Fe^{3+}:

$$MnO_4^- + 5Fe^{2+} + 8H^+ = Mn^{2+} + 5\ Fe^{3+} + 4H_2O$$

$$\therefore MnO_4^- \equiv 5Fe^{2+}$$

$$\therefore MnO_4^-/5 \equiv Fe^{2+}$$

Mohr's salt (Fe^{2+}) solution may be estimated by titrating it, in 2(N) H_2SO_4 medium with a standardised solution of $KMnO_4$ at room temperature in presence of H_3PO_4.

$$1000\ ml\ 1(N)\ KMnO_4 \equiv 55.847\ g\ of\ Fe^{2+}$$

Experimental Data:

(a) Standardization of supplied ~(N/20) KMnO₄ solution:

No of titration	Volume of standard oxalic acid taken (ml)	Volume of KMnO₄ solution required (ml)	Mean volume of KMnO₄ solution required (ml)	Strength of KMnO₄ solution
1	25	23.2	23.2	
2	25	23.1		1.0830 (N/20)
3	25	23.3		

Applying $V_1 \times S_1 = V_2 \times S_2$

$25 \times 1.005\ (N/20) = 23.2 \times S_2$ V_1 = Vol. of oxalic acid

S_2 (Strength of KMnO₄)$= \dfrac{25 \times 1.005}{23.2}$ (N/20) S_1 = Strength of oxalic acid

$\qquad\qquad = 1.0830(N/20)$ V_2 = Vol. of KMnO₄

(b) Standardization of Mohr's salt solution/ Estimation of Fe(II) in Mohr's salt by standardized KMnO₄ solution:

No. of titrations	Vol. of Mohr's salt solution taken (ml)	Burette reading of standardized KMnO₄ solution (ml)			
		Initial	Final	Difference	Mean
1	25	0	22.6	22.6	
2	25	0	22.6	22.6	22.6
3	25	0	22.6	22.6	

Applying $V_1 \times S_1 = V_2 \times S_2$

$25 \times S_1 = 22.6 \times 1.0830\ (N/20)$ V_1 = Vol. of Mohr's salt solution

S_1 (Strength of Mohr's salt solution)$= \dfrac{22.6 \times 1.0830}{25}$ (N/20) V_2 = Vol. of KMnO₄ solution

$\qquad\qquad = 0.9790(N/20$ S_2 = Strength of KMnO₄ solution)

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Calculation for the estimation of Fe(II) in Mohr's salt solution

1000 ml 1(N) $KMnO_4$ $\equiv 55.847$ g of Fe^{2+}

22.6 ml 1.0830 (N/20) " $\equiv \dfrac{55.847 \times 22.6 \times 1.0830}{1000 \times 20} = 0.0683$ g of Fe^{2+}

25 ml supplied Mohr's salt solution contains 0.0683 g Fe^{2+}

1000 ml   " $= \dfrac{0.0683 \times 1000}{25}$ g $Fe^{2+} = 2.732$ g Fe^{2+}

$\therefore$Therefore, Fe^{2+} present in supplied Mohr's salt solution = 2.732 g/L

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