East Point College of Pharmacy

East Point Campus, Jnana Prabha, Virgo
Nagar PostBengaluru – 560049, Karnataka

Approved
by
Pharmacy Council of India, New Delhi

Affiliated
to
Rajiv Gandhi University of Health Sciences
Karnataka
Bengaluru – 560041
India

LAB MANUAL

PHARMACEUTICAL ANALYSIS-I

B. PHARM 1st SEMESTER

EAST POINT COLLEGE OF PHARMACY
East Point Campus, Jnana Prabha, Virgo Nagar Post,
Bengaluru – 560049, Karnataka

B Pharmacy

Program Outcomes (PO's)

PO 1- Pharmacy Knowledge

Possess knowledge and comprehension of the core and basic knowledge associated with theprofession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.

PO 2- Planning Abilities

Demonstrate effective planning abilities including time management, resource management,delegation skills and organizational skills. Develop and implement plans and organize workto meet deadlines.

PO 3- Problem analysis

Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, whilesolving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions

PO 4- Modern tool usage

Learn, select, and apply appropriate methods and procedures, resources, and modernpharmacy-related computing tools with an understanding of the limitations.

PO 5- Leadership skills

Understand and consider the human reaction to change, motivation issues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and wellbeing.

PO 6- Professional Identity

Understand, analyse and communicate the value of their professional roles in society (e.g.health care professionals, promoters of health, educators, managers, employers, employees).

PO 7- Pharmaceutical Ethics

Honor personal values and apply ethical principles in professional and social contexts. Demonstrate behaviour that recognizes cultural and personal variability in values,communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions

PO 8- Communication

Communicate effectively with the pharmacy community and with society at large, such as,being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions

PO 9- The Pharmacist and society

Apply reasoning informed by the contextual knowledge to assess societal, health, safety andlegal issues and the consequent responsibilities relevant to the professional pharmacy practice.

PO 10- Environment and sustainability

Understand the impact of the professional pharmacy solutions in societal and environmentalcontexts, and demonstrate the knowledge of, and need for sustainable development.

PO 11- Life-long learning

Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-access and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

EAST POINT COLLEGE OF PHARMACY
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Programme Specific Outcomes (PSO's)	
PSO 1	Acquire a thorough foundational knowledge in pharmaceutical sciences, including pharmacology, pharmaceutics, medicinal chemistry, and pharmacognosy, to excel in further academic pursuits
PSO 2	Gain expertise in the application of contemporary pharmaceutical techniques and technologies, enhancing employability across various sectors including the pharmaceutical industry, academia, and research institutions.
PSO 3	Equip with entrepreneurial skills and knowledge of pharmaceutical business management, including market analysis, product development, regulatory affairs, and financial planning, to initiate and run successful ventures in the pharmacy sector

Course Outcomes (CO's)	
Code: BP108P Pharmaceutical Analysis I	
CO 1	Perform limit test and identify the impurities in the given compounds, preparation of standard solutions
CO 2	Standardization of the secondary standard solutions by the use of primary standard solutions.
CO 3	Determine the percentage purity of drugs by volumetric analysis
CO 4	Determine the percentage purity of drugs by electrochemical analysis

Experiment No. 1

LIMIT TEST FOR CHLORIDES I.P

Aim: To perform the Limit test for Chlorides

References:

- Indian Pharmacopoeia 2007
- Bentley and Driver's text book of pharmaceutical chemistry. Page No: 112
- Perform the limit test for chloride on the given sample and report on its standard.

Principle:

The limit test for chlorides is based on the reaction between silver nitrate and soluble chlorides forming a precipitate of silver chloride which is insoluble in dilute nitric acid.

$$Cl^- + AgNO_3 \longrightarrow \downarrow AgCl + NO_3^-$$

Chloride ion — Silver nitrate — Silver chloride — Nitrate ion

A specified amount of drug is dissolved in water, acidified with dil. nitric acid and treated with silver nitrate solution. The opalescence produced depends upon the amount of chlorides present in the sample. It is compared with the opalescence produced in a standard solution containing the prescribed quantity of chloride similarly treated. If the opalescence in the sample is less than that in the standard, it passes the test and is declared as standard. If it is more, it fails the test and is declared as substandard. Nitric acid is included to convert the insoluble chloride to soluble one and to minimize the solubility of slightly soluble silver chloride.

Procedure:

Preparation of chloride standard solution (25 ppm Cl^-)

Dilute 5 volume of 0.0824% w/v solution of sodium chloride to 100 volume with distilled water and mixing well. Take two 50 ml Nessler cylinders; label one as "Test" and the other as "Standard".

Test	Standard
Dissolve the specified quantity of the substance in water or prepare a solution as directed in individual monograph and transferred to a Nessler cylinder	Pipette out 10 ml of chloride standard solution (25 ppm Cl^-) into the Nessler cylinder and add 5ml water
Add 10 ml of dilute nitric acid	Add 10 ml of dilute nitric acid
Dilute to 50 ml with water	Dilute to 50 ml with water
Add 1 ml of 0.1 M silver nitrate solution	Add 1 ml of 0.1 M silver nitrate solution
Stir immediately with a glass rod and allow to stand for five minutes protected from light (keep in a dark place)	Stir immediately with a glass rod and allow to stand for five minutes protected from light (keep in a dark place)

Compare the opalescence produced in the test with that of the standard opalescence against a black back ground. Observe whether the test has greater or lesser opalescence than the standard.

Observation:

Sample-I

Sample-II

Report:

Sample-I

Sample-II

Experiment No. 2

LIMIT TEST FOR SULPHATES

Aim: To Perform the limit test for sulphate on the given sample and report on its standard.

References:

- Indian Pharmacopoeia 2007
- Text book of pharmaceutical inorganic chemistry by V.N. Rajasekaran. Page No: 401

Principle:

This is based on the reaction between barium chloride and soluble sulphate which forms the turbidity of barium sulphate.

$$SO_4^{2-} + BaCl_2 \longrightarrow \downarrow BaSO_4 + 2Cl^-$$

Sulphate ion Barium chloride Barium sulphate Chloride ion

A specified amount of sample is dissolved in water and treated with barium chloride. The turbidity is produced by the precipitation of barium sulphate. This is compared with the turbidity produced in a standard containing a known quantity of sulphate and similarly treated. The test substance passes the limit test if the turbidity in it is less intense than that in the standard. If the turbidity is found to be more, then it fails the test.

Acetic acid makes the solution acidic. Barium chloride forms turbidity with sulphate ion. Ethanolic standard solution contains potassium sulphate and alcohol. The addition of potassium sulphate increases the sensitivity of the test. Alcohol prevents super saturation and a more uniform turbidity is formed.

Procedure:

Preparation of sulphate standard solution (10 ppm SO_4^{2-}):

0.1089g of potassium sulphate dissolves in small quantity of water and make up the volume to 100ml.

Ethanolic sulphate standard solution (10ppm):

Test	Standard
Take 1ml of 25% barium chloride solution	Take 1ml of barium chloride solution
Add 1.5ml of ethanolic sulphate standard solution	Add 1.5ml of ethanolic sulphate standard solution
Mix and allow to stand for 2min	Mix and allow to stand for 2min
Add test solution as per monograph or add the solution of substance in 15ml of water	Add 1ml of sulphate standard solution
0.15ml of 5M acetic acid and make up to 50ml with water.	0.15ml of 5M acetic acid and make up to 50ml with water.
Stir immediately and keep aside for 5 min	Stir immediately and keep aside for 5 min

Dilute 1ml of 0.181% w/v solution of potassium sulphate in 30% ethanol to 100ml with 30% ethanol. Take two 50 ml Nessler cylinders. Label one as 'Test' and the other as 'Standard'

Compare the intensity of turbidity produced in test with that of standard opalescence against a black back ground. Observe whether the test has greater or lesser turbidity than the standard.

Observation:

Sample-I

Sample-II

Report:

Sample-I

Sample-II

Experiment No. 3

LIMIT TEST FOR IRON

Aim: To Perform the limit test for iron on the given sample and report on its standard.

References:

- Bentley and Driver's text book of pharmaceutical chemistry. Page No: 112
- Indian Pharmacopoeia 2007

Principle:

The test depends upon the reaction between ferrous iron and thioglycollic acid in the presence of ammonia produce a pale pink to deep reddish purple colour.

$$Fe^{3+} + \begin{array}{l} CH_2COOH \\ | \\ 2Fe^{2+} + 2H^{+}\ SH \end{array} \longrightarrow \begin{array}{l} SCH_2COOH \\ | \\ SCH_2COOH \end{array} +$$

A specified amount of drug is dissolved in water, treated with citric acid and thioglycollic acid then it is made alkaline with ammonia solution. The purple colour produced is compared with the standard. If the intensity of purple colour produce in sample is less than the standard, the sample complies the limit test for iron otherwise it fails the limit test for iron.

Thioglycollic acid reduces ferric iron to ferrous iron and produces the purple coloured complex (ferrous thioglycollate) with ferrous iron. **Citric acid** prevents the precipitation of ferrous hydroxide by forming soluble iron citric acid complex. **Ammonia solution m**akes the solution alkaline. Ferrous thioglycollate is colourless in neutral or acid

solution. The colour develops only in the presence of alkali. It is stable in the absence of air but fades when exposed to air due to oxidation to the ferric compound.

Procedure:

Preparation of standard solution

Test	Standard
Dissolve a specified quantity of the substance in 20 ml of water prescribed in the monograph in a Nessler cylinder.	Dilute 2 ml of standard iron solution (20 ppm Fe) with 20 ml of water in a Nessler cylinder
Add 2 ml of a 20% w/v solution of iron-free citric acid and 0.1 ml of thioglycollic acid and mix	Add 2 ml of a 20% w/v solution of iron- free citric acid and 0.1 ml of thioglycollic acid and mix
Make alkaline with iron-free ammonia solution	Make alkaline with iron-free ammonia solution
Dilute to 50 ml with water	Dilute to 50 ml with water
Allow to stand for five minutes	Allow to stand for five minutes

Dissolve 0.1726g of ferric ammonium sulphate in 10ml of 0.1M sulphuric acid and make up the volume to 1000ml with water.

Take two 50 ml Nessler cylinders. Label one as ‘Test’ and other as ‘Standard’.
Compare the intensity of colour produced in test with that of standard colour against a white back ground. Observe whether the test has greater or lesser intensity of colour than the standard.

Observation:

Sample-I

Sample-II

Report:

Sample-I

Sample-II

Experiment No. 4

LIMIT TEST FOR ARSENIC

Aim:

To Carry out the limit test for arsenic on the given sample and report its standard.

References:

1. Indian Pharmacopoeia 2007

Principle:

This is based on the reaction between arsine gas and mercuric chloride forms a yellow stain. Sample solution is prepared as directed in monograph and placed in Gutzeit apparatus. Potassium iodide, granulated zinc, hydrochloric acid, stannous chloride are added. The arsenic present in the substance is converted to either arsenious acid (if the arsenic is trivalent) or arsenic acid (if the arsenic is pentavalent). Then it is further treated with a reducing agent stannous chloride or sulphurous acid. All the arsenic acid is reduced to arsenious acid. In I.P stannated hydrochloric acid is added.

$$H_3AsO_4 \longrightarrow H_3AsO_3$$

Arsenic acid Arsenious acid

The arsenious acid further reduced to arsine (arsenious hydride) by nascent hydrogen which is produced by the action of granulated zinc and hydrochloric acid.

$$H_3AsO_3 + 6[H] \longrightarrow AsH_3 + 3H_2O$$

Arsenious acid Arsine

Arsine comes into contact with dry paper saturated with mercuric chloride, produces yellow or brown stain.

The intensity of the stain is compared in day light with a standard stain which is similarly prepared but taking a specified quantity of standard dilute arsenic solution in place of the substance. If the test stain is less than the standard stain, the sample passes the test. Otherwise it fails the test. Potassium iodide, stannous chloride, zinc *a*ct as a reducing agent and reduces arsenic acid to arsenious acid. Nascent hydrogen reduces arsenious acid to arsine.

Procedure:

Preparation of arsenic standard solution (10 ppm Arsenic)

Dissolve 0.330g of arsenic trioxide in 5ml of 2M sodium hydroxide and dilute to 250ml with water. Dilute 1 volume of this solution to 100 volumes with water. Take two 120ml wide-mouthed bottles or conical flask fitted with the attachments and label one as Test and the other as Standard.

Test	Standard
Weigh accurately 10g of the sample and dissolve in 50ml of water. Transfer to the bottle or flask.	1ml of arsenic standard solution (10 ppm) is taken in the bottle or flask. Add 50ml of water
Add 10ml of stannated hydrochloric acid AsT	Add 10ml of stannated hydrochloric acid AsT
Add 5ml of 1M potassium iodide and 10g of zinc AsT.	Add 5ml of 1M potassium iodide and 10g of zinc AsT.
Place the cork immediately over the bottle with the attachments and immerse the bottle in water bath at a suitable temperature.	Place the cork immediately over the bottle with the attachments and immerse the bottle in water bath at a suitable temperature.
Allow the reaction to go on for 40 min	Allow the reaction to go on for 40 min

Compare the depth of colour in the test stain with the standard stain by day light. Observe whether the colour produced in test is more intense or less intense than the standard.

Report:

Experiment No. 5

PREPARATION AND STANDARDIZATION OF 0.1N SODIUM HYDROXIDE

Aim: To prepare and standardize 0.1M sodium hydroxide

Chemicals required:

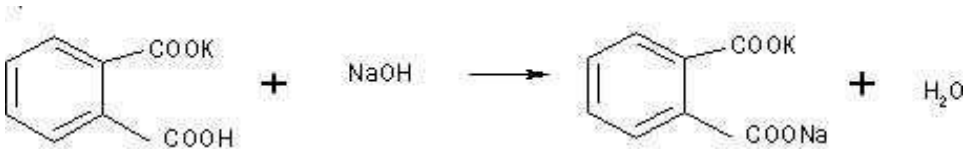
- Potassium hydrogen phthalate
- Sodium hydroxide
- Phenolphthalein
- Distilled water

Principle:

0.1M Sodium hydroxide is prepared by dissolving sodium hydroxide pellets in carbon dioxide free water. Sodium hydroxide is a secondary standard which is standardized by using various primary standards such as oxalic acid, sodium acetate and benzoic acid and potassium hydrogen phthalate.

In this experiment sodium hydroxide is standardized by using potassium hydrogen phthalate as a primary standard. Sodium hydroxide reacts with potassium hydrogen phthalate and gives potassium sodium phthalate.

Reaction:



Potassium hydrogen Phthalate

potassium sodium phthalate

Procedure:

Preparation of 0.1M sodium hydroxide

Dissolve 0.42 g of sodium hydroxide in sufficient carbon dioxide-free water to produce 100 ml.

Standardization of 0.1M sodium hydroxide

Weigh accurately about 0.5 g of potassium hydrogen phthalate, previously powdered and dried at 120° for 2 hours, and dissolve in 75 ml of carbon dioxide-free water. Add 0.1 ml of phenolphthalein solution and titrate with the sodium hydroxide solution until a permanent pink colour is produced. Each ml of 1M sodium hydroxide is equivalent to 0.02042 g of $C_8H_5KO_4$.

References:

1. Practical pharmaceutical chemistry by M.M. Alam
2. Indian Pharmacopeia 1996 –Volume-2

Report: The strength of the prepared 0.1M sodium hydroxide solution was found to be ------

Experiment No. 6

PREPARATION AND STANDARDIZATION OF 0.1N SULPHURIC ACID

Aim: To prepare and standardize 0.5M sulphuric acid solution.

Requirements:

a) Apparatus: Conical flask, Burette, Pipette, Water bath, Burner, Measuring Cylinder, Beaker, Dropper , Glass rod, Butter paper, .Etc.

b) Chemicals: Sulphuric acid, Anhydrous sodium carbonate, Methyl red and Distilled Water.

c) Instruments : Analytical Balance.

Reference:

1) Practical pharmaceutical chemistry, Fourth edition-part one, edited by A H BECKETT, J B STENLAKE, CBS Publishers and Distributors, 2005, P-140.

2) Indian Pharmacopoeia by ministry of health and family welfare, Govt. of India, Volume 1, 2007, P-313,316

Principle:

This is direct acid base titration in which sulphuric acid reacts with sodium carbonate to form CO2 and water. As CO2 formed in reaction can change color of the indicator before equivalence point it is advisable to boil solution to perform the errorless titration.

$Na_2CO_3 + H_2SO_4 \text{----------} Na_2SO_4 + CO_2 + H_2O$

Procedure:

PREPARATION OF 0.5 M SULPHURIC ACID:

Add slowly, with stirring , 30ml of sulphuric acid to about 100ml of distilled water allow to cool 25 degree and standardize.

SAFETY TIP

1. Sulphuric acid in higher concentration forms acidic mist. Both the mist and solution have a corrosive effect on tissues with the potential to damage respiratory organs, eyes, skin and intestine.
2. While preparing Sulphuric acid solution, take water in a beaker and add the acid drop by drop with mixing. If you do the reverse it will burst.

STANDARDISATION PROCEDURE:

Weigh accurately about 1.5g of anhydrous sodium carbonate previously heated at about 270degree for one hour. Dissolve it in 100ml of water and add 0.1ml of methyl red solution. Add the acid slowly from a burette, with constant stirring until the solution become faintly pink. Heat the solution to boiling , cool and continue the titration . Heat again to boiling and titrate further as necessary until the faint pink color is no longer affected by continued boiling.

Report:

Molarity of Sulphuric acid was found to be _____

Experiment No. 7

PREPARATION AND STANDARDIZATION OF 0.1M SODIUM THIOSULPHATE

Aim:

To prepare and standardize 0.1M Sodium thiosulphate.

Chemicals required:

- Sodium thiosulphate
- Sodium carbonate
- Potassium dichromate
- Potassium iodide
- Hydrochloric acid
- Starch
- Distilled water

Principle:

Sodium thiosulphate is a secondary stanadard which is standardized against the primary standard potassium dichromate. Potassium dichromate in acid solution is reduced by potassium iodide to liberate an equivalent amount of iodine which in turn reacts with sodium thiosulphate. Free iodine forms blue colour with starch. At the end point all the iodine reacts with thiosulphate which causes disappearance of blue colour. In this titration the dichromate ion gets converted to green chromic salt which gives green colour at the end point.

Reaction:

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

$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_2O_6 + NaI$$

Procedure:

Preparation of 0.1M sodium thiosulphate

Dissolve 2.5 g of sodium thiosulphate and 0.02 g of sodium carbonate in carbon dioxide-free water and dilute to 100 ml with the same solvent.

NOTE:

- ★ Sodium thiosulphate is a reducing agent and efflorescent in nature. If the solution of sodium thiosulphate is prepared by using distilled water, the excess carbon dioxide present in the distilled water may decompose the thiosulphate with the formation of sulphur. This decomposition also caused by bacterial action.
- ★ Use freshly boiled and cooled water to expel carbon dioxide
- ★ Add a small amount of sodium carbonate to maintain pH between 9 and 10 at which bacterial activity is least.

Standardization of 0.1M sodium thiosulphate

Weigh accurately 1.2 g of potassium dichromate and dissolve in 250 ml of distilled water. From the solution, pipette out 25 ml and transfer it to an iodine flask. Dilute the solution with 5o ml of water and add 2 g of KI, 5 ml of concentrated hydrochloric acid. Stopper the flask and allow to stand for 5 minutes. Dilute it with 50 ml of water and titrate with sodium thiosulphate solution until the color becomes light yellow. Further add 1 ml of starch solution. The solution turns to blue colour. Continue the titration until the blue colour disappears green colour appears.

Reference:

1. Practical Pharmaceutical Analytical chemistry by M.M.Alam.
2. Indian pharmacopoeia 1996-Volume-II

Report:

Molarity of Sodium thio Sulphate was found to be

Experiment No. 8

PREPARATION AND STANDARDIZATION OF 0.02M POTASSIUM PERMANGANATE

Aim: To prepare and standardize 0.02M Potassium permanganate Solution.

Requirements:

- Apparatus: Iodine flask, Conical flask ,Burette ,Pipette ,Water bath, Burner ,Measuring Cylinder ,Beaker ,Dropper , Glass rod ,Butter paper ,.Etc.,
- Chemicals: Sodium thiosulfate (0.1M), potassium Permanganate, Potassium iodide, Sulphuric acid (1M) and Starch solution.
- Instruments: Analytical Balance, Sonicator.

Reference:

1) Practical pharmaceutical chemistry, Fourth edition-part one, edited by A H BECKETT, J B STENLAKE, CBS Publishers and Distributors, 2005, P-177,178

2) Indian Pharmacopoeia by ministry of health and family welfare, Govt. of India, Volume 1, 2007, P-315.

Principle:

Potassium permanganate often contains a small proportion of manganese dioxide; volumetric solutions must be made up approximately and then standardized. The intense color of the solution makes difficult the detection of undissolved solid. The use of heat in the preparation of potassium permanganate solutions is also undesirable, since traces of other contaminants on the glass vessels used can catalyze its decomposition.

Potassium permanganate may be standardized with either sodium oxalate or oxalic acid. The former is preferred because it is readily available to higher standard of purity (99.95%) and unlike oxalic acid, it is available in the anhydrous state. The standardization depends upon the reactions expressed as follows:

$$8\ H_2SO_4 + 2\ KMnO_4 + 5\ Na_2C_2O_4 \rightarrow 2\ MnSO_4 + 10\ CO_2 + K_2SO_4 + 5\ Na_2SO_4 + 8\ H_2O$$

Procedure:

Preparation of 0.02 M potassium permanganate solutions:

Dissolve 3.2g of potassium permanganate in 1000ml of water, heat on a water bath for 1 hour; allow standing for 2 days and filtering through glass wool. Store protected from light.

Standardization procedure:

Method A: With Sodium Oxalate:

Weigh out sodium oxalate (6.7g) accurately into a liter graduated flask, dissolve in water and make up to the volume. Pipette out 20ml of this solution add concentrated sulphuric acid about (5ml) and warm to about 70 degree. Add the potassium permanganate solution from the burette. The first few drop results in a pink color persisting for about 20 seconds. Wait until the color disappears and then continue the titration. Formation of a brown color during the titration is caused by insufficient acid, by using too high a temperature or by the use of dirty flask. The end point is reached when a faint pink color persists for about 30 seconds upon the shaking the flask.

1ml of 0.02M potassium permanganate is equivalent to 0.0067g of sodium oxalate.

Method B: With Sodium Thio Sulphate:

Preparation of 0.1 M sodium thiosulphate:

Dissolve 25g of sodium thiosulfate and 2.0g of sodium carbonate in CO2 free water and dilute to 1000ml with the same solvent. Standardize the solution in the following manner:

Standardization procedure:

Dissolve 0.200g of potassium bromated, weighed accurately, in sufficient water to produce 250.0ml. To 50.0ml of this solution add 2gm of potassium iodide and 3ml of 2M HCL and titrate with the sodium thiosulphate solution using starch solution added towards the end of the titration as the indicator until the blue color is discharged.

1ml of 0.1M sodium thiosulphate is equivalent to 0.002784g of KBrO3.

Standardization Of KMnO4 Procedure:

To 25.0ml of the solution in a glass-stoppered flask add 2g of potassium iodide, followed by 10ml of 1M sulphuric acid. Titrate the liberated iodine with 0.1M sodium thiosulphate, using 3 ml of starch solution, added towards the end of the titration, as indicator. Perform a blank determination and make necessary correction.

1ml of 0.1M sodium thiosulphate is equivalent to 0.003161 g of KMnO4.

Report:

The molarity of Sodium ThioSulphate was found to be _____

Experiment No. 9

PREPARATION AND STANDARDIZATION OF 0.1M CERRIC AMMONIUM SULPHATE

Aim: To prepare and standardize 0.1M cerric ammonium sulphate.

Requirements:

- Apparatus: Iodine flask, Conical flask ,Burette ,Pipette ,Water bath, Burner
- Measuring Cylinder ,Beaker ,Dropper , Glass rod ,Butter paper ,.Etc.,
- Chemicals: cerric ammonium sulphate, sulphuric acid, arsenic trioxide, sodium hydroxide(8%w/w), osmic acid solution, ferroin sulphate solution and distilled water.
- Instruments: Analytical Balance, Sonicator.

Reference:

1) Practical pharmaceutical chemistry, Fourth edition-part one, edited by A H BECKETT, J B STENLAKE, CBS Publishers and Distributors, 2005, P-194,195

2) Indian Pharmacopoeia by ministry of health and family welfare, Govt. of India, Volume 1, 2007, P-313.

Principle:

Cerium sulphate is a power full oxidizing agent in acid solution (1-8N). It is bright yellow color and corresponding cerium salt form by reduction is colorless strong solutions are self indicating. However since dilute solutions are used hence indicators are used for observation of end point. Arsenic trioxides used as primary standard in presence of sulphuric acid and osmic acid using ferroin sulphate as an indicator for standardization of the solution. The standardization depends on the reactions expressed by the following equations:

Procedure:

Preparationof 0.1M cerric ammonium sulphate

Dissolve 65g of cerric ammonium sulphate, with the aid of gentle heat, in a mixture of 30ml of sulphuric acid and 500ml of water. Cool, filter the solution, if turbid, and dilute to 1000ml with water .Standardize the solution in the following manner.

Standardization of 0.1M cerric ammonium sulphate

Weigh accurately about 0.5 gm of **Ferrous ammonium Sulphate** previously dried at 105ºCfor 1 hour and transfer to a 500 ml conical flask. Add 20 ml of dilute sulphuric acid and 30 ml of water, 0.1 ml of ferroin sulphate solution and slowly titrate with the cerric ammonium sulphate solution until the pink colour is changed to a green colour,

Each ml of 0.1M Ceric Ammonium sulphate is equivalent to .0392 gm of FAS 1 ml of 0.1M cerric ammonium sulphate is equivalent to 0.004946g of As2O3.

Report:

Molarity of Cerric Ammonium Sulphate was found to ~~be~~ _____

Experiment No. 10

PREPARATION AND STANDARDIZATION OF 0.1M PERCHLORIC ACID

Aim: To prepare and standardize 0.1M Perchloric acid.

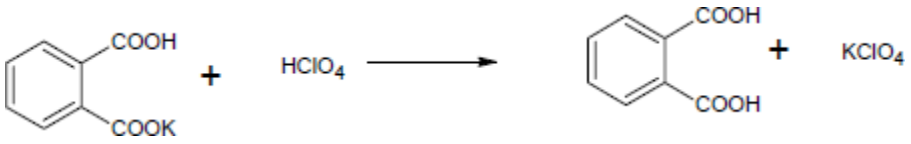
Chemicals required:

- Perchloric acid
- Glacial acetic acid
- Crystal violet
- Potassium hydrogen phthalate
- Acetic anhydride

Principle:

0.1M perchloric acid is a secondary standard which is standardized against potassium hydrogen phthalate. Potassium hydrogen phthalate behaves as a base in acetic acid, which is titrated against perchloric acid.

Reaction:



Procedure:

Preparation of 0.1M perchloric acid

Mix 0.85 ml of perchloric acid with 50 ml of anhydrous glacial acetic acid and 2.5 ml of acetic anhydride, cool and add anhydrous glacial acetic acid to produce 100 ml. Allow the prepared solution to stand for 1 day.

Precautions:

1. The perchloric acid must be well diluted before adding the acetic anhydride.
2. Failure to observe this precaution leads to the formation of explosive acetyl perchlorate.

Standardization of 0.1M perchloric acid

Weigh accurately about 0.35 g of potassium hydrogen phthalate, previously powdered lightly and dried at 120^{o} for 2 hours and dissolve it in 50 ml of anhydrous glacial acetic acid. Add 0.1 ml of crystal violet solution and titrate with the perchloric acid solution until the violet colour changes to emerald-green. Perform a blank determination and make any necessary correction. Each ml of 0.1 M perchloric acid is equivalent to 0.02042 g of $C_8H_5KO_4$.

Reference:

1. Practical pharmaceutical Analysis by Dr.G.Devala Rao
2. Indian Pharmacopoeia 1996-Volume I & II

Report:

Molarity of Perchloric acid was found to be _____

Experiment No. 11

ASSAY OF AMMONIUM CHLORIDE

Aim: Determine the percentage purity of the given sample of ammonium chloride and report it.

Chemicals required:

- Ammonium chloride
- Sodium hydroxide
- Formaldehyde
- Phenolphthalein

Reference:

- Pharmaceutical titrimetric analysis by A. Napoleon Page No: 11.24
- IP 1996. Page No: 46-47
- Practical pharmaceutical analysis by G.Devalarao. Page No: 33

STANDARD

It contains not less than 99% and not more than 100.5% w/w of ammonium chloride calculated with reference to the dried substance.

Principle:

Ammonium chloride is estimated by Alkalimetry method. When ammonium chloride is treated with formaldehyde, ammonium chloride decomposed to hexamine with liberation of an equivalent amount of hydrochloric acid. The liberated hydrochloric acid is titrated with sodium hydroxide using phenolphthalein indicator.

$$NH_4Cl + H_2O \longrightarrow NH_4OH + HCl$$

Ammonium chloride — Ammonium hydroxide

$$4NH_4OH + 6HCHO \longrightarrow (CH_2)_6N_4 + 10H_2O$$

Formaldehyde — Hexamine

$$\text{Liberated. } HCl + NaOH \longrightarrow NaCl + H_2O$$

Procedure:

Standardization of 0.1M sodium hydroxide solution

Weigh accurately about 0.5g of PHP previously powdered and dried at 120°C for 2hrs and dissolve in 75ml of water in a conical flask. Add 1-2 drops of phenolphthalein solution and titrate with sodium hydroxide solution until a permanent pale pink colour is produced. Each ml of 0.1M sodium hydroxide is equivalent to 0.02042g of $C_8H_5KO_4$ (PHP)

Assay:

Weigh accurately about 0.1g of ammonium chloride in 20ml of water and add 5ml of previously neutralized formaldehyde solution. Allow to stand for 2mins. Then titrate slowly with 0.1M sodium hydroxide using a phenolphthalein solution as indicator. The endpoint is the appearance of pale pink colour. Each ml of 0.1M sodium hydroxide is equivalent to 0.005349g of ammonium chloride.

Report:

1. The strength of 0.1M Sodium hydroxide was found to be----------.
2. The % purity of given sample of ammonium chloride was found to be---------- --.

Experiment No. 12

ASSAY OF BORIC ACID

Aim: Determine the percentage purity of the given sample of boric acid and report it.

Chemicals required:

- Sodium hydroxide
- Potassium hydrogen phthalate
- Glycerol
- Phenolphthalein

References:

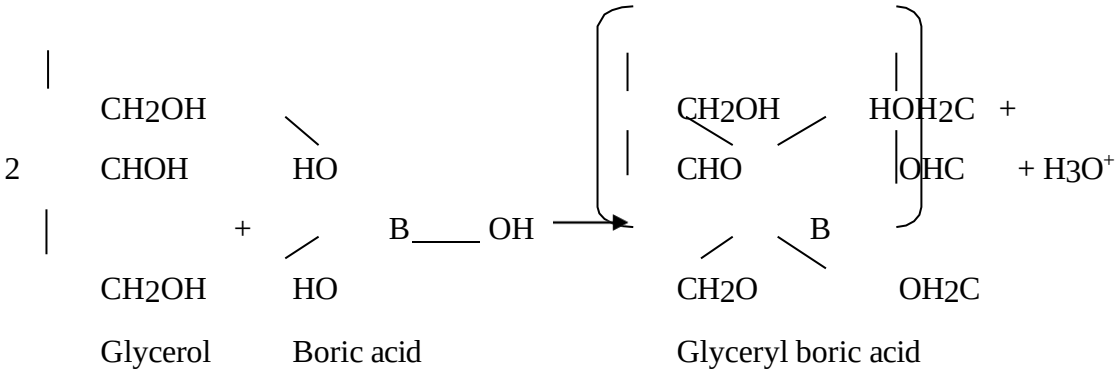
- Indian Pharmacopoeia 1996. Vol-1, Page No: 111
- Pharmaceutical titrimetric analysis A.Napoleon. Page No: 11.21.

STANDARD

It contains not less than 99.5% and not more than 100.5% w/w of H_3BO_3 calculated with reference to the dried substance.

Principle:

Boric acid is a weak tribasic acid, hence it cannot be titrated with standard alkali to get accurate end point. So, boric acid is made strong by dissolving in solution of poly hydroxy compounds such as glycerol, mannitol or sorbitol in water. Boric acid reacts with glycerin and converted in to glyceryl boric acid which is a strong monobasic acid and this is titrated with sodium hydroxide using phenolphthalein as indicator.



Procedure:

Standardization of 0.1M sodium hydroxide solution

Weigh accurately about 0.5g of PHP previously powdered and dried at 120°C for 2hrs and dissolve in 75ml of water in a conical flask. Add 1-2 drops of phenolphthalein solution and titrate with sodium hydroxide solution until a permanent pale pink colour is produced. Each ml of 0.1M sodium hydroxide is equivalent to 0.02042g of $C_8H_5KO_4$ (PHP)

Assay:

Weigh accurately about 0.2g of given sample of boric acid, dissolve in a mixture of 5ml water and 10ml glycerin previously neutralized to phenolphthalein solution. Titrate the solution against 1M sodium hydroxide using phenolphthalein as indicator. Each ml of 1M sodium hydroxide is equivalent to 0.06183g of H_3BO_3.

Report:

The % purity of given sample of Boric acid was found to be---------- --.

Experiment no. 13

ASSAY OF CALCIUM GLUCONATE

Aim: To determine the percentage purity of given sample of Sodium sulphate.

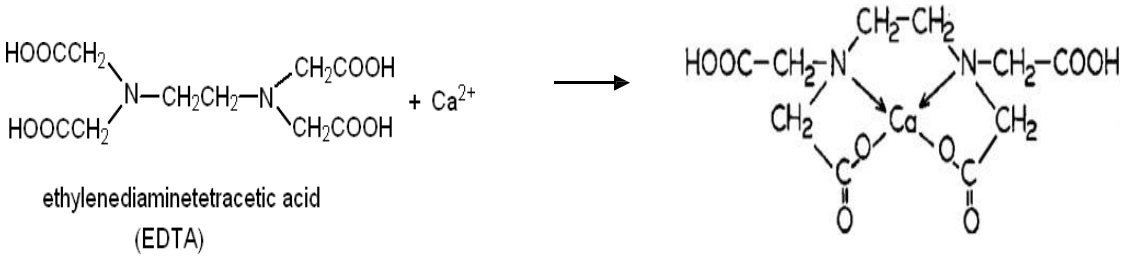
Chemicals required:

- Calcium carbonate
- Dilute hydrochloric acid
- Sodium hydroxide solution
- Disodium EDTA
- Magnesium sulphate
- Ammonia-ammonium chloride buffer
- Calcon mixture
- Mordent black-II
- Calcium gluconate
- Distilled water

Principle:

Calcium gluconate is assayed by complexometric titration using disodium EDTA a titrant. This is a type of replacement titration. In this assay magnesium sulphate is added to increase the sensitivity and to give sharp end point. In the beginning magnesium forms complex with indicator and produces pink colour. The magnesium indicator complex is more stable than that of calcium indicator complex. When EDTA solution is added, it forms complex with EDTA. When all the calcium has been consumed the nest drop of EDTA will break the magnesium indicator complex. Thus the free indicator will show blue colour at the end point. During the titration the pH is maintained at 10 by the addition of ammonia-ammonium chloride buffer and mordent black-II is used as indicator.

REACTION:



Procedure:

Standardization of 0.05M Disodium EDTA

Weigh accurately about 0.3 gm of anhydrous magnesium sulphate, dissolve in 50 ml of water. Add 10 ml of (pH 10) ammonia buffer and 50 mg mixture of Mordant black II and sodium chloride as indicator and titrate with 0.05M disodium edetate until the solution becomes blue.

Each ml of 0.05M disodium edetate is equivalent to 0.012325 gm of magnesium sulphate.

Assay of calcium gluconate

Weigh accurately about 0.5 g and dissolve in 50 ml of warm *water*; cool, add 5.0 ml of 0.05M magnesium sulphate and 10 ml of strong ammonia solution and titrate with 0.05M disodium edetate using mordant black II mixture as indicator. From the volume of 0.05M disodium edetate required subtract the volume of the magnesium sulphate solution added. Each ml of the remainder of 0.05M disodium edetate is equivalent to 0.02242 g of $C_{12}H_{22}CaO_{14},H_2O$.

STANDARD

Calcium gluconate contains not less than 98.5% and not more than 102% of $C_{12}H_{22}CaO_{14}, H_2O$.

Reference:

1. Practical Pharmaceutical Analytical chemistry by M.M.Alam.
2. Practical pharmaceutical Analysis by Dr.G.Devala Rao
3. Indian Pharmacopoeia 1996-Volume-I

Report:

The % purity of given sample of Calcium Gluconatewas found to be --.

Experiment No. 14

ASSAY OF COPPER SULPHATE

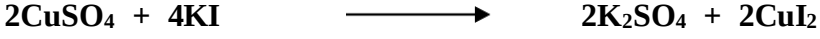
AIM: To determine the percentage purity of given sample of Copper sulphate.

CHEMICALS REQUIRED

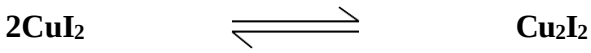
- Sodium thiosulphate
- Copper sulphate
- Sodium carbonate
- Acetic acid
- Potassium dichromate
- Potassium thiocyanate
- Potassium iodide
- Starch
- Hydrochloric acid
- Distilled water

PRINCIPLE

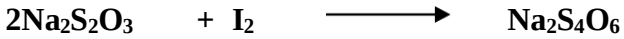
Assay of copper sulphate is based on iodometry. Copper sulphate in the presence of acetic acid reacts with potassium iodide to give cupric iodide.



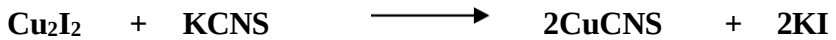
Cupric iodide formed is unstable and it decomposes into cuprous iodide and iodine..



Formed iodine is titrated with standard sodium thiosulphate solution using starch as an indicator. Starch is added towards the end point of titration.



The decomposition of cupric iodide to cuprous iodide is reversible. In the presence of potassium thiocyanate, cuprous iodide gets converted into cuprous thiocyanate which is more sparingly soluble than cuprous iodide. Potassium thiocyanate is added towards the end of reaction to prevent adsorption of iodine by the cuprous thiocyanate.



PROCEDURE

Standardization of 0.1M sodium thiosulphate

Weigh accurately 1.2 g of potassium dichromate and dissolve in 250 ml of distilled

water. From the solution, pipette out 25 ml and transfer it to an iodine flask. Dilute the solution with 5o ml of water and add 2 g of KI, 5 ml of concentrated hydrochloric acid. Stopper the flask and allow to stand for 5 minutes. Dilute it with 50 ml of water and titrate with sodium thiosulphate solution until the color becomes light yellow. Further add 1 ml of starch solution. The solution turns to blue colour. Continue the titration until the blue colour disappears green colour appears.

Assay of copper sulphate

Dissolve I g of copper sulphate in 50 ml of water followed by the addition of 3 g of potassium iodide, 5 ml acetic acid and titrate the liberated iodine with 0.1M sodium thiosulphate using starch solution as indicator. Continue the titration until a faint blue colour remains, add 2 g of potassium thiocyanate stir well and continue the titration till a milky white colour appears. 1 ml of 1M sodium thiosulphate is equivalent to 0.249 g of $CuSO_4.5H_2O$.

Reference:

1. Practical Pharmaceutical Analytical chemistry by M.M.Alam.

Report:

The % purity of given sample of Copper sulphate was found to be _____

Experiment No. 15

ASSAY OF HYDROGEN PEROXIDE

Aim: To carry out the assay of hydrogen peroxide by permanganometry.

Requirements:

- Apparatus: Conical flask ,Burette ,Pipette ,Water bath, Burner ,Measuring Cylinder
- ,Beaker ,Dropper , Glass rod ,Butter paper ,.Etc.,
- Chemicals: Potassium permanganate, Potassium iodide, Sulphuric acid (1M), and Sodium Thiosulphate, Starch solution.
- Instruments: Analytical Balance, Sonicator.

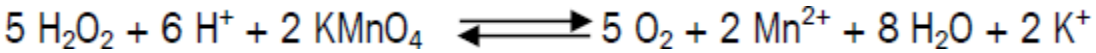
Reference:

1) Indian Pharmacopoeia by ministry of health and family welfare, Govt. of India, Volume 1, 2007, P-313.

2) Practical pharmaceutical chemistry, Fourth edition-part one, edited by A H BECKETT, J B STENLAKE, CBS Publishers and Distributors, 2005, P-178.

Principle:

This determination depends upon mutual oxidation reduction with use of potassium permanganate as oxidising agent. The assay is as expressed as following equation.



$$5\ H_2O_2 + 6\ H^+ + 2\ KMnO_4 \rightleftharpoons 5\ O_2 + 2\ Mn^{2+} + 8\ H_2O + 2\ K^+$$

Procedure:

STANDARDISATION PROCEDURE:

METHOD A: With SODIUM OXALATE:

Weigh out sodium oxalate (6.7g) accurately into a liter graduated flask, dissolve in water and make up to the volume. Pipette out 20ml of this solution add concentrated sulphuric acid about (5ml) and warm to about 70 degree. Add the potassium permanganate solution

from the burette. The first few drop results in a pink color persisting for about 20 seconds. Wait until the color disappears and then continue the titration. Formation of a brown color during the titration is caused by insufficient acid, by using too high a temperature or by the use of dirty flask. The end point is reached when a faint pink color persists for about 30 seconds upon the shaking the flask.

1ml of 0.02M potassium permanganate is equivalent to 0.0067g of sodium oxalate.

METHOD B: with SODIUM THIO SULPHATE:

PREPARATION OF 0.1M SODIUM THIOSULPHATE:

Dissolve 25g of sodium thiosulfate and 2.0g of sodium carbonate in CO2 free water and dilute to 1000ml with the same solvent. Standardize the solution in the following manner:

STANDARDISATION PROCEDURE:

Dissolve 0.200g of potassium bromated, weighed accurately, in sufficient water to produce 250.0ml. To 50.0ml of this solution add 2gm of potassium iodide and 3ml of 2M HCL and titrate with the sodium thiosulphate solution using starch solution added towards the end of the titration as the indicator until the blue color is discharged.

1ml of 0.1M sodium thiosulphate is equivalent to 0.002784g of KBrO3.

STANDARDISATION OF KMnO4 Procedure:

To 25.0ml of the solution in a glass-stoppered flask add 2g of potassium iodide, followed by 10ml of 1M sulphuric acid. Titrate the liberated iodine with 0.1M sodium thio sulphate, using 3 ml of starch solution, added towards the end of the titration, as indicator. Perform a blank determination and make necessary correction.

1ml of 0.1M sodium thiosulphate is equivalent to 0.003161 g of KMnO4.

Assay procedure (hydrogen peroxide):

Dilute about 1.0 g to 100ml with water. To 10 ml of the resulting solution add 20 ml of 1M sulphuric acid and titrate with 0.02M potassium permanganate.

1ml of 0.02M potassium permanganate is equivalent to 0.001701 g of H_2O_2 or 0.56ml of oxygen.

Report:

The % purity of given sample of Hydrogen peroxide was found to be _____

Experiment No. 16

ASSAY OF SODIUM BENZOATE

Aim: To determine the percentage purity of given sample of sodium benzoate.

Chemicals required:

- Perchloric acid
- Glacial acetic acid
- Crystal violet
- Potassium hydrogen phthalate
- Acetic anhydride
- 1-naphtholbenzein

Procedure:

Standardization of 0.1M Perchloric acid

Weigh accurately about 0.35 g of potassium hydrogen phthalate, previously powdered lightly and dried at 120^{o} for 2 hours and dissolve it in 50 ml of anhydrous glacial acetic acid. Add 0.1 ml of crystal violet solution and titrate with the perchloric acid solution until the violet colour changes to emerald-green. Perform a blank determination and make any necessary correction. Each ml of 0.1 M perchloric acid is equivalent to 0.02042 g of $C_8H_5KO_4$.

Assay of sodium benzoate

Weigh accurately about 0.25 g, dissolve in 20 ml of anhydrous glacial acetic acid, warming to 50^{o} if necessary, cool and titrate against 0.1M perchloric acid using 0.05 ml of 1-naphtholbenzein solution as indicator. Perform a blank determination and make any necessary correction. Each ml of 0.1M perchloric acid is equivalent to 0.01441 g of $C_7H_5NaO_2$.

STANDARD:

Reference:

1. Practical pharmaceutical Analysis by Dr.G.Devala Rao
2. Indian Pharmacopoeia 1996-Volume-II

Report:

The % purity of given sample of Sodium Benzoate was found to be _____

Experiment No. 17

ASSAY OF FERROUS SULPHATE

Aim:

Determine the percentage purity of the given sample of ferrous sulphate and report

Chemicals required:

- Ferrous ammonium sulphate
- Ceric ammonium sulphate
- Ferrous sulphate
- Dil. Sulphuric acid
- Ferroin solution

References:

- Pharmaceutical titrimetric analysis A.Napoleon, Page No: 11.48-11.50
- Indian Pharmacopoeia 2007. Vol- II, Page No: 1125
- British Pharmacopoeia 2003. Vol- I, Page No: 790-791.

Standard:

It contains not less than 86.0% and not more than 90.0% w/w of ferrous sulphate.

Principle:

Ferrous sulphate is a reducing agent and it can be assayed by titrating with powerful oxidizing agent ceric ammonium sulphate in presence of dil. sulphuric acid solution using ferroin solution as indicator. The basic principle involved in this titration is oxidation and reduction. Ceric ammonium sulphate oxidizes ferrous sulphate to ferric sulphate and ceric ion itself is converted to cerous ion. It is represented as ionic equation

$$Fe^{2+} \longrightarrow Fe^{3+} + e^{-}$$

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

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

$$2FeSO4 + 2(NH4)2Ce(SO4) \longrightarrow Fe3(SO4)3 + 2(NH4)2SO4 + Ce2(SO4)3$$

Ferrous sulphate	CAS	Ferric sulphate	Ammonium sulphate	Cerous sulphate

Procedure:

Standardization of 0.1M ceric ammonium sulphate

Weigh accurately about 0.5g of ferrous ammonium sulphate; dissolve in 20ml dilute sulphuric acid in a conical flask. Shake the solution and titrate against approximately prepared 0.1M ceric ammonium sulphate using ferroin solution as indicator. The end point is the appearance of green colour. Each ml of 0.1M ceric ammonium sulphate is equivalent to 0.03291g of ferrous ammonium sulphate.

Assay:

Weigh accurately about 0.5g of sample of dried ferrous sulphate and dissolve in mixture of 20ml of 1M Sulphuric acid and 30ml of water in a clean conical flask. Titrate with 0.1M ceric ammonium sulphate using ferroin solution as indicator. The end point is the appearance of green colour. Each ml of 0.1M ceric ammonium sulphate is equivalent to 0.03291g of ferrous sulphate.

Report:

The % purity of given sample of Ferrous sulphate was found to be _____

Experiment No. 18

ASSAY OF SODIUM CHLORIDE

Aim: To carry out the assay of Sodium chloride by Precipitation titration.

Requirements:

- Apparatus: Conical flask, Burette ,Pipette ,Water bath, Burner ,Measuring Cylinder
- ,Beaker ,Dropper , Glass rod ,Butter paper ,.Etc.,
- Chemicals: Silver nitrate, Sodium Chloride, Acetic acid, Methanol, Eosin Solution, Ammonium Thiocyanate, Nitric Acid, Ferric Ammonium sulphate , Dibutyl phthalate.
- Instruments: Analytical Balance, Sonicator.

Reference:

1) Indian Pharmacopoeia by ministry of health and family welfare, Govt. of India, Volume 1, 2007, P-313,V-II page;585,86.

2) Practical pharmaceutical chemistry, Fourth edition-part one, edited by A H BECKETT, J B STENLAKE, CBS Publishers and Distributors, 2005, P-197.

Principle:

It is a precipitation type of titration. Titration is carried out by Volhard's method of chloride estimation. Sodium chloride is first reacted with excess of acidified silver nitrate which results in formation of a white precipitate of silver chloride.

$$AgNO^3 + NaCl \longrightarrow AgCl\downarrow + NaNO^3$$

$$Ag^+ + Cl^- \longrightarrow AgCl\downarrow$$

Then the excess silver nitrate is back titrated with ammonium thiocyanate giving white precipitate of silver thiocyanate.

$$SCN^- + \text{excess } Ag+ \rightarrow AgSCN(s)$$

Chloride ppt is coagulated by means of dibutyl phthalate, because silver chloride reacts slowly with ammonium thiocyanate and makes the end point flat since the end point involves the production of red ferric thiocyanate complex with the thiocyanate ions.

$$SCN^- + Fe^{3+} \rightarrow Fe(SCN)^{2+}$$

Procedure:

PREPARATION OF 0.1M SILVER NITRATE (METHOD A)

Dissolve 17.0g in sufficient water to produce 1000ml. Standardize the solution in the following manner;

STANDARDISATION OF 0.1M SILVER NITRATE:

Weigh accurately about0.1g of sodium chloride, previously dried at 110degree for two hours, and dissolve in 5ml of water. Add 5ml of acetic acid, 50 ml of methanol and 0.15ml of EOSIN solution. Stir preferably with magnetic stirrer, and titrate with the silver nitrate solution.1ml of 0.1M silver nitrate is equivalent to 0.005844g of NaCl.

STANDARDIZATION OF 0.1M SILVER NITRATE (METHOD B)

Dry some analar sodium chloride at about 300 degree for two hours, cool in desiccators and use to prepare an accurate 0.1M solution of sodium chloride. Pipette 25ml of this solution into conical flask, add potassium chromate solution (5% w/v) and titrate with silver nitrate solution.

1ml of 0.1M silver nitrate is equivalent to 0.005844 g of NaCl.

PREPARATION OF 0.1M AMMONIUM THIOCYANATE:

Dissolve 7.612g of ammonium thiocyanate in sufficient water to produce 1000ml. Standardize the solution in the following manner;

STANDARDIZATION OF 0.1M AMMONIUM THIOCYANATE:

Pipette 30.0ml of 0.1M silver nitrate into a glass stoppered flask, dilute with 50ml of water, add 2ml of nitric acid and 2ml of ferric ammonium sulphate solution and titrate with ammonium thiocyanate solution to the first appearance of a red brown color.1ml of 0.1M silver nitrate is equivalent to 0.007612g of NH4SCN.

Assay procedure:

Weigh accurately about 0.1g and dissolve in 50 ml of water in a glass stoppered flask. Add 50.0ml of 0.1M silver nitrate, 5ml of 2M nitric acid and 2ml of dibutyl phthalate, shake well and titrate with 0.1Mammonium thio cyanate using 2ml of ferric ammonium sulphate solution as

indicator, until the color becomes reddish yellow.

1ml of 0.1M silver nitrate is equivalent to 0.005844g of NaCl.

Report: The % purity of given sample of Sodium chloride was found to be

Experiment No. 19

ASSAY OF STRONG ACID VS STRONG BASE BY CONDUCTOMETRIC TITRATION

Aim: To carry out conductometric titration of strong acid against strong base.

Requirements:

- Apparatus: Conical flask, Burette ,Pipette ,Water bath, Burner ,Measuring Cylinder, Beaker ,Dropper , Glass rod ,Butter paper ,.Etc.,
- Chemicals: Hydrochloric acid, Sodium hydroxide, Potassium hydrogen phthalate, distilled water.
- Instruments: Analytical Balance, Sonicator.

Reference:

1) Indian Pharmacopoeia by ministry of health and family welfare, Govt. of India, Volume 1, 2007, P-115.

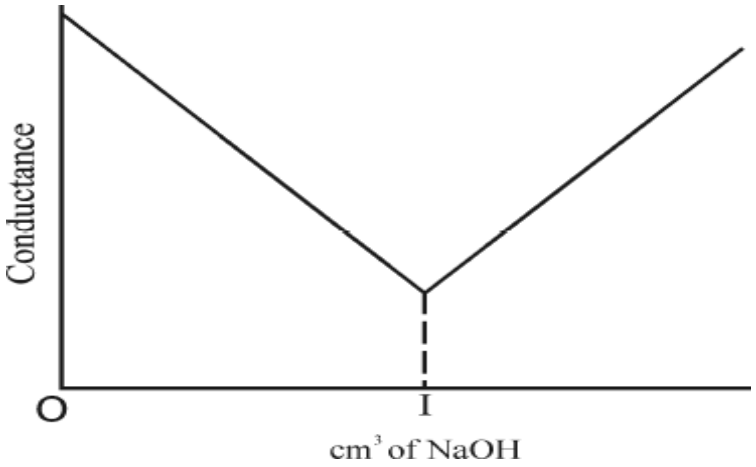
2) Vogel's textbook of quantitative chemical analysis, fifth edition, revised by G H JEFFERY, J BASSETT, J MENDHAM and R C DENNEY, LONGMAN scientific and technical, New York, 1989. P-525, 26.

Principle:

Hydrochloric acid is strong acid and sodium hydroxide is a strong base. The strength of HCL can be determined by titrating it directly with sodium hydroxide. The conductance first falls due to the replacement of the hydrogen ion by the added cation and then after the equivalence point has been reached, rapidly rises with further addition of strong alkali due to the large values of the hydroxyl ions (198). The two branches of the curve are straight lines provided the volume of the reagent added is negligible and their intersection gives the end point curves 1 and 2, figure below.

$HCl + NaOH \rightarrow NaCl + H_2O$

$H^+ + Cl^- + Na^+ + OH^- \rightarrow Na^+ + Cl^- + H_2O$



PROCEDURE:

PREPARATION OF 1M SODIUM HYDROXIDE:

Dissolve 42 g of sodium hydroxide in sufficient CO2 free water to produce 1000ml. Store in bottles with well fitted suitable stoppers which prevent access to atmospheric carbon dioxide.

Solutions of lower concentrations are prepared by quantitatively diluting accurately measured volumes of 0.1 M sodium hydroxide with sufficient CO2 free water to give the desired concentration.

STANDARDISATION PROCEDURE:

Weigh accurately about 5g of potassium hydrogen phthalate, previously powdered and dried at 120 degrees for 2 hours, and dissolve in 75 ml of CO2 free water. Add 0.1 ml of phenolphthalein solution and titrate with the sodium hydroxide solution until a permanent pink color is produced. ml of 1M sodium hydroxide is equivalent to 0.2042g of C8H5KO4.

FOR STRONG ACID CONTENT DETERMINATION:

Wash and dry all glassware as per standard laboratory procedure. Rinse all glassware's with distilled water. Rinse the burette with small portion of NaOH solution and fill the burette with

standard NaOH solution. Add 50ml of hydrochloric acid or unknown sample of given strong acid in a beaker. Immerse the platinum electrodes in this solution (add some distilled water if sample amount is small). Note the initial conductance of sample before starting titration. Add 1ml of NaOH at a time from burette and note down the change in conductance after each ml. Continue until the conductance value after falling once starts increasing (end point). Plot the graph between conductance vs. volume of NaOH added. The point of intersection between two curves indicates the end point of titration.

Report:

Percentage purity of strong acid was found to be

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Vision

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Mission

M1 Create pharmacy graduates through **quality education**

M2 Promote innovation, **creativity**, and excellence **in teaching**, learning, and **research**

M3 **Inspire** integrity, teamwork, critical thinking**, personal** development, and ethics in **students** and lay **the** foundation for lifelong learning

M4 **Serve** the **healthcare, technological, scientific**, and **economic** needs of then **society**.