II SEMESTER

PRACTICAL MANUAL FOR FIRST B.Sc. CHEMISTRY

(w. e. f. 2020 – 2021)

PRACTICAL – 2(VOLUMETRIC ANALYSIS)

PREPARED BY

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SYLLABUS

CHEMISTRY PRACTICAL – II (AT THE END OF II SEMESTER)
(w. e. f. 2020-2021)

Course outcomes: At the end of the course, the student will be able to;

- Use glassware, equipment and chemicals and follow experimental procedures in the laboratory
- Understand and explain the volumetric analysis based on fundamental concepts learnt in ionic equilibria
- Learn and identify the concepts of a standard solutions, primary and secondary standards
- Facilitate the learner to make solutions of various molar concentrations.
- This may include: The concept of the mole; Converting moles to grams; Converting grams to moles; Defining concentration; Dilution of Solutions; Making different molar concentrations.

VOLUMETRIC ANALYSIS

1. Estimation of sodium carbonate and sodium hydrogen carbonate present in a mixture.
2. Determination of Fe (II) using $KMnO_4$ with oxalic acid as primary standard.
3. Determination of Cu (II) using $Na_2S_2O_3$ with $K_2Cr_2O_7$ as primary standard.
4. Estimation of water of crystallization in Mohr's salt by titrating with $KMnO_4$

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SCHEME OF VALUATION

Time: 3 Hours | Max. Marks: 50M

Record | Marks: 5M

Viva-Voce | Marks: 5M

Writing procedure in 15 minutes	10 Marks
For tables and correct calculations	10 Marks
For value with < 0.1% error or less	20 Marks
(For every 0.1% error >0.1% 2 mark should be deducted from 20 marks)	
Minimum	2 Marks

HOD OF CHEMISTRY

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EXPERIMENT – 1

ALKALIMETRY OR ACIDIMETRY

PREPARATION OF STANDARD SODIUM CARBONATE SOLUTION

AIM

To prepare 250 ml of 0.05 M standard solution of sodium carbonate solution.

Procedure

Approximately 1.325 g of Na_2CO_3 is taken in a dry weighing bottle. The weight of empty weighing bottle and Na_2CO_3 (W_1) is weighed with the help of analytical balance. The substance is transferred into 250 ml volumetric flask. After transfer of substance in to volumetric flask empty weighing bottle weight (W_2) is weighed with the help of analytical balance. The difference between two weights ($W_1 - W_2$) gives the actual amount of Na_2CO_3 transferred into the volumetric flask. The Na_2CO_3 present in the volumetric flask is dissolved with distilled water and the flask is shake well till the Na_2CO_3 is completely dissolved and make up to the mark.

From the weight of Na_2CO_3 (W), the molarity of Na_2CO_3 is calculated.

REPORT

The molarity of Na_2CO_3 (M_1) is ----------m/L

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CALCULATIONS

Weight of empty weighing bottle + Na_2CO_3 W_1= --------- g

Weight of empty weighing bottle W_2 = --------- g

Accurate weight of the substance (Na_2CO_3) W= $W_1 - W_2$

Molarity of sodium carbonate (M_1) = $\frac{Weight\ of\ the\ substance\ X\ 1000}{Gram\ molecular\ weight\ X\ Volume}$ m/L

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STANDARDISATION OF HYDROCHLORIC ACID

AIM

Standardisation of the given HCl solution by using standard Na_2CO_3 solution.

PRINCIPLE

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + H_2O + CO_2$$

$$\frac{M1V1}{n1} = \frac{M2V2}{n2}$$

$$M2 = \frac{M1\ X\ V1\ X\ n2}{n1}\ \ m/L$$

PROCEDURE

Burette is rinsed with the given HCl solution and filled with the same. Take 20 ml of standard Na_2CO_3 solution in to a conical flask. To this add 1 or 2 drops of methyl orange indicator. The solution in the conical flask changes to pale yellow. Now the conical flask is placed under the burette and HCl rundown from the burette into conical flask. The titration is continued till the solution in the conical flask changes pale yellow to pale pink colour. This is the end point. The experiment is repeated until two consecutive readings are obtained.

REPORT: The molarity of HCl (M_2) = ---------- m/L

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CALCULATIONS

S.NO	Volume of standard Na_2CO_3 solution	Burette readings		Volume of HCl
		Initial	Final	

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + H_2O + CO_2$$

$$\frac{M1V1}{n1} = \frac{M2V2}{n2}$$

$$M_2 = \frac{M1\ X\ V1\ X\ n2}{n1} m/L$$

M_1 = Molarity of Na_2CO_3	M_1 =Molarity of HCl
V_1 = Volume of Na_2CO_3	V_1 =Volume of HCl
n_1=Number of moles of Na_2CO_3	n_1 =Number of moles of HCl

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ESTIMATION OF SODIUM CARBONATE AND SODIUM BI CARBONATE IN A MIXTURE

AIM

Estimate the amount of sodium carbonate and sodium bi carbonate in a given 100 ml problem solution.

PRINCIPLE

$$Na_2CO_3 + HCl \longrightarrow NaHCO_3 + NaCl$$

$$NaHCO_3 + HCl \longrightarrow NaCl + H_2O + CO_2\uparrow$$

PROCEDURE

The burette is filled with the given HCl. The given problem solution is diluted with distilled water and made up to the mark and shake well thoroughly.

Take 20 ml of problem solution into a conical flask and add 100 ml ice cold water and one or two drops of phenolphthalein indicator. The solution in the conical flask changes into pale pink colour. The acid is rundown carefully from the burette into the conical flask with constant shaking. The titration is continued till the solution in the conical flask changes to colourless. This is the first end point. Burette reading is noted. Then two drops of methyl orange indicator is added to the colourless solution. A light yellow colour solution is formed. Titration is continued till the light yellow colour solution in the conical flask changes to pale pink colour. This is the end point. The experiment is repeated until two consecutive readings are obtained.

REPORT

The amount of sodium carbonate present in the given problem solution= g

The amount of sodium bi carbonate present in the given problem solution= g

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CALCULATIONS

S.NO	VOLUME OF MIXTURE SOLUTION	BURETTE READINGS			2X	Y – 2X
		I	X	Y		
1	20 mL	0				
2	20 mL	0				

Volume of HCl required for the half neutralisation of Na_2CO_3 = X ml

Volume of HCl required for the complete neutralisation of Na_2CO_3 = 2X ml

Volume of HCl required for complete neutralisation of mixture solution =Y ml

Volume of HCl required for the neutralisation of $NaHCO_3$ present in the given problem solution =Y – 2Xml

VALUES OF $NaHCO_3$	VALUES OF Na_2CO_3
Values of HCl values of $NaHCO_3$	Values of HCl values of Na_2CO_3
M_2 = M_3=	M_2 = M_4 =
V_2 = V_3 =	V_2 = V_4 =
n_2 = n_3 =	n_2= n_4=
Molarity of $NaHCO_3$ in mixture $M_3= \frac{M_2V_2n_3}{V_3n_2}$ m/L	Molarity of Na_2CO_3 in mixture $M_4= \frac{M_2V_2n_4}{V_4n_2}$ m/L
Amount of $NaHCO_3$ in 100 mL solution $= \frac{M_3 \times GMWt \times 100}{1000}$ grams	Amount of Na_2CO_3 in 100 mL solution $= \frac{M_4 \times GMWt \times 100}{1000}$ grams

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EXPERIMENT – 2

PREPARATION OF STANDARD OXALIC ACID SOLUTION

AIM

To prepare 250 mL of about 0.05 M standard oxalic acid solution.

Principle

Oxalic acid is a very good primary standard. Its formula is $(COOH)_2.2H_2O$ Its formula weight is 126

Procedure

Approximately 1.6 g (1.575g) of oxalic acid is taken in a dry weighing bottle. The weight of empty weighing bottle and oxalic acid is weighed (W_1) with the help of analytical balance. The substance is transferred into 250 mL volumetric flask. After transfer of the substance into volumetric flask, empty weighing bottle is weighed (W_2). The difference between two weights ($W_1 - W_2$) gives the actual amount of oxalic acid transferred into the volumetric flask. The oxalic acid present in the volumetric flask is dissolved in distilled water and the flask is shaken well till the oxalic acid is completely dissolved and made up to the mark.

The molarity (M_1) of the prepared oxalic acid solution is calculated from the following expression.

$$M_1 = \frac{1000\,W}{mV}$$

Where W is weight of oxalic acid taken

V is volume of the solution

REPORT

Molarity of the prepared standard oxalic acid solution (M_1) is ------- moles/liter

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CALCULATIONS

Weight of empty weighing bottle plus oxalic acid, W_1 = --------- g

Weight of empty weighing bottle, W_2 = --------- g

Weight of the oxalic acid taken, W = $W_1 - W_2$ = --------- g

Volume of the solution, V = 250 mL

Formula, $(COOH)_2\ 2H_2O$, weight of oxalic acid, m = 126

Molarity of the prepared standard oxalic acid solution, $M_1 = \frac{1000\ W}{mV}$

$$M_1 = \frac{1000\ X \ldots\ldots\ldots}{126\ X\ 250}$$

M_1 = …… moles/liter

Report:

Molarity of the prepared standard oxalic acid solution, M_1 = …… moles/liter

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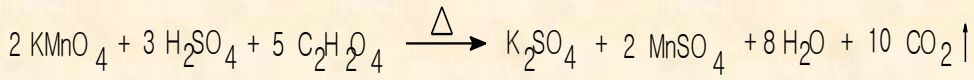
STANDARDISATION OF POTASSIUM PERMANGANATE

AIM

To standardize the given potassium permanganate solution using the prepared standard oxalic acid solution.

PRINCIPLE

Permanganate in acid medium oxidises oxalic acid quantitatively as follows:



PROCEDURE

Burette is rinsed with the given $KMnO_4$ solution and filled with the same. 20 mL of the standard oxalic acid solution is pipetted into a conical flask. To this about 20 mL(one test tube full) of 3M sulphuric acid is added. The conical flask is heated to about 90^0C (just short of boiling). Then into this hot solution permanganate is run down from the burette. In the beginning the reaction is slow, but takes place fast later (due to auto catalysis) and hence fast decolourisation takes place. The addition of permanganate is continued till a pale pink colour remains for about30 seconds. This is the end point. The titrations are repeated till two consecutive readings coincide.

REPORT:

Molarity of the given $KMnO_4$ solution (M_2) = ... moles/ liter

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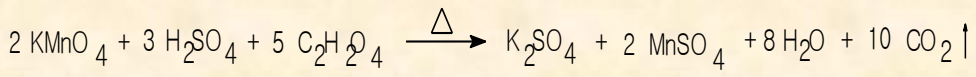
CALCULATIONS

S.No	Volume of standard Oxalic acid solution	Burette readings		Volume of Potassium permanganate
		Initial	Final	
1				
2				
3				



$$2\,KMnO_4 + 3\,H_2SO_4 + 5\,C_2H_2O_4 \xrightarrow{\Delta} K_2SO_4 + 2\,MnSO_4 + 8\,H_2O + 10\,CO_2\uparrow$$

$5C_2H_2O_4 \equiv 2\,KMnO_4$

Molarity of oxalic acid, M_1 = Volume of oxalic acid, V_1 = Number of moles of oxalic acid, n_1 =	Molarity of $KMnO_4$, M_2 = Volume of $KMnO_4$, V_2 = Number of moles of $KMnO_4$, n_2 =

$$\frac{M2\,V2}{n2} = \frac{M1\,V1}{n1}$$

$$\text{Molarity of } KMnO_4,\ M_2 = \frac{M_1\,V_1\,n_2}{n_1\,V_2}$$

= .....

= ..... moles/liter

Report

Molarity of the given $KMnO_4$ solution, M_2 = ... moles/liter

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ESTIMATION OF FERROUS ION

AIM

To estimate the amount of Ferrous iron present in whole of the problem solution.

PRINCIPLE

Ferrous ammonium sulphate (Mohr's salt) is represented simply as $FeSO_4$ in the equation as ammonium sulphate has nothing to do in the redox process. Ferrous sulphate is oxidised to ferric sulphate by potassium permanganate in acid medium quantitatively as follows.

$$2KMnO_4 + 8\,H_2SO_4 + 10\,FeSO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 5\,Fe_2(SO_4)_3 + 8H_2O$$

PROCEDURE

Burette is rinsed with the given $KMnO_4$ solution and filled with the same. The problem solution in 100 mL volumetric flask is made up to the mark. 20 mL of the problem solution is pipetted into a conical flask. To this 20 mL of 3M-H_2SO_4 is added. This solution is titrated with $KMnO_4$ solution from the burette. The titration is continued till the solution in the conical flask changes into pale pink colour. This is the end point. The experiment is repeated till two consecutive readings obtained.

REPORT

Amount of Ferrous iron present in the given problem solution = ... grams.

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CALCULATIONS

S.No	Volume of Mohr's solution	Burette readings		Volume of $KMnO_4$ solution
		Initial	Final	
1 2 3				

$$2KMnO_4 + 8\ H_2SO_4 + 10\ FeSO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 5\ Fe_2(SO_4)_3 + 8H_2O$$

$10\ FeSO_4 \equiv 2KMnO_4$	
Molarity of ferrous sulphate solution, M_3 = Volume of ferrous sulphate solution, V_3 = No. of moles of ferrous sulphate solution, n_3=	Molarity of permanganate solution, M_2 = Volume of permanganate solution, V_2 = No. of moles of permanganate solution, n_2 =

$$\frac{M3\ V3}{n3} = \frac{M2\ V2}{n2}$$

$$M_3 = \frac{M_2\ V_2\ n_3}{n_2\ V_3}$$

Molarity of ferrous sulphate solution, M_3 = ... moles/liter

Amount (A or W) of ferrous iron = $\frac{mMV}{1000}$(formula)

A or W of ferrous iron in 100 mL solution $= \frac{55.85\ X\ M_3\ X\ 100}{1000}$

$=$ .... grams

Report

Amount of ferrous iron present in the problem solution = ... grams

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EXPERIMENT – 3

PREPARATION OF STANDARD POTASSIUM DICHROMATE SOLUTION

AIM

To prepare 250 mL of about 0.016 M standard potassium dichromate solution.

Procedure

The formula weight of potassium dichromate is 294.22 A standard solution of about 0.016M is prepared by dissolving about 1.2 g of the substance in water in 250 mL volumetric flask.

About 1.2 g of AR grade potassium dichromate is taken in a dry weighing bottle. The weight of weighing bottle and potassium dichromate, W_1 is weighed with the help of analytical balance. The substance is transferred into 250 mL volumetric flask. Then, empty weighing bottle, W_2 is weighed. The potassium dichromate present in the volumetric flask is dissolved in distilled water and the flask is shaken well till the substance is completely dissolved to make the solution homogeneous. The solution is made up to the mark.

Molarity of potassium dichromate, M_1 is calculated from the following expression.

$$M_1 = \frac{1000\ W}{mV}$$

Where W is weight of potassium dichromate taken = W_1-W_2

V is volume of the solution = 250 mL

m is formula weight of potassium dichromate = 294.22

REPORT

Molarity of the prepared potassium dichromate solution, M_1 is --------- moles/liter

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CALCULATIONS

Weight of empty weighing bottle plus potassium dichromate, W_1 = --------- g

Weight of empty weighing bottle, W_2 = --------- g

Weight of potassium dichromate taken, $W = W_1 - W_2$ = --------- g

Volume of the solution, V = 250 mL

Molarity of the prepared standard potassium dichromate, $M_1 = \frac{1000\ W}{mV}$

$$M_1 = \frac{1000\ X\ (W1 - W2)}{294.22\ X\ 250}$$

M_1 = …… moles/liter

Report:

Molarity of the prepared standard potassium dichromate solution, M_1 = … moles/liter

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STANDARDISATION OF HYPO

AIM

To standardize the given hypo solution by standard potassium dichromate solution.

PRINCIPLE

Potassium dichromate solution in acid medium liberates iodine from potassium iodide. The liberating iodine can be titrated against hypo using starch as indicator.

$$K_2Cr_2O_7 + 14\ HCl + 6KI \longrightarrow 8\ KCl + 2\ CrCl_3 + 7\ H_2O + 3I_2$$

$$6\ Na_2S_2O_3 + 3I_2 \longrightarrow 6\ NaI + 3\ Na_2S_4O_6$$

PROCEDURE

The burette is filled with hypo solution. 20 mL of the standard $K_2Cr_2O_7$ solution is pipetted out into a conical flask. To this one test tube full of 10% KI solution (about 20 mL) and 1/3 test tube of conc.HCl is added. The mixture is allowed to stand in dark for 5 minutes by covering the conical flask with paper and keeping in a closed shelf.

Then, solution in the conical flask is titrated with hypo solution from the burette with constant shaking till the solution gets greenish yellow colour. Then 1 mL of 1% starch solution is added to it as an indicator. Then the solution is turned into deep blue colour. Now hypo solution is added drop by drop till the blue colour just turns to light green by the addition of one drop. This is the end point. The titrations are repeated till two consecutive readings coincide.

REPORT

Molarity of hypo solution, M_2 = ... moles/liter

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CALCULATIONS

S.No	Volume of $K_2Cr_2O_7$ solution	Burette readings		Volume of Hypo solution
		Initial	Final	
1				
2				
3				

$$K_2Cr_2O_7 + 6\ Na_2S_2O_3 + 6KI + 14\ HCl \longrightarrow 2CrCl_3 + 6NaI + 3Na_2S_4O_6$$

$1\ K_2Cr_2O_7 \equiv 6\ Na_2S_2O_3$	
Molarity of $K_2Cr_2O_7$ solution, M_1= Volume of $K_2Cr_2O_7$ solution, V_1= Number of moles of $K_2Cr_2O_7$ solution, n_1=	Molarity of hypo solution, M_2= Volume of hypo solution, V_2= Number of moles of hypo solution, n_2=

$$\frac{M2\ V2}{n2} = \frac{M1\ V1}{n1}$$

Molarity of hypo, $$M_2 = \frac{M_1\ V_1\ n_2}{n_1\ V_2}$$

= …… moles/liter

Report

Molarity of the given hypo solution, M_2 = ... moles/liter

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ESTIMATION OF COPPER

AIM

To estimate the amount of copper present in whole of the problem solution.

PRINCIPLE

In neutral or weakly acidic solution cupric copper reacts with KI to form cupric iodide. Cupric iodide is unstable and decomposes to give iodine which is titrated with hypo solution.

$$2\ CuSO_4 + 4\ KI \longrightarrow 2\ CuI_2 + 2\ K_2SO_4$$

$$2CuI_2 \longrightarrow 2\ CuI + I_2$$

$$2\ Na_2S_2O_3 + I_2 \longrightarrow 2\ NaI + Na_2S_4O_6$$

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

PROCEDURE

The problem copper sulphate solution is made up to the mark with distilled water and made up to the mark and shaken well. 20 mL of this solution is pipette into a clean conical flask a few drops of dilute sodium carbonate solution or dilute ammonia solution is added to it till a permanent ppt. remains in the solution. To it just few drops of acetic acid added to dissolve the ppt. completely. This is to replace the mineral acid medium. To it one test tube full of 10% KI is added. Three test tubes full of water is added to it. Conical flask is entirely covered with paper and kept inside of a closed shelf for at least 5 minutes.

Standard hypo solution rundown from the burette into the conical flask till light yellow appears. To it 1 mL starch indicator is added. Then the solution turned to blue. The titration is continued drop wise till cream colour (yellowish white) appeared. This is the end point. Titrations are repeated till two consecutive readings coincide.

REPORT

The amount of copper present in the problem solution = ... grams

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CALCULATIONS

S.No	Volume of copper Sulphate solution	Burette readings		Volume of hypo solution
		Initial	Final	
1 2 3				

$1\ CuSO_4 \equiv 1\ Na_2S_2O_3$	
Molarity of $CuSO_4$ solution, M_3= Volume of $CuSO_4$ solution, V_3= Number of moles of $CuSO_4$ solution, n_3=	Molarity of hypo solution, M_2= Volume of hypo solution, V_2= Number of moles of hypo solution, n_2=

$$\frac{M3\ V3}{n3} = \frac{M2\ V2}{n2}$$

$$M_3 = \frac{M_2\ V_2\ n_3}{n_2\ V_3}$$

Molarity of copper sulphate solution, M_3 = ... moles/liter

Amount (A) or Weight (W) of a substance in whole of the solution $= \frac{mMV}{1000}$

A or W of copper present in 100 mL solution $= \frac{63.54\ X\ M_3\ X\ 100}{1000}$

= ..... grams

Report

Amount of copper present in the whole problem solution = …….. grams

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