S.A.RAJA PHARMACY COLLEGE

VADAKANGULAM- 627 116

TIRUNELVELI DISTRICT

SUBJECT: PHARMACEUTICAL ANALYSIS

PRACTICAL MANUAL BOOK

LAB MANUAL
PHARMACEUTICAL ANALYSIS – I

LIST OF EXPERIMENTS FOR I-B PHARM(2017-2018)– Projected
PHARMACEUTICAL ANALYSIS I

SL. No.	DATE	NAME OF THE EXPERIMENT	MARK	REMARKS
		STANDARDIZATION		
1		SULPHURIC ACID		
2		SODIUM HYDROXIDE		
3		SODIUM THIOSULPHATE		
4		POTASSIUM PERMANGNATE		
5		CERRIC AMMONIUM SULPHATE		
		ASSAY		
6		AMMONIUM CHLORIDE BY ACID BASE TITRATION		
7		FERROUS SULPHATE BY CERIMETRY		
8		COPPER SULPHATE BY IODOMETRY		
9		CALCIUM GLUCONATE BY COMPLEXOMERY		
10		HYDROGEN PEROXIDE BY PERMANGANOMETRY		
11		SODIUM BENZOATE BY NON AQUEOUS TITRATION		
12		SODIUM CHLORIDE BY PRECIPITATION TITRATION		
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13		CONDUCTIMETRIC TITRATION OF STRONG ACID AGAINST STRONG BASE		
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15		LIMIT test for chlorides,sulphates & iron		

Experiment No. 1 **Date: ……..**

STANDARDISATION OF 0.1N H_2SO_4

AIM:

To determine the Normality of given sample of sulphuric acid.

REFERENCE:

1. Dr. G.Devalo Rao. Practical pharmaceutical inorganic chemistry
2. Anees Ahamed Siddiqui and Mohammed Ali. Practical pharmaceutical chemistry.

REQUIREMENTS:

Apparatus : conical flask, burette, pipette, standard flak

Reagents : H_2SO_4, sodium carbonate, methyl orange

PRINCIPLE:

Standardization of sulphuric acid is done by acid base titration. When sulphuric acid is allowed to react with sodium carbonate, carbon dioxide and water is produced. In this titration, to locate the endpoint methyl orange indicator is used. Appearance of the pale yellow colour is the endpoint.

$$H_2SO_4 + Na_2CO_3 \rightarrow Na_2SO_4 + CO_2 + H_2O$$

PROCEDURE:

Standardisation of 0.1 N H_2SO_4

Weigh accurately 0.15 g of anhydrous sodium carbonate previously heated at about 270°C for 1 hour. Dissolve in 100 ml water ,add methyl orange indicator, titrate until solution becomes faintly pink which persists for 30 seconds. Heat the solution to boiling cool and continue the titration until faint pink colour is no longer affected by heat.

Each ml of 0.1 M sulphuric acid is equivalent to 0.005299g of sodium carbonate

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2}$$

REPORT: The strength of the given solution of H_2SO_4 is ……..

Experiment No. 2 **Date: …….**

STANDARDISATION OF 0.1N NaOH

AIM:

To determine the Normality of given sample of sodium hydroxide.

REFERENCE:

1. Dr. G.Devalo Rao. Practical pharmaceutical inorganic chemistry.
2. Anees Ahamed Siddiqui and Mohammed Ali. Practical pharmaceutical chemistry.

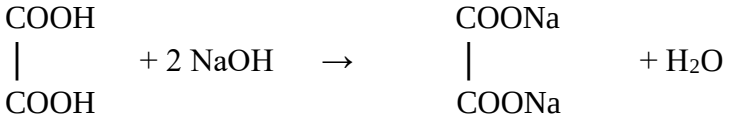
REQUIREMENTS:

Apparatus : conical flask, burette, pipette, standard flask

Reagents : NaOH, Oxalic acid, phenolphthalein

PRINCIPLE:

Standardization of sodium hydroxide is done by acid base titration. When oxalic acid is allowed to react with sodium hydroxide, sodium oxalate and water are obtained. In this titration, to locate the end point phenolphthalein indicator is used. Appearance of the pale pink colour which persists for 30 sec is the endpoint.



PROCEDURE:

Weigh accurately 0.63 g of oxalic acid into a 100 ml volumetric flask and make up to 100 ml volume with distilled water. Pipette out 20 ml solution and titrated 0.1 NaOH using phenolphthalein as indicator. Continue the titration to get the concordant value.

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2}$$

REPORT: The strength of the given solution of sodium hydroxide is……

Experiment No. 3 **Date:…….**

STANDARDISATION OF 0.1N SODIUM THIOSULPHATE

AIM:

To determine the normality of given sample of sodium thiosulphate.

REFERENCE:

1. Dr. G.Devalo Rao. Practical pharmaceutical inorganic chemistry.
2. Anees Ahamed Siddiqui and Mohammed Ali. Practical pharmaceutical chemistry.
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REQUIREMENTS:

Apparatus : Burette, Volumetric flask, Pipette,

Reagents : 0.1 N sodium thiosulphate, Potassium iodate, Potassium iodide.

PRINCIPLE:

The principle of standardization of sodium thiosulphate is based on redox iodometric titration with potassium iodate as primary standard. Potassium iodate a strong oxidizing agent is treated with excess potassium iodide in acidic media which liberates iodine which is back titrated with sodium thioslphate. Uniformity of reactions between iodine and sodium thiosulphate forms basis for utilizing the standard solution of iodine in the analysis of sodium thiosulphate and use of sodium thiosulphate in the analysis of iodine.Starch indicator is used as indicator showing disappearance of blue colour

$$KIO_3 + 5KI + 3H_2SO_4 \rightarrow 3K_2SO_4 \quad 3I_2 + 3H_2O$$

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

PROCEDURE:

Preparation of 0.1N Sodium thiosulphate

Dissolve 24.8g of sodium thiosulphate pentahydrate($Na_2S_2O_3.5H_2O$) in 800 ml of freshly boiled and cooled water and mix thoroughly by shaking for approximately 15 minutes. Make up the volume to 1000 ml.

Preparation of 0.1N Potassium Iodate

Weigh accurately about 356 mg of KIO_3 and dissolve in 100 ml distilled water

Preparation of Starch indicator

Take 1 gm of soluble starch and triturate with 5 ml of water and add it to 100 ml of Boiling water containing 10 mg of Mercuric iodide with continous stirring

Standardisation of 0.1N sodium thiosulphate

Take 10 ml of Potassium Iodate solution .Add 2 gm of Potassium Iodide and 5 ml of dilute H_2SO_4 ,keep it in dark for 10 minutes,add 2 to 3 drops of starch indicator and

titrate with sodium thiosulphte using starch solution as indicator until the blue colour is disappeared.

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$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2}$$

REPORT: The strength of the given solution of sodium thiosulphate is …….

Experiment No: 4 **Date: …….**

STANDARDISATION OF 0.1 N POTASSIUM PERMANGANATE

AIM:

To determine the Normality of given sample of 0.1N Potassium permanganate.

REFERENCE:

1. Dr. G.Devalo Rao. Practical pharmaceutical inorganic chemistry.
2. Anees Ahamed Siddiqui and Mohammed Ali. Practical pharmaceutical chemistry.

REQUIREMENTS:

Apparatus : Burette, Iodine flask, Pipette,

Reagents : Potassium Permanganate and oxalic acid.

PRINCIPLE:

The principle of standardization of potassium permanganate is based upon redox titration in which strength of an oxidizing agent is estimated by titrating it with a reducing agent and viceversa.Potassium permanganate acts as an strong oxidizing agent in acidic medium that oxidizers oxalic acid in to carbondioxide . Known strength of oxalic acid is titrated directly with potassium permanganate .End point can be detected with appearance of permanent pink colour potassium permanganate acts as self indicator

$2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$

PROCEDURE:

Preparation of 0.1 N Potassium permanganate solution

Dissolve 3.2g of potassium permanganate in 1000ml of water, heat on a water bath for 1 hour, allow to stand for 2 days. Filter the solution through glass wool.

Preparation of 0.1 N Oxalic acid:

6.3 gm of oxalic acid dissolve in 1000 ml of distilled water

Standardisation of 0.1N Potassium permanganate

Take 20 ml of Oxalic acid solution .add 5 ml of 1m sulphuric acid.warm the mixture to about 70^oC .titrate with potassium permanganate solution taken in the burette. End point is appearance of pink colour that persist for 30 sec

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2}$$

REPORT: The strength of the given solution of potassium permanganate is**……**

Experiment No. 5 **Date: ……**

STANDARDISATION OF 0.1 M CERIC AMMONIUM SULPHATE

AIM:

To determine the Normality of given sample of 0.1 M Ceric ammonium sulphate.

REFERENCE:

1. Dr. G.Devalo Rao. Practical pharmaceutical inorganic chemistry.
2. Anees Ahamed Siddiqui and Mohammed Ali. Practical pharmaceutical chemistry.

REQUIREMENTS:

Apparatus : Burette, Conical flask, Pipette,

Reagents : Ceric ammonium sulphate, Sulphuric acid, Arsenic trioxide, Sodium hydroxide, .Sulphuric acid

PRINCIPLE:

Ceric ammonium sulphate is titrated with Arsenic trioxide solution (Primary standard) in presence of sodium hydroxide, and osmic acid solution.

$As_2O_3 + 2NaOH \rightarrow 2NaAsO_2 + H_2O$

$NaAsO_2 + 2H_2O \rightarrow NaH_2AsO_4 + 2H^+ + 4e^-$

$Ce^{4+} + e^- \rightarrow Ce^{3+}$

PROCEDURE:

Preparation of 0.1 M Ceric ammonium sulphate

Dissolve 65 g of ceric mmonium sulphate with the aid of gentle heat in a mixture of 30 ml of sulphuric acid and 500ml of water. Cool,filter dilute to 1000ml with water.

Standardization 0f 0.1 M Ceric ammonium sulphate

Weigh accurately 0.2g of arsenic trioxide previously dried at 105 C for 1 hour and transferred to a 500ml conical flask. Wash the inner walls of the flask with 25 ml of 8 %w/v solution of sodium hydroxide dissolve and add 100 ml of water. Add 30 ml of dilute sulphuric acid , 0.15 ml of osmic acid, 0.1 ml of ferroin sulphate and titrate with ceric ammonium sulphate until the pink colour changes to pale blue

Each ml of ceric ammonium sulphate is equivalent to 0.004946 g of arsenic trioxide.

Molarity of cerric ammonium sulphate = $\frac{W \times RM}{V \times E}$

W=Weight of arsenic trioxide

RM =Required molarity

E = Equivalent weight factor

V = Volume of cerric ammonium sulphate

REPORT: The strength of cerric ammonium sulphate is ……

ASSAY

Experiment No. 6 **Date: ……**

ASSAY OF AMMONIUM CHLORIDE

AIM:

To determine the percentage purity of given sample of ammonium chloride.

REFERENCE:

1. Indian Pharmacopoeia -1985, volume I,
2. Practical pharmaceutical inorganic chemistry, Dr. G.Devalo Rao
3. Practical pharmaceutical chemistry by Anees Ahamed Siddiqui and Mohammed Ali

CHEMICALS USED:

Ammonium chloride, NaOH, Oxalic acid, formaldehyde

PRINCIPLE:

Ammonium chloride is assayed by indirect assay method. The sample is dissolved in water and treated with previously neutralised formaldehyde solution.(Methyleneimine is formed which polymerises to form Hexamine). This results in the quantitative liberation of HCl equivalent to NH_4Cl. The liberated HCl is titrated with the standard solution of NaOH using phenolphthalein as indicator, appearance of pale pink colour is the indication of end point.

$$4NH_4Cl + 4H_2O \rightarrow 4NH_4OH + 4HCl$$
$$4\ NH_4OH + 6HCHO \rightarrow (CH_2)6N_4 + 10\ H_2O$$
$$4HCl + 4NaOH \rightarrow 4NaCl + 4H_2O$$

PROCEDURE:

Standardisation of 0.1 NaOH

Weigh accurately 0.63 g of oxalic acid into a 100 ml volumetric flask and make up to 100 ml volume with distilled water. Pipette out 20 ml solution and titrated with 0.1 M NaOH using phenolphthalein as indicator. Continue the titration to get the concordant value.

Assay of NH_4Cl

Weigh 0.1gm of NH_4Cl dissolve in 20 ml of H_2O and add a mixture of 5 ml of previously neutralized formaldehyde solution and 20 ml water. After 2 minutes the contents of the conical flask is titrated against 0.1N NaOH using phenolphthalein as indicator. End point is the appearance of permanent pale pink colour.
Each ml of 0.1N NaOH is equivalent to 0.005349 gm of NH_4Cl.

Percentage purity can be determined by the following formula.

$$\% = \frac{\text{Titre value x Equivalent wt factor x Normality of NaOH(actual)}}{\text{Weigh of sample x Normality of titrant (expected)}} \times 100$$

REPORT: The percentage purity of the given sample of ammonium chloride is……

ASSAY

Experiment No. 7 **Date: ……**

ASSAY OF FERROUS SULPHATE

AIM:

Determine the percentage purity of given sample of ferrous sulphate.

REFERENCES:

1. Practical pharmaceutical inorganic chemistry by Dr. Deval Rao

CHEMICALS USED:

Ferrous suphate, Cerric ammonium Sulphate, dil H_2SO_4.

PRINCIPLE:

$FeSO_4$ is an example of reducing agent and In the presence of dil H_2SO_4, $FeSO_4$, is titrated with ceric ammonium sulphate using ferroin as indicator which gives a colour change from red to pale blue. Cerri ammonium sulphate is a powerful oxidizing agent and it is used only in acidic media because if the solution is neutral cerric hydroxide precipitates out. Cerric ion solution itself has intense yellow colour can be used as self indicator, but to increase the sensitivity of endpoint detection internal redox indicator like ferroin is added

$$Fe^{2+} \rightarrow Fe^{3+} + e^-$$
$$Ce^{4+} + e^- \rightarrow Ce^{3+}$$
$$Fe^{2+} + Ce^{4+} \rightarrow Fe^{3+} + Ce^{3+}$$

PROCEDURE:

Preparation of 0.1 M Ceric ammonium sulphate

Dissolve 65 g of ceric mmonium sulphate with the aid of gentle heat in a mixture of 30 ml of sulphuric acid and 500ml of water. Cool,filter dilute to 1000ml with water.

Standardization 0f 0.1 M Ceric ammonium sulphate

Weigh accurately 0.2g of arsenic trioxide previously dried at 105 C for 1 hour and transferred to a 500ml conical flask. Wash the inner walls of the flask with 25 ml of 8 %w/v solution of sodium hydroxide ,dissolve and add 100 ml of water. Add 30 ml of dilute sulphuric acid , 0.15 ml of osmic acid, 0.1 ml of ferroin sulphate and titrate with ceric ammonium sulphate until the pink colour changes to pale blue

Each ml of ceric ammonium sulphate is equivalent to 0.004946 g of arsenic trioxide.

Molarity of cerric ammonium sulphate = $\frac{W \times RM}{V \times E}$

W=Weight of arsenic trioxide

RM =Required molarity

E = Equivalent weight factor

V = Volume of cerric ammonium sulphate

Assay of $FeSO_4$

Add 0.5 g of $FeSO_4$ and 30 ml water .Add 20 ml of 1m sulphuric acid shake well, then titrate with 0.1N ceric ammonium sulphate solution using 0.1 ml ferroin sulphate as indicator until red colour disappears.

Each ml 0.1M Ceric ammonium sulphate is equivalent to 0.01519 gm of Ferrous sulphate.

Percentage purity can be determined by the following formula.

$$\% = \frac{\text{Titre value x Equivalent wt factor x Normality of titrant(actual)}}{\text{Weigh of sample x Normality of titrant (expected)}} \times 100$$

REPORT: The percentage purity of Ferrous sulphate is…….

ASSAY

Experiment No. 8 **Date: ……..**

ASSAY OF COPPER SULPHATE

AIM:

Determine the percentage purity of given sample of copper sulphate sulphate.

REFERENCES:

1. Practical pharmaceutical inorganic chemistry by Dr. Deval Rao

REQUIREMENTS

Apparatus: Iodine flask, Burette, conical flask

Reagents : Copper suphate, 0.1N Sodium thiosulphate solution, starch solution, 0.1 N KIO_3 solution

PRINCIPLE:

This is an iodometry type of titration. It depends upon the instability of cupric iodide which is formed in the reaction between $CuSO_4$ and potassium iodide with the liberation of free iodine. When $CuSO_4$ was allowed to react with KI in the presence of acetic acid, cupric iodide is formed.

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

CUPRIC IODIDE

The cupric iodide formed in the above reaction is unstable, so it decompose to give cuprous iodide with the liberation of free iodine.

$$2CuI_2 \rightarrow 2Cu_2I_2 + I_2$$

CUPROUS IODIDE

The liberated iodine is titrated with 0.1N sodium thiosulphate using starch solution as indicator.

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

Sodium tetrathionate

PROCEDURE:

Preparation of 0.1N Sodium thiosulphate

Dissolve 24.8 g of sodium thiosulphate pentahydrate in 800 ml of boiled and cooledwater and mix thoroughly by shaking for approximately 15 minutes. Make up the volume to 1000 ml.

Standardisation of 0.1N sodium thiosulphate

Take 10 ml of Potassium Iodate solution .Add 2 gm of Potassium Iodide and 5 ml of dilute H_2SO_4 ,keep it in dark for 10 minutes,add 2 to 3 drops of starch indicator and titrate with sodium thiosulphte using starch solution as indicator until the blue colour is disappeared.

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$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2}$$

Assay of copper sulphate

Weigh accurately about 1 gm and dissolve in 50 ml of water, add 3gm of KI, 5 ml of acetic acid and titrate the liberated iodine with 0.1n sodium thiosulphate using starch solution as indicator. Continue the titration till a faint blue colour remains, add 2g of potassium thiocyanate stir well and continue the titration until the blue colour disappears.

Each ml of 0.1N sodium thiosulphate = 0.02497 gm of $CuSO_4$

Percentage purity of the given Copper sulphate = $\frac{VxE \times AN \times 100}{W \times RN}$

V =Volume of $Na_2S_2O_3$

E = Equivalent weight factor

AN= actual normality of $Na_2S_2O_3$

W = weight of sample

RN = Required Normality of $Na_2S_2O_3$

REPORT : The percentage purity of the given sample of copper sulphate is……..

Experiment no.9 **Date:……..**

ASSAY OF CALCIUM GLUCONATE

AIM:-

To perform the assay of given sample of calcium gluconate

REFERENCE:-

I.p volume-I-1996 page no: 129.

REQUIREMENTS:-

Apparatus : Conical flask, standard flask ,Burette,Pipette, Funnel

Reagents: Calcium gluconate**,**eriochrome black T indicator**,**Buffer**,**Disodium EDTA

PRINCIPLE

Calcium salt is assayed by titration with 0.05m EDTA using ph buffer and eriochrome black T as indicator. EDTA forms calcium EDTA complex, and the color change of indicator, is from wine red to blue at the end point.This is based on Replacement titration.Magnesium forms complex with indicator

Mg^{2+} + indicator → Mg- ind

This magnesium indicator complex is more stable than Calcium indicator complex.Therefore Calcium has no effect with magnesium indicator complex.On titration with disodium edetate calcium edetate complex is formed

Ca^{2+} + EDTA → Ca-EDTA

When calcium is completely consumed ,next drop of EDTA breaks the mg-indicator complex,thus liberating the free indicator ,which gives the colour at the endpoint

PROCEDURE

Standardization of 0.05 M EDTA

Take 10 ml of cacl2 ,add 5 ml of ammonia buffer (PH 10),2 drops of nindicator and titrate with EDTA(0.05 m)

Assay of calcium gluconate

Weigh 0.5g calcium gluconate and dissolve it in 50ml of warm water. and add 5ml of 0.05m magnesium sulphate and 10ml of strong ammonia-ammonium chloride buffer solution ,titrate with 0.05m disodium EDTA using mordant black II as indicator .The endpoint is appearance of blue colour

Each ml of 0.05m disodium EDTA is equivalent to 0.02242g of calcium gluconate .

REPORT

The percentage purity of calcium gluconate is……..

Experiment : 10 **Date…….**

ASSAY OF HYDROGEN PEROXIDE

AIM

To perform the assay of given sample of H_2O_2

REQUIREMENTS :

Apparatus : conical flask , burette, pipette

Reagents : hydrogen peroxide, potassium permanganate, sulphuric acid

PRINCIPLE

H2O2 is usually encountered in the form of an aqueous solution containing 6%,12%,30% volume and H_2O_2 is frequently is known as 20,40 and 100ml volume. This terminology is based on the volume of O2 librated when the solution is decomposed by boiling. The assay is done by volumetrically by oxidation reduction titration. When H_2O_2 is being titrated in an acidic medium $KMnO_4$ is used as reducing agent.

$2KMnO_4 + 3H_2SO_4 + 5\ H_2O_2 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 5O_2$

H_2O_2 is oxidized to give water and O_2

$2\ H_2O_2 \xrightarrow{[O]} 2\ H_2O + O_2$

In this titration $KMnO_4$ act as a self indicator 68.04gm of H_2O_2 gives 22,400ml O_2 1gm of H_2O_2 is equivalent to 329.2 ml of O_2

PROCEDURE

Preparation of 0.1 N Potassium permanganate solution

Dissolve 3.2g of potassium permanganate in 1000ml of water, heat on a water bath for 1 hour, allow to stand for 2 days. Filter the solution through glass wool.

Standardisation of 0.1 M Potassium permanganate

To 25 ml of potassium permanganate solution in a glass stoppered flask, add 2g of potassium iodide, followed by 10 ml of sulphuric acid. Titrate the liberated iodine with 0.1M Sodium thiosulphate using 3 ml of starch solution , as an indicator.

$$N_1V_1 = N_2V_2$$

$$N_2 = \frac{N_1V_1}{V_2}$$

Assay of hydrogen peroxide

To 1ml of H_2O_2 add 20 ml of 1M H_2SO_4.titrate against 0.1N $KMnO_4$

Each ml of 0.02M $KMnO_4$ is equivalent to 0.001701 gm of H_2O_2

REPORT:

The given sample of H_2O_2 solution contains…….

Experiment no.11 **Date:**

ASSAY OF SODIUM BENZOATE BY NON-AQUEOUS TITRATION

AIM:-

To find out the percent age purity of given sample sodium benzoate.

REFERRENCE:-

1. Pharmaceutical analysis. Anees A Siddiqui

REQUIREMENTS

Apparatus: Conical flask, Burette

Reagents : Sodium benzoate, 0.1 M $HClO_4$, Potassium hydrogen phthalate, Glacial acetic acid

PRINCIPLE:-

sodium benzoate is the sodium salt of benzoic acid. It is widely used as chemical preservative in carbonated and still beverages, syrups, olives, sauces, relishes, jellies jams and pastry low fat salad dressing, fruit salads, prepared salads and in storage of vegetables.

It is a basic compound . On adding Glacial acetic acid and then titrating with strong acid perchloric acid ,this Glacial acetic acid acts as a base.

$$CH3COOH + HClO_4 \rightarrow CH_3COOH2+ + ClO_4^-$$

Assay of sodium benzoate

weigh 0.25g sodium benzoate and dissolved in 20ml Anhydrous glacial acetic acid in a 250 ml volumetric flask.mix it well warm to 50^0c. Add 2-3 drops of Crystal violet indicator. Titrate with 0.1M $HClO_4$. While mixing the water and ether layer well by shaking until a light green color persists in the water layer.

Each ml of 0.1M $HClO_4$ is equivalent to 0.01441g of sodium benzoate.

REPORT:

The percentage purity of sodium benzoate is…….

Experiment no.12 **Date:……**

ASSAY OF SODIUM CHLORIDE BY PRECIPITATION TITRATION

AIM:-

To find out the percent age purity of given sample sodium chloride.

REFERRENCE:-

Practical pharmaceutical analysis. G.Devala rao

REQUIREMENTS

Apparatus: Conical flask, Burette

Reagents : Sodium chloride, Potassium chromate.

PRINCIPLE:-

sodium chloride is an example of electrolyte replenisher. It is assayed by Mohr's method. The sample is dissolved in water and titrated against a standard solution of silver nitrate using potassium chromate as indicator. At the end point due to the complete precipitation of chloride as silver chloride, a slight excess of silver nitrate reacts with potassium chromate to give a red colored silver chromate.

$NaCl + Ag NO_3 \rightarrow NaNO_3 + AgCl$

$K_2CrO_4 + 2AgNO_3 \rightarrow Ag_2CrO_4 + 2 KNO_3$

This method is based on the fact that silver halide is more insoluble than silver chromate. Hence as long as there is any chloride left in the solution, no silver chromate is formed. Even though if formed, will immediately change to silver chloride according to the following equation

$Ag_2CrO_4 + 2Cl^- \rightarrow 2AgCl + CrO_4^{-2}$

Assay of sodium chloride

Weigh accurately 0.1g sodium chloride previously dried at 110 C for 2 hours and dissolve in 5 ml of water. Add 2 drops of potassium chromate as indicator and titrate with silver nitrate solution until a permanent reddish brown precipitate is formed. Repeat the titration for concordant values.

Each ml of 0.1M silver nitrate is equivalent to 0.005844 g of sodium chloride

REPORT

The Percentage purity of sodium chloride is……..

ELECTRO-ANALYTICAL METHODS

Experiment :13 **Date:…….**

CONDUCTOMETRIC TITRATION OF STRONG ACID WITH STRONG BASE

AIM

To perform conductometric titration of hydrochloric acid with sodium hydroxide and to determine the end point.

REFERENCE

1. Bhoomika R Gpoal. Hardik G Bhatt. Mayur M Patel. Pharmaceutical analysis –I. Page No: 99-100.

REQUIREMENTS

Apparatus : Beaker, Pipette, Volumetric flask, conductivity cell

Reagents : 0.01 M Hydrochloric acid, 0.01 M Sodium hydroxide

PRINCIPLE

The solution of HCl shows fairly significant conductivity mainly due to two species H+ and Cl-. For each amount of NaOH added, an equivalent amount of H+ ion is consumed according to the following reaction.

$H+ + NaOH == Na+ + H_2 O$

The highly conducting H+ cations are effectively replaced by the relatively poorly conducting Na+ ions and consequently the conductance decreases. This continues until sufficient NaOH has been added to react with all the H+ ions present and the mixture contains only Na+ and Cl- ions in water

$HCl + NaOH == H_2 O + NaCl$

This is called equivalence point. Further addition of NaOH simply augments the quantity of Na+ and OH- ion in the mixture and increase the conductance due to higher OH- anions. As a result, the conductomeric titration curve (conductance vs. volume of NaOH) has a minimum at the equivalence point. The position of the equivalence Point can be localized as the point of inter section of two straight lines, a descending segment before and an ascending segment after the minimum observed. Usually in this experiment, the concentration of solution in the burette should be approximately 10 times than that of the other solution, under these conditions the change in concentration due to dilution of the solution during the titration , will have minimum effect on the conductance measurements.

PROCEDURE

Transfer 50 ml 0.01M hydrochloric acid solution in a 100 ml beaker. Immerse the conductivity cell in the solution in the beaker such that the tip of the electrode remains dipped in the solution. Measure the conductance for the pure HCl solution. Titrate with 0.1 M NaOH solution using conductometer, adding aliquot of 0.5ml of 0.1 M NaOH solution each time. Remember to mix well after each addition of titrant before measuring the conductance. Continue in this way until you have measured about six points beyond the endpoint. Plot the graph of volume of titrant against conductance.

REPORT

From graph, endpoint of the conductometric titration is found to be ……. ml.

Experiment : 14 **Date :**

POTENTIOMETRIC TITRATION OF A STRONG ACID WITH A STRONG BASE

AIM

To perform the titration of 0.1 N HCl with 0.1 N NaOH by potentiometry and to locate the end point.

REFERRENCE

1. G.devala rao. Pharmaceutical analysis. Page No: 136

REQUIREMENTS

Apparatus : pH meter, Magnetic stirrer, Beaker

Reagent : 0.1 N HCl, 0.1 N NaOH, Distilled water.

PRINCIPLE

Potentiometric determination of the end point depends on the fact that the potential across the two electrodes (reference and indicator) immersed in the solution changes sharply at the equivalence or end [point. This change is similar to the colour change by an indicator in usual method. But the potentiometric method is more accurate. These titrations are useful when no suitable colour indicators are available. Equivalence point can be accurately found out after plotting normal plot (volume of titrrant vs. potential)

PROCEDURE

In a 250 ml beaker add 10 ml of 0.1N HCl and add around 100 ml of waater of dip the electrodes properly. Keep the beaker on magnetic stirrer. Note the potential without adding any alkali slowly add with stirring, known volumes of 0.1 N NaOH solution, and note the potentials. enter the values in a tabular form. from the data (volume of titrant vs. potential). Plot the graph and calculate the end point.

REPORT

The end pont in the titration of 0.1 N HCl with 0.1 N NaOH by potentiometry is