

19BCU213	CHEMISTRY PRACTICAL-II	Semester-II
		4H-2C
Instruction Hours/week :L: 0 T:0 P:3		Marks: Internal:40 External: 60 Total:100

Course Objective

The student on successful completion of the course should learn the principles of volumetric analysis and to estimate the compounds by acidimetry, alkalimetry and permanganometry.

Course Outcome

1. The student learnt the principles of quantitative analysis of inorganic compounds.
2. Learnt the estimation of sample present in a solution by volumetric analysis.

Volumetric analysis

A. Acidimetry & Alkalimetry

1. Estimation of sodium carbonate using standard sodium hydroxide.
2. Estimation of sodium hydroxide using standard sodium carbonate.
3. Estimation of sulphuric acid using standard oxalic acid.
4. Estimation of potassium permanganate using standard sodium hydroxide.

B. Permanganometry

1. Estimation of ferrous sulphate using standard Mohr's salt.
2. Estimation of oxalic acid using standard ferrous sulphate.
3. Estimation of calcium-direct method.

References:

- 1.Thomas, A.O. (2012). *Practical Chemistry for B.Sc. Main Students*. Cannanore: Kerala, Scientific Book Centre.
2. Ramasamy, R. (2011). *Allied Chemistry Practical Book*. Karur: Priya Publications.

3. Venkateswaran, V., Veeraswamy, R., & Kulandaivelu A. R. (2015). *Basic Principles of Practical Chemistry* (2nd edition). New Delhi: S. Chand Publications.



KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University)

(Established Under Section 3 of UGC Act 1956)

Coimbatore – 641 021.

LECTURE PLAN

DEPARTMENT OF CHEMISTRY

STAFF NAME: B. Prabha

SUBJECT NAME: CHEMISTRY PRACTICAL-II

SUB.CODE:19BCU212

SEMESTER: II

CLASS: I B.Sc (BIO-CHEMISTRY)

S.No.	Lecture Duration Period	Topics to be Covered	Support Material/Page Nos
1	3	Volumetric analysis-Introduction-classification.	R1:138-146
2	3	Estimation of sodium carbonate using standard sodium hydroxide.	R2: 4-28
3	3	Estimation of sodium hydroxide using standard sodium carbonate.	R1:162-163
4	3	Estimation of sulphuric acid using standard oxalic acid.	R1:158-160
5	3	Estimation of potassium permanganate using standard sodium hydroxide.	R2:24-28
6	3	Viva-voce questions discussion-Acidimetry , Alkalimetry & Permanganometry	R1:519-522
7	3	Estimation of ferrous sulphate using standard Mohr's salt.	R1:189-191
8	3	Estimation of oxalic acid using standard ferrous sulphate.	R1:185-187
9	3	Estimation of calcium-direct method.	R1:191-193
10	3	Model Practical Examination.	
	Total No. of Hours Planned For Practical's = 30		

Reference Books:

R1.Thomas, A.O. (2012). *Practical Chemistry for B.Sc. Main Students*. Cannanore: Kerala, Scientific Book Centre.

R2. Ramasamy, R. (2011). *Allied Chemistry Practical Book*. Karur: Priya Publications.

A Laboratory Manual on VOLUMETRIC ANALYSIS

B. Prabha

Department of Chemistry



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VOLUMETRIC ANALYSIS

Introduction: Quantitative chemical analysis is ordinarily done by two methods namely volumetric and gravimetric methods. Volumetric analysis involves estimation of a substance in solution by neutralization, reduction, oxidation or precipitation by means of another solution of accurately known strength. Gravimetric analysis involves estimation of a substance by process of weighing. Of the two methods of analysis, the volumetric analysis is much more rapidly carried out. Due to simplicity, accuracy and wide applicability, volumetric analysis is preferred to gravimetric analysis.

Volumetric analysis depends on measurements of the volumes of solutions of the interacting substances. A measured volume of the solution of a substance 'A' is allowed to react completely with the solution of definite strength of another substance 'B'. The volume of 'B' is noted. Thus we know the volumes of the two solutions used in the reaction and the strength of solution 'B'. From this data, we can find the strength of the solution 'A'.

Titration: The process of finding out the volumes of reagents required to bring out a definite reaction just to completion is termed as titration.

End Point: The end point of a reaction is the stage at which complete reaction takes place between two solutions. The end point is determined by an indicator which shows a marked color change at the completion of the chemical reaction.

Standard Solution: Volumetric analysis depends on the use of one standard solution; i.e., a solution of known strength or concentration. A standard solution is prepared by dissolving an accurately weighed amount of a substance called primary standard substance in a definite volume of the solution. The primary standard substance should fulfill the following characteristics:-

- (a) It should be available in a high degree of purity.
- (b) It should be stable and unaffected by the atmosphere.
- (c) It should not be efflorescent or deliquescent.
- (d) It should be readily soluble in water and
- (e) Its solution in distilled water should not deteriorate on keeping.

Examples of primary standard substances are crystalline oxalic acid, anhydrous sodium carbonate, potassium dichromate, Mohr's salt, sodium chloride etc. If a substance

does not conform to one or more of the above characteristics, its standard solution is prepared indirectly, i.e; preparing a solution of approximately higher concentration than the required one and subsequently determining its exact concentration by titrating it against a solution of a suitable primary standard substance. The solution is then diluted to such an extent so as to get the required concentration. Solution of potassium permanganate, inorganic acid, caustic alkalies etc. are prepared by indirect method.

Normality: The concentration of solution is generally expressed in terms of normality. A normal solution is a solution which contains one gram equivalent mass of the substance dissolved in one liter of a solution. Thus a normal (N) solution of sodium hydroxide contains 40 g of sodium hydroxide in one liter of the solution. 2N solution of sodium hydroxide contains 80 g of sodium hydroxide in one liter of the solution. A decinormal solution (0.1 N) of sodium hydroxide contains 4g of sodium hydroxide per liter of the solution.

$$\text{Normality} = \frac{\text{Mass in grams per liter of solution}}{\text{Gram equivalent mass of the substance}}$$

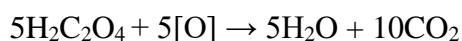
Classification of Volumetric Reactions:

Volumetric reactions are classified under the following heads.

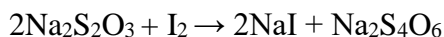
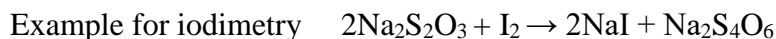
1. Acidimetry and alkalimetry: Acidimetry is the estimation of alkali solution using standard acid solution. Alkalimetry is the estimation of an acid solution using standard alkali solution.



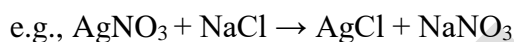
2. Oxidation-Reduction Titrations: Here an oxidizing agent is estimation by titrating it with a standard reducing agent and vice-versa. In this type of reaction oxidation and reduction take place simultaneously.



3. Iodimetry and iodometry: Estimations using standard iodine solution are called iodimetry and those involving iodine liberated from the potassium iodide solution by a chemical reaction is iodometry.



4. Precipitation Titrations: In this type of titration, the strength of a solution is determined by its complete precipitation with a standard solution of another substance.



5. Complexometric Titrations: This type of titration depends upon the concentration of ions (other than H^+ and OH^-) to form a soluble ion or compound.



The common complexing agent used in complexometric titration ethylene diamine tetra acetic acid (EDTA).

Equivalent masses of compounds:

In volumetric analysis a knowledge of the equivalent mass of substance to be estimated and whose standard solutions are to be prepared should be known.

1. Equivalent mass of an acid: Equivalent mass of an acid is the number parts by mass of it which contains 1.008 parts by mass of replaceable hydrogen.

$$\text{Equivalent mass of an acid} = \frac{\text{Molecular mass of the acid}}{\text{Basicity of the acid}}$$

Acid	Molecular mass	Number of replaceable H atoms (Basicity)	Equivalent mass
HCl	36.5	1	36.5
HNO ₃	63	1	63
H ₂ SO ₄	98	2	49
H ₂ C ₂ O ₄ .2H ₂ O	126	2	63

2. Equivalent mass of a base: Equivalent mass of a base is the number parts by mass of it which will completely react with one equivalent of an acid.

$$\text{Equivalent mass of an alkali} = \frac{\text{Molecular mass of the alkali}}{\text{Acidity of the alkali}}$$

Acidity is the number of hydrogen ions which react with one molecule of alkali.

Alkali	Molecular mass	Acidity	Equivalent mass
NaOH	40	1	40
KOH	56	1	56
NaHCO ₃	84	1	84
KHCO ₃	100	1	100
Na ₂ CO ₃	106	2	53
K ₂ CO ₃	138	2	69
CaCO ₃	100	2	50

3. Equivalent mass of an oxidizing agent: Equivalent mass of oxidizing agent is numerically equal to the number of parts by mass of it which contains 8 parts by mass of available oxygen. Available oxygen means oxygen capable of being utilized for oxidation.

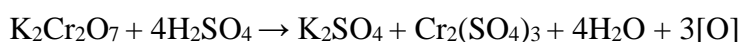
(a) Equivalent mass of KMnO₄ in acid medium. In acid medium two molecules of potassium permanganate give 5 atoms of oxygen for oxidation.



316 parts by mass of potassium permanganate give 80 parts by mass of oxygen for oxidation.

$$\text{Equivalent mass of potassium permanganate in acid medium} = \frac{316 \times 8}{80} = 31.6$$

(b) Equivalent mass of K₂Cr₂O₇ in acid medium



294 parts by mass of potassium dichromate give 48 parts by mass of oxygen.

$$\text{Equivalent mass of potassium dichromate in acid medium} = \frac{294 \times 8}{48} = 49$$

(c) Equivalent mass of MnO₂ in acid medium



87 parts by mass of manganese dioxide give 16 parts by mass of oxygen.

$$\text{Equivalent mass of manganese dioxide in acid medium} = \frac{87 \times 8}{16} = 43.5$$

4 Equivalent mass of a reducing agent: Equivalent mass of a reducing agent is the number of parts by mass of it which can be oxidized by 8 parts (one equivalent) by mass of oxygen.

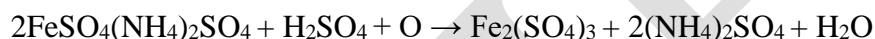
(a) Equivalent mass of ferrous sulphate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$

In the presence of dilute sulphuric acid, ferrous sulphate is oxidized as follows:



The equivalent mass of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ is the same as its molecular mass i.e., 278.

(b) Equivalent mass of Mohr's salt, $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$



The equivalent mass of Mohr's salt is the same as its molecular mass i.e., 392.

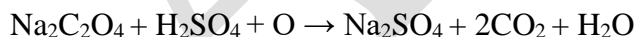
(c) Equivalent mass of oxalic acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$



One equivalent of oxygen oxidizes half the molecular mass of oxalic acid. Therefore, the equivalent mass of oxalic acid is

$$\frac{\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}}{2} = \frac{126}{2} = 63$$

(d) Equivalent mass of sodium oxalate, $\text{Na}_2\text{C}_2\text{O}_4$



Equivalent mass of sodium oxalate is half its molecular mass i.e., 67

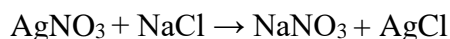
5. Equivalent mass of metal halides

$$\text{Equivalent mass of metal halide} = \frac{\text{Molecular mass of halide}}{\text{No. of halogen ion furnished by one molecule}}$$

$$\text{Thus, equivalent mass of NaCl} = \frac{58.46}{1} = 58.46$$

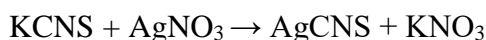
$$\text{KCl} = \frac{74.46}{1} = 74.46$$

(a) Equivalent mass of silver nitrate



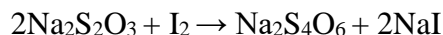
$$\text{Equivalent mass of silver nitrate} = \frac{\text{AgNO}_3}{1} = \frac{170}{1} = 170$$

(b) Equivalent mass of Potassium thiocyanate



Hence, equivalent mass of potassium thiocyanate = $\frac{\text{KCNS}}{1} = 166$

(c) Equivalent mass of sodium thiosulphate and iodine



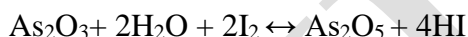
Equivalent mass of sodium thiosulphate is its own molecular mass i.e., $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ = 248 and that of iodine is its own atomic mass i.e., 127.

(d) Equivalent mass of copper sulphate



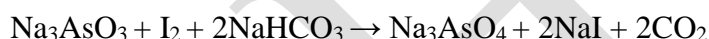
Therefore the equivalent mass of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is its own molecular mass = 249.5.

(e) Equivalent mass of arsenious oxide



Therefore the equivalent of arsenious oxide = $\frac{\text{As}_2\text{O}_3}{4} = 49$

(f) Equivalent mass of sodium arsenite



Therefore the equivalent of sodium arsenite = $\frac{\text{Na}_3\text{AsO}_3}{2} = 98$

Principle of volumetric analysis:

When two solutions compound react with each other, the product of volume and normality of one solution will be equal to the product of volume and normality of the other solution.

$V \times N = V_1 \times N_1$ where V and N are the volume and normality of the first solution and V_1 and N_1 are the volume and normality of the second solution. Thus, if the volumes of the two solutions which compound react with each other are determined, and if the normality of one solution is known, the normality of the other solution can be calculated. By multiplying the normality factor by the equivalent mass, the mass of the substance in one liter of the solution is found out.

Normality \times Equivalent mass = Mass per litre of the solution

Estimation of Na_2CO_3 using standard NaOH

Normality of standard NaOH = 0.1N

Titration I:

Standard NaOH Vs link HCl solution:

Burette solution – Link HCl solution

Pipette solution – NaOH solution

Indicator – Methyl orange

End point – Change of colour from golden yellow to pale pink.

S.No.	Volume of NaOH (V_1) (ml)	Burette reading		Volume of HCl (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of NaOH (V_1) = 20 ml

Normality of NaOH (N_1) = 0.1 N

Volume of HCl (V_2) = _____ ml

Normality of HCl (N_2) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{20 \times 0.1}{\quad}$$

Normality of HCl (N_2) = _____ N

Titration II:

Link HCl solution Vs unknown Na_2CO_3 :

Burette solution – Link HCl solution

Pipette solution – Na_2CO_3 solution

Indicator – Methyl orange

End point – Change of colour from golden yellow to pale pink.

Ex. No.: 1

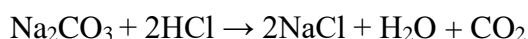
Estimation of sodium carbonate using standard sodium hydroxide

Aim:

To estimate the amount of sodium carbonate present in the whole of the given solution using a standard solution of sodium hydroxide containing 4 g of substance in 1000 ml and an approximately decinormal solution of hydrochloric acid.

Principle:

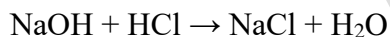
The estimation depends on the reactions between Na_2CO_3 and HCl and that of NaOH and HCl . Na_2CO_3 reacts with HCl by a simple neutralization reaction.



The end point of the reaction is determined by using methyl orange as indicator. Na_2CO_3 is dibasic and its equivalent weight is

$$\text{Formula} = \frac{\text{Weight}}{2} = \frac{106}{2} = 53$$

Similarly, NaOH reacts with HCl as follows.



The end point of this reaction is determined by using methyl orange as indicator. NaOH is monobasic and its equivalent weight is

$$\text{Formula} = \frac{\text{Weight}}{1} = \frac{40}{1} = 40$$

Procedure:

Titration 1: Standardization of hydrochloric acid

Titration of standard NaOH Vs HCl

Exactly 20 ml of standard NaOH solution was pipetted out into a clean conical flask, and a drop of methyl orange indicator was added. This solution was titrated against HCl taken in burette. The end point was the change of colour from golden yellow to pale pink. The titration was repeated for concordant value. Using this value, the strength of HCl solution was calculated.

S.No.	Volume of Na_2CO_3 (V_2) (ml)	Burette reading		Volume of HCl (V_1) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of HCl (V_1) = _____ ml

Normality of HCl (N_1) = _____ N

Volume of Na_2CO_3 (V_2) = 20 ml

Normality of Na_2CO_3 (N_2) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{\quad}{20}$$

Normality of Na_2CO_3 (N_2) = _____ N

Amount of Na_2CO_3 present in 1 litre of the given solution

= Normality x Equivalent weight

= _____ x 53

= _____ g

The amount of Na_2CO_3 present in the whole of the given solution

= Normality x Equivalent weight x 100

1000

= _____ x 100

1000

= _____ g.

Titration 2: Estimation of unknown Na_2CO_3

Titration of standard HCl Vs unknown Na_2CO_3

The given unknown solution was transferred to a 100 ml standard flask using a glass rod and funnel and was diluted upto the mark. The solution was shaken thoroughly for uniform concentration. The given pipette was washed with water and rinsed with the made up solution. Exactly 20 ml of this solution was pipetted out into a clean conical flask and a drop of methyl orange indicator was added and the solution had golden yellow. The solution was titrated against standardized HCl taken in the burette. The end point is the change of colour from golden yellow to pink. The final burette reading was noted. The titration was repeated for concordant value. Using the concordant value, the strength and the amount of Na_2CO_3 present in the whole of the given solution was calculated.

Result:

The amount of Na_2CO_3 present in the whole of the given solution is = _____ g.

Estimation of NaOH using standard Na_2CO_3

Normality of standard $\text{Na}_2\text{CO}_3 = 0.1\text{N}$

Titration I:

Standard Na_2CO_3 Vs link HCl solution

Burette solution – Link HCl solution

Pipette solution – Na_2CO_3 solution

Indicator – Methyl orange

End point – Change of colour from golden yellow to pale pink.

S.No.	Volume of NaOH (V_1) (ml)	Burette reading		Volume of HCl (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of Na_2CO_3 (V_1) = 20 ml

Normality of Na_2CO_3 (N_1) = 0.1 N

Volume of HCl (V_2) = _____ ml

Normality of HCl (N_2) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{20 \times 0.1}{\quad}$$

Normality of HCl (N_2) = _____ N

Titration II:

Link HCl solution Vs unknown NaOH

Burette solution – Link HCl solution

Pipette solution – NaOH solution

Indicator – Phenolphthalein

End point – Disappearance of pink colour.

Ex. No.: 2

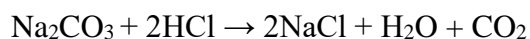
Estimation of sodium hydroxide using standard sodium carbonate

Aim:

To estimate the amount of sodium hydroxide present in the whole of the given solution using a standard solution of sodium carbonate containing 5.3 g of substance in 1000 ml and an approximately decinormal solutions of hydrochloric acid.

Principle:

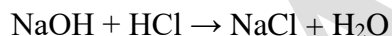
The estimation depends on the reaction between Na_2CO_3 and HCl and that of NaOH and HCl . Na_2CO_3 reacts with HCl by a simple neutralization reaction.



The end point of the reaction is determined by using methyl orange as indicator. Na_2CO_3 is dibasic and its equivalent weight is

$$\text{Formula} = \frac{\text{Weight}}{2} = \frac{106}{2} = 53$$

Similarly, NaOH reacts with HCl as follows.



The end point of the reaction is determined by using phenolphthalein as indicator. NaOH is monobasic and its equivalent weight is

$$\text{Formula} = \frac{\text{Weight}}{1} = \frac{40}{1} = 40$$

Procedure:

Titration: Standardization of HCl

Titration of link HCl Vs standard Na_2CO_3

Exactly 20 ml of standard Na_2CO_3 solution was pipetted out into a clean conical flask and a drop of methyl orange indicator was added. This solution was titrated against HCl taken in the burette. The end point is the change of colour from golden yellow to pale pink. The titration was repeated for concordant value. Using this value, the strength of HCl solution was calculated.

S.No.	Volume of NaOH (V ₂) (ml)	Burette reading		Volume of HCl (V ₁) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of HCl (V₁) = _____ ml

Normality of HCl (N₁) = _____ N

Volume of NaOH (V₂) = 20 ml

Normality of NaOH (N₂) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{20}{20}$$

Normality of NaOH (N₂) = _____ N

Amount of NaOH in 1 litre of the given solution

= Normality x Equivalent weight

$$= \frac{20}{20} \times 40$$

$$= 40 \text{ g}$$

The amount of NaOH present in the whole of the given solution

$$= \frac{\text{Normality} \times \text{Equivalent weight} \times 100}{1000}$$

$$= \frac{20 \times 40 \times 100}{1000}$$

$$= 80 \text{ g.}$$

Titration II: Estimation of NaOH

Titration of link HCl Vs unknown NaOH solution

The given unknown solution was transferred to a 100 ml standard flask using a glass rod and funnel and was diluted upto the mark and the solution was shaken thoroughly for uniform concentration. The given pipette was washed with water and rinsed with the made up solution. Exactly 20 ml of this solution was pipetted out into a conical flask and a drop of phenolphthalein indicator was added. The solution turns pink in colour. This solution was titrated against standardized HCl taken in the burette with the disappearance of pink colour. The final burette reading was noted. The titration was repeated for concordant value. Using the concordant value, the strength and the amount of NaOH present in the whole of the given solution was calculated.

Result:

The amount of NaOH present in the whole of the given solution is = _____g.

Estimation of H_2SO_4 using standard oxalic acid

Normality of standard Oxalic acid = 0.1 N

Titration I:

Standard $\text{H}_2\text{C}_2\text{O}_4$ Vs link NaOH solution

Burette solution – NaOH solution

Pipette solution – Oxalic acid solution

Indicator – Phenolphthalein

End point – Appearance of pale pink colour.

S.No.	Volume of $\text{H}_2\text{C}_2\text{O}_4$ (V_1) (ml)	Burette reading		Volume of NaOH (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of oxalic acid (V_1) = 20 ml

Normality of oxalic acid (N_1) = 0.1 N

Volume of NaOH (V_2) = _____ ml

Normality of NaOH (N_2) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{\times 0.1}{20}$$

Normality of NaOH (N_2) = _____ N

Titration II:

Unknown H_2SO_4 Vs standardized NaOH

Burette solution – H_2SO_4 solution

Pipette solution – NaOH solution

Indicator – Phenolphthalein

End point – Disappearance of pink colour.

Ex. No.:3

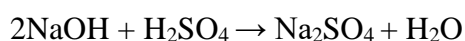
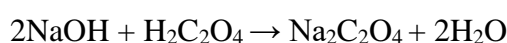
Estimation of sulphuric acid using standard oxalic acid

Aim:

To estimate the amount of sulphuric acid present in the whole of the given solution using a standard solution of oxalic acid containing 6.3 g of the substance in 1000 ml and an approximately decinormal solution of sodium hydroxide.

Principle:

NaOH is a strong base and it can react with oxalic acid and H_2SO_4 follows.



The end point of these reactions can be determined by using phenolphthalein as indicator. Oxalic acid is dibasic and its equivalent weight is

$$\begin{aligned}\text{Formula} &= \frac{\text{Molecular weight}}{2} = \frac{\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}}{2} \\ &= \frac{126}{2} = 63\end{aligned}$$

Sulphuric acid is dibasic and its equivalent weight is

$$\text{Formula} = \frac{\text{Molecular weight}}{2} = \frac{98}{2} = 49$$

One molecule of H_2SO_4 reacts with two molecules of NaOH. Therefore equivalent weight of H_2SO_4 is 49.

Procedure:

Titration I: Standardization of NaOH

Titration of standard oxalic acid Vs NaOH solution

Exactly 20 ml of standard oxalic acid solution was pipetted out into a clean conical flask, and one or two drops of phenolphthalein indicator was added. This solution was titrated against NaOH taken in the burette till the appearance of pink colour. The titration was repeated for concordant value. Using this value, the strength of NaOH solution was calculated.

S.No.	Volume of NaOH (V ₂) (ml)	Burette reading		Volume of H ₂ SO ₄ (V ₁) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of NaOH (V₂) = 20 ml

Normality of NaOH (N₂) = _____ N

Volume of H₂SO₄ (V₁) = _____ ml

Normality of H₂SO₄ (N₁) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_1 = \frac{V_2 \times N_2}{V_1}$$

$$N_1 = \frac{20 \times 0.1}{}$$

Normality of H₂SO₄ (N₁) = _____ N

Amount of H₂SO₄ in 1 litre of the given solution

= Normality x Equivalent weight

= _____ x 49

= _____ g

The amount of H₂SO₄ present in the whole of the given solution

$$= \frac{\text{Normality} \times \text{Equivalent weight} \times 100}{1000}$$

$$= \frac{ }{10}$$

$$= \text{_____ g.}$$

Titration II: Estimation of unknown H_2SO_4

Titration of unknown H_2SO_4 Vs standard NaOH

The given unknown solution was transferred to a 100 ml standard flask. Using a glass rod and funnel and was diluted upto the mark. The solution was shaken thoroughly for uniform concentration. Exactly 20 ml of standardized NaOH solution was pipetted out into a clean conical flask and one or two drops of phenolphthalein indicator was added. This solution was titrated against the made-up H_2SO_4 solution taken in the burette. The end point was the disappearance of pink colour. Repeat the titration for concordant value. Using this value, the strength and the amount of H_2SO_4 present in the whole of the given solution was calculated.

Result:

The amount of H_2SO_4 present in the whole of the given solution=_____g.

Estimation of KMnO_4 using standard NaOH

Normality of standard NaOH = 0.1N

Titration I:

Titration of standard NaOH Vs link oxalic acid solution

Burette solution – Standard NaOH solution

Conical flask solution – 20 ml of oxalic acid solution

Indicator – Phenolphthalein

End point – Appearance of permanent pale pink colour.

S.No.	Volume of oxalic acid (V_2) (ml)	Burette reading		Volume of NaOH (V_1) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of NaOH (V_1) = _____ ml

Normality of NaOH (N_1) = 0.1 N

Volume of oxalic acid (V_2) = 20 ml

Normality of oxalic acid (N_2) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{\times 0.1}{20}$$

Normality of oxalic acid (N_2) = _____ N

Titration II:

Unknown KMnO_4 Vs standardized oxalic acid solution

Burette solution – Unknown KMnO_4

Conical flask solution – 20 ml of oxalic acid solution + 20 ml of H_2SO_4

Indicator – Self

End point – Appearance of permanent pale pink colour.

Ex. No.: 4

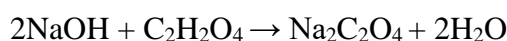
Estimation of potassium permanganate using standard sodium hydroxide

Aim:

To estimate the amount of potassium permanganate present in the whole of the given solution using a standard solution of sodium hydroxide containing 4 g of the substance in 1000 ml and an approximately decinormal solution of oxalic acid.

Principle:

NaOH is a strong base and it reacts with oxalic acid to form sodium oxalate.



The end point of the reaction can be determined by using phenolphthalein as indicator. Oxalic acid is dibasic and its equivalent weight is

$$\begin{aligned}\text{Formula} &= \frac{\text{Molecular weight}}{2} = \frac{\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}}{2} \\ &= \frac{126}{2} = 63\end{aligned}$$

KMnO₄ is an oxidizing agent and it can react with oxalic acid in the presence of a mineral acid like, H₂SO₄.



$$\begin{aligned}\text{Equivalent mass of KMnO}_4 &= \frac{\text{Molecular weight}}{\text{Change in oxidizing state}} \\ &= \frac{158}{5} = 31.6\end{aligned}$$

Procedure:

Titration I: Standardization of oxalic acid

Titration of standard NaOH Vs link oxalic acid solution

Exactly 20 ml of oxalic acid solution was pipetted out into a clean conical flask, and one or two drops of phenolphthalein indicator was added. This solution was titrated against standard NaOH taken in the burette. The end point was the appearance of permanent pale pink colour. Repeated the titration for concordant value. Using the concordant value, the strength of oxalic acid solution was calculated.

S.No.	Volume of oxalic acid (V_1) (ml)	Burette reading		Volume of KMnO_4 (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of Oxalic acid (V_1) = 20 ml

Normality of Oxalic acid (N_1) = _____ N

Volume of KMnO_4 (V_2) = _____ ml

Normality of KMnO_4 (N_2) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{20 \times \quad}{\quad}$$

Normality of KMnO_4 (N_2) = _____ N

Amount of KMnO_4 present in 1 litre of the given solution

= Normality x Equivalent weight

= _____ x 31.6

= _____ g

Amount of KMnO_4 present in the whole of the given solution

= $\frac{\text{Normality} \times \text{Equivalent weight} \times 100}{1000}$

= $\frac{\quad}{10}$

= _____ g

Titration II: Estimation of unknown KMnO_4

Titration of unknown KMnO_4 Vs standardized oxalic acid

The given unknown KMnO_4 solution was transferred to a 100 ml of standard flask using a glass rod and funnel and was diluted upto the mark and shaken thoroughly for uniform concentration. Exactly 20 ml of standardized oxalic acid solution was pipetted out into a clean conical flask. About 20 ml of dilute H_2SO_4 was added and the mixture was heated to bearable warmth. It was then titrated against the unknown KMnO_4 solution taken in the burette. The end point was the appearance of permanent pale pink colour. Repeat the titration for concordant value. Using this value, the strength and the amount of KMnO_4 present in the whole of the given solution was calculated.

Result:

The amount of KMnO_4 present in the whole of the given solution = _____ g.

Estimation of FeSO_4 using standard Mohr's salt

Normality of Standard Mohr's salt = 0.1 N

Titration 1:

Standard Mohr's salt Vs link KMnO_4 solution

Burette solution – Link KMnO_4 solution

Conical flask solution – 20 ml Mohr's salt + 20 ml of dilute H_2SO_4

Indicator – Self

End point – Appearance of permanent pale pink colour.

S.No.	Volume of Mohr's Salt (V_1) (ml)	Burette reading		Volume of KMnO_4 (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of Mohr's salt (V_1) = 20 ml

Normality of Mohr's salt (N_1) = 0.1 N

Volume of KMnO_4 (V_2) = _____ ml

Normality of KMnO_4 (N_2) = ?

According to Volumetric Principle

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{20 \times 0.1}{\quad}$$

Normality of KMnO_4 (N_2) = _____ N

Titration II:

Unknown FeSO_4 Solution Vs Standardized KMnO_4

Burette solution – Link KMnO_4 solution

Conical flask solution – 20 ml FeSO_4 + 20 ml of dilute H_2SO_4

Indicator – Self

End point – Appearance of permanent pale pink colour.

Ex. No.: 5

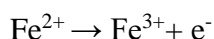
Estimation of ferrous sulphate using standard Mohr's salt

Aim:

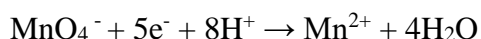
To estimate the amount of ferrous sulphate present in the whole of the given solution using a standard solution of Mohr's salt, containing 39.18 g of the substance in 1000 ml and an approximately decinormal solution of potassium permanganate.

Principle:

FeSO_4 exists in the crystalline state as, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. It contains one Fe^{2+} ions such as ferrous ion is present in Mohr's salt, $[\text{FeSO}_4 (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}]$ also. Both salts act as reducing agents because of the following tendency of Fe^{2+} .



Equivalent weight of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ is 278 and that of Mohr's salt is 392. KMnO_4 is an oxidizing agent because MnO_4^- ion has the following tendency.



$$\begin{aligned}\text{Equivalent mass of } \text{KMnO}_4 &= \frac{\text{Molecular weight}}{\text{Change in oxidizing state}} \\ &= \frac{158}{5} = 31.6\end{aligned}$$

Oxidation- reduction in these cases takes place in the presence of H^+ ions.

Procedure:

Titration I: Standardization of KMnO_4

Titration of standard Mohr's salt solution Vs link KMnO_4 solution

Exactly 20 ml of standard Mohr's salt solution (FAS) was pipetted out into a clean conical flask and about equal volume of dilute H_2SO_4 was added to it. This mixture was titrated against KMnO_4 solution taken in a burette. The end point was the appearance of permanent pale pink colour. The titration was repeated for concordant value. Using this value the strength of KMnO_4 solution was calculated.

S.No.	Volume of FeSO ₄ (V ₂) (ml)	Burette reading		Volume of KMnO ₄ (V ₁) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of KMnO₄ (V₁) = _____ ml

Normality of KMnO₄ (N₁) = 0.1 N

Volume of FeSO₄ (V₂) = 20 ml

Normality of FeSO₄ (N₂) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{\times 0.1}{20}$$

Normality of FeSO₄ (N₂) = _____ N

Amount of FeSO₄ present in 1 litre of the given solution

= Normality x Equivalent weight

$$= \text{_____} \times 278$$

$$= \text{_____} \text{ g}$$

The amount of FeSO₄ in the whole of the given solution

$$= \frac{\text{Normality} \times \text{Equivalent weight} \times 100}{1000}$$

$$= \frac{\text{_____}}{10}$$

$$= \text{_____} \text{ g.}$$

Titration 2: Estimation of unknown FeSO_4

Titration of unknown FeSO_4 Vs standardized KMnO_4 solution

The given unknown FeSO_4 solution was transferred to a 100 ml of standard flask using a glass rod and funnel and was then diluted upto the mark. The solution was shaken thoroughly for uniform concentration and about equal volume of dilute H_2SO_4 was added. This solution was titrated against standardized KMnO_4 solution taken in the burette. The end point was the appearance of permanent pale pink colour. The titration was repeated for concordant value. Using this value, the strength and the amount of FeSO_4 present in the whole of the given solution was calculated.

Result:

The amount of FeSO_4 present in the whole of the given solution = _____ g.

Estimation of oxalic acid using standard FeSO_4

Normality of standard $\text{FeSO}_4 = 0.1 \text{ N}$

Titration 1:

Standard FeSO_4 Vs link KMnO_4 solution

Burette solution – Link KMnO_4 solution

Conical flask solution – 20 ml of FeSO_4 + 20 ml of dilute H_2SO_4 solution

Indicator – Self

End point – Appearance of permanent pale pink colour.

S.No.	Volume of FeSO_4 (V_1) (ml)	Burette reading		Volume of KMnO_4 (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of FeSO_4 (V_1) = 20 ml

Normality of FeSO_4 (N_1) = 0.1 N

Volume of KMnO_4 (V_2) = _____ ml

Normality of KMnO_4 (N_2) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{20 \times 0.1}{\quad}$$

Normality of KMnO_4 (N_2) = _____ N

Titration II:

Unknown oxalic acid Vs Standardized KMnO_4

Burette solution – Link KMnO_4 solution

Conical flask solution – 20 ml oxalic acid + 20 ml of dilute H_2SO_4

Indicator – Self

End point – Appearance of pale pink colour.

Ex. No.:6

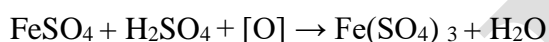
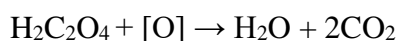
Estimation of oxalic acid using standard ferrous sulphate

Aim:

To estimate the amount of oxalic acid present in the whole of the given solution using a standard solution of ferrous sulphate containing 27.8 g in 1000 ml and an approximately decinormal solution of potassium permanganate.

Principle:

The reaction between oxalic acid and KMnO_4 and that of FeSO_4 and KMnO_4 takes place in the presence of a mineral acid, H_2SO_4 .



Equivalent weight of crystalline oxalic acid is 63 and that of FeSO_4 is 278.

Procedure:

Titration I: Standardization of KMnO_4

Titration of standard FeSO_4 Vs link KMnO_4

Exactly 20 ml of standard FeSO_4 was pipetted out into a clean conical flask. About equal volume of dilute H_2SO_4 was added and the solution was titrated against KMnO_4 solution taken in the burette. The end point was the appearance of permanent pink colour. Repeat the titration for concordant value. Using this value, the strength of the KMnO_4 solution was calculated.

S.No.	Volume of oxalic (V ₂) (ml)	Burette reading		Volume of KMnO ₄ (V ₁) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of KMnO₄ (V₁) = _____ ml

Normality of KMnO₄ (N₁) = _____

Volume of oxalic acid (V₂) = 20 ml

Normality of oxalic acid (N₂) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{\times}{20}$$

Normality of oxalic acid (N₂) = _____ N

Amount of oxalic acid present in 1 litre of the given solution

= Normality x Equivalent weight

= _____ x 63

= _____ g

The amount of oxalic acid in the whole of the given solution

$$= \frac{\text{Normality} \times \text{Equivalent weight} \times 100}{1000}$$

$$= \frac{\quad}{10}$$

= _____ g.

Titration II: Estimation of unknown oxalic acid

Titration of unknown oxalic acid Vs standardized KMnO_4

The given unknown oxalic acid solution was transferred to a 100 ml standard flask using a glass rod and funnel and was diluted upto the mark. The solution was shaken thoroughly for uniform concentration. Exactly 20 ml of the solution was pipetted out into a clean conical flask. About 20 ml of dilute H_2SO_4 was added and the mixture was heated to a bearable warmth. It was then titrated against the KMnO_4 solution taken in the burette. The end point was the appearance of permanent pale pink colour. Repeat the titration for concordant value. Using this value, the strength and the amount of oxalic acid present in the whole of the given solution was calculated.

Result:

The amount of oxalic acid present in the whole of the given solution = _____ g.

Estimation of calcium

Normality of standard $\text{CaCl}_2 = 0.1 \text{ N}$

Titration 1:

Standard CaCl_2 Vs EDTA solution

Burette solution – EDTA solution

Conical flask solution – 20 ml CaCl_2 + 100 ml of distilled water + 2 ml of buffer solution

Indicator – Erichrome Black-T

End point – Change of colour from wine red to blue.

S.No.	Volume of CaCl_2 (V_1) (ml)	Burette reading		Volume of EDTA (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of CaCl_2 (V_1) = 20 ml

Normality of CaCl_2 (N_1) = 0.1 N

Volume of EDTA (V_2) = _____ ml

Normality of EDTA (N_2) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{20 \times 0.1}{\quad}$$

Normality of EDTA (N_2) = _____ N

Titration II:

Unknown CaCl_2 Vs EDTA solution

Burette solution – EDTA solution

Conical flask solution – 20 ml of CaCl_2 + 100 ml of distilled water + 2 ml of buffer solution

Indicator – Erichrome Black-T

End point – Change of colour from wine red to blue.

Ex. No.:7

Estimation of calcium-Direct method

Aim:

To estimate the amount of calcium present in the whole of the given solution

Principle:

Calcium ion forms a wine red coloured complex with Erichrome Black-T (EBT) indicator. At the end point calcium ions are complexed with EDTA solution. The liberated metal ion indicator gives blue colour. A drop of Mg-EDTA complex is added before titration.

Procedure:

Titration I: Standardization of EDTA solution

Titration of standard CaCl_2 Vs EDTA solution

A standard solution of calcium carbonate is prepared by dissolving 1.25 g in 1:1 HCl into 250 ml standard flask using distilled water. The solution is shaken well for uniform concentration. 20 ml of this solution is pipetted out into a clean conical flask. This solution is diluted to 100 ml with distilled water. 1 ml of Mg-EDTA complex, about 2 ml of buffer solution and 2 mg of EBT indicator is added. The solution turns wine red colour. This is titrated against EDTA taken in the burette. The end point is the colour change from wine red to blue. The titration is repeated for concordant value. From this value, the strength of EDTA solution is calculated.

S.No.	Volume of CaCl ₂ (V ₂) (ml)	Burette reading		Volume of EDTA (V ₁) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of EDTA solution (V₁) = _____ ml

Normality of EDTA solution (N₁) = _____

Volume of CaCl₂ (V₂) = 20 ml

Normality of CaCl₂ (N₂) = ?

According to Volumetric Principle,

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 \times N_1}{V_2}$$

$$N_2 = \frac{\times}{20}$$

Normality of CaCl₂ (N₂) = _____ N

Amount of calcium present in 1 litre of the given solution

= Normality x Equivalent weight

= _____ x 40

= _____ g

The amount of calcium in the whole of the given solution

= $\frac{\text{Normality} \times \text{Equivalent weight} \times 100}{1000}$

= $\frac{\quad}{10}$

= _____ g.

Titration II: Estimation of unknown calcium

Titration of unknown CaCl_2 Vs standardization EDTA solution

The given unknown calcium chloride solution is made upto 100 ml in a standard flask 20 ml of this solution is pipetted into a clean conical flask and diluted to 100 ml with distilled water. 1 ml of Mg-EDTA complex, 2 ml of buffer solution and 2 mg of EBT is added. The solution turns wine red colour. This is titrated against EDTA solution taken in the burette. The end point is change of colour from wine red to blue. This titration is repeated for concordant value. From this value, the strength and the amount of calcium present in the whole of the given solution was calculated.

Result:

The amount of calcium present in the whole of the given solution = _____ g.

DEPARTMENT OF CHEMISTRY

Name of the Staff : **B. Prabha**
Department : Chemistry
Title of the Paper : **CHEMISTRY PRACTICAL-II**
Paper Code : **19BCU212**
Class : I-B. Sc-Bio-Chemistry
Year and Semester : I-Year (2019-20) and II-Semester
Batch : 2019-2022

VIVA-VOCE QUESTIONS

1. What do you mean by volumetric analysis?

Answer: The quantitative analysis which involves exact measurement of volumes of titre and titrant is termed as volumetric analysis.

2. What is titration?

Answer: The process of interaction between two solutions in a controlled way is called titration.

3. What is a titrant?

Answer: A solution which is taken in a burette is called titrant.

4. What is the end point?

Answer: The point in a titration at which the reaction gets completed is called the end point.

5. How is the completion of reaction or end point in a titration observed?

Answer: This is observed with the help of indicators.

6. What is an indicator?

Answer: The substance which denotes the completion of a reaction in a titration by colour change is called as indicator

7. Why do burettes and pipettes be rinsed with the solution for which they are to be used?

Answer: Burettes and pipettes should be rinsed before use so that they do not affect the concentration of the solutions.

8. Should a titration flask be rinsed?

Answer: No, rinsing of the flask will increase the volume more than the pipetted one.

9. Why the last drop of solution should not be blown out of a pipette?

Answer: Since the drop in the jet end is extra of the volume measured by the pipette.

10. Why is it essential to removed air bubbles from the nozzle of the burette before starting titration?

Answer: Because the air bubbles may escape any time during titration and thus affecting the volume of titrant.

11. It is customary to read lower meniscus in case of colourless solution. Why is then upper meniscus read in highly coloured solutions like KMnO_4 solution?

Answer: Because, in case of coloured solutions lower meniscus is not clearly visible.

12. What is a primary standard?

Answer: The substance whose standard solution can be prepared directly by weighing.

13. Define a standard solution?

Answer: A solution whose strength is known is called standard solution.

14. Name the indicator used for titrating a strong base and a weak acid?

Answer: Phenolphthalein.

15. Which indicator will you use while titrating a strong base and a strong acid?

Answer: Phenolphthalein and methyl orange.

16. Name the indicator for titrating a weak acid and a weak base?

Answer: No suitable indicator is available for the purpose.

17. How will you detect two bottles one containing an alkali and the other acid?

Answer: With the help of an acid base indicator.

18. Name the indicator used in the titration of oxalic acid with NaOH ? Which solution is taken in the burette and what is the end point?

Answer: Phenolphthalein. NaOH solution is taken in the burette. Appearance of pink colour at the completion of the reaction is the end point.

19. Name indicator used in the titration of sodium carbonate against hydrochloric acid.

Which solution is taken in the burette and what is the end point?

Answer: Methyl orange. HCl solution is taken in the burette. Change of colour from yellow to light pink is the end point.

20. Which indicator is used in HCl- NaOH titration?

Answer: Phenolphthalein or Methyl orange.

21. What type of titrations are permanganate titrations?

Answer: Redox titrations.

22. What is the indicator used in permanganate titrations?

Answer: Potassium permanganate itself acts as an indicator.

23. Why do we heat the solution to 60°C in case of oxalic acid against permanganate titrations?

Answer: Heating is done to enhance the rate of the reaction.

24. Ferrous sulphate- permanganate titrations is done in cold. Why not in hot?

Answer: Ferrous salt is oxidized to ferric by oxygen of air at higher temperature.

25. Why is dilute sulphuric acid added while preparing standard solution of Mohr's salt?

Answer: It is added to prevent hydrolysis of ferrous salt.