AS PER PCI REGULATIONS

FIRST YEAR B. PHARM | SEMESTER-I

A PRACTICAL BOOK OF PHARMACEUTICAL ANALYSIS

Mrs. PRITI NISHAD PATIL

NIRALI PRAKASHAN
ADVANCEMENT OF KNOWLEDGE

A Practical Book of

PHARMACEUTICAL ANALYSIS

As Per PCI Regulations

FIRST YEAR B. PHARM.

Semester - I

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Last but not the least: I beg forgiveness of all those who have been with me over the course of the years and whose names I have failed to mention."

Preface

My goal in writing this book is to provide guidelines to the students related to basic concept of Pharmaceutical Analysis (Practical). I will present for you is not just the overview of the syllabus but the detail technique and logic behind them.

I will look at the design of syllabus given by PCI and with the help of that assemble all the matter.

It gives good support to the syllabus of Pharmaceutical Analysis (Practical) as well as through light on some related theoretical parts.

Basic concepts of Pharmaceutical Analysis involve the molarity and normality concept. On base of that some calculations are performed related to titration which is elaborated in detail.

The titration will give you the concentration of analyte and amount of purity of that substance. It also involves the standardisation of secondary standard analyte by using primary standard.

This book also involves the method related to electroanalytical techniques, the use of different electrodes, detection of end point without indicator and reading of graph is also involved.

I am thankful to the publisher Shri. Dineshbhai Furia, Shri. Jignesh Furia, Mr. Malik Shaikh, Mr. Kiran Velankar, Mrs. Anjali Muley, Mrs. Roshan Khan and all staff of Nirali Prakasha for their efforts and keen interest in publishing this book in a very limited span of time.

The preparation of solution is done by using Indian Pharmacopoeia.

Syllabus

I. Limit Test of the following:

1. Chloride
2. Sulphate
3. Iron
4. Arsenic

II. Preparation and Standardization of:

1. Sodium hydroxide
2. Sulphuric acid
3. Sodium thiosulfate
4. Potassium permanganate
5. Ceric ammonium sulphate

III. Assay of the following compounds along with standardization of titrant:

1. Ammonium chloride by acid-base titration
2. Ferrous sulphate by cerimetry
3. Copper sulphate by iodometry
4. Calcium gluconate by complexometry
5. Hydrogen peroxide by permanganometry
6. Sodium benzoate by non-aqueous titration
7. Sodium chloride by precipitation titration

IV. Determination of Normality by electro-analytical methods

1. Conductometric titration of strong acid against strong base
2. Conductometric titration of strong acid and weak acid against strong base
3. Potentiometric titration of strong acid against strong base.

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Contents

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Chapter ... 1

LIMIT TESTS

Experiment No. 1

Aim: *To perform the limit test of chloride.*

Requirements:

Apparatus: Nessler cylinders, Glass rod, Stand.

Chemicals: Dilute Nitric acid (10%), Silver nitrate (5%), Sodium chloride.

Principle:

It is based upon the chemical reaction between silver nitrate and soluble chlorides in the presence of dilute nitric acid to give opalescence of silver chloride. The opalescence produced is compared with the standard solution.

If the opalescence in the sample is less than the standard, it passes the test. If it is more than the standard, it fails the test.

$$Cl^- + AgNO_3 \xrightarrow{\text{dil. } HNO_3} AgCl + NO_3$$

Procedure: (Indian Pharmacopoeia 2010)

- Dissolve the specified quantity of the substance under examination in water, or prepare a solution as directed in the individual monograph and transfer to a Nessler cylinder.
- Add 10 ml of dilute nitric acid, except when nitric acid is used in the preparation of the solution, dilute to 50 ml with water and add 1 ml of 0.1 M silver nitrate.
- Stir immediately with a glass rod and allow to stand for 5 minutes protected from light. When viewed transversely against a black background any opalescence produced is not more intense than that obtained by treating a mixture of 10.0 ml of chloride standard solution (25 ppm) and 5 ml of water in the same manner.

Observation: When viewed transversely against a black background any opalescence produced is **not more intense/more intense** than a mixture of 10.0 ml of chloride standard solution (25 ppm).

Result: The sample **passes/fails** the limit test as per Indian Pharmacopoeia.

***

Experiment No. 2

Aim: *To perform limit test of sulphate.*

Requirements:

Apparatus: Nessler cylinders, Glass rod, Stand.

Chemicals: Dilute Hydrochloric acid and Barium sulphate reagent.

Principle:

It is based upon the chemical reaction between barium chloride and soluble sulphate in the presence of dilute hydrochloric acid. The turbidity produced is compared with the standard solution. Barium sulphate reagent contains barium chloride, sulphate-free alcohol and small quantity of potassium sulphate. The inclusion of the small quantity of potassium sulphate in the reagent increases the sensitivity of the test.

Alcohol prevents super saturation and more uniform turbidity develops. If the turbidity produced in the test is more intense than the standard turbidity it fails the test, otherwise, it passes the test.

$$SO_4^{2-} + BaCl_2 \xrightarrow{\text{dil. HCl}} BaCl_2 + 2Cl^-$$

Procedure: (Indian Pharmacopoeia 2010)

- To 1.0 ml of a 25.0 per cent w/v solution of barium chloride in a Nessler cylinder add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO_4), mix and allow to stand for 1 minute.
- Add 15 ml of the solution prepared as directed in the monograph or a solution of the specified quantity of the substance under examination in 15 ml of water and 0.15 ml of 5 M acetic acid. Add sufficient water to produce 50 ml, stir immediately with a glass rod and allow to stand for 5 minutes.
- The turbidity produced in sample solution should not be greater than standard solution. If the turbidity (opalescence) produced in sample solution is less than standard solution, the sample will pass the limit test.

Observation: When viewed transversely against a black background any opalescence produced is **not more intense/more intense** than a mixture of 15 ml of sulphate standard solution (10 ppm).

Result: The sample **passes/fails** the limit test as per Indian Pharmacopoeia.

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Experiment No. 3

Aim: *To perform limit test of iron.*

Requirements:

Apparatus: Nessler cylinders, Glass rod, Stand.

Chemicals:

1. Standard iron solution (0.1726 gm of ferric ammonium sulphate and dissolve in 10 ml of 0.1 N H_2SO_4 and sufficient water to produce 1000 ml).
2. 0.1 N H_2SO_4 (4.904 gm in 1000 ml of water).
3. 20% Iron free citric acid.
4. Thioglycollic acid.
5. Ammonia solution.

Principle:

The test depends upon the reaction between ferrous iron and thioglycollic acid in the presence of ammonia, when a pale pink to deep reddish purple colour is produced. Ferric iron is reduced to ferrous iron by the thioglycollic acid and the compound produced is ferrous thioglycollate.

Citric acid forms a soluble complex with iron and prevents its precipitation by ammonia as ferrous hydroxide. Ferrous thioglycollate is colourless in neutral or acid solutions. 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 of the ferric compound.

Therefore the colours should be compared immediately after the time allowed for full development of colours is over.

Procedure: (Indian Pharmacopoeia 2010):

- Dissolve the specified quantity of the substance under examination in water, or prepare a solution as directed in the monograph, and transfer to a Nessler cylinder.
- Add 2 ml of a 20 per cent w/v solution of iron-free citric acid and 0.1 ml of thioglycollic acid, mix, make alkaline with iron-free ammonia solution, dilute to 50 ml with water and allow to stand for 5 minutes.
- Any colour produced is not more intense than that obtained by treating in the same manner 2.0 ml of iron standard solution (20 ppm Fe) in place of the solution under examination.

Observation: When viewed transversely against a black background any opalescence produced is **not more intense/more intense** than a 2.0 ml of iron standard solution (20 ppm Fe).

Result: The sample **passes/fails** the limit test as per Indian Pharmacopoeia.

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Experiment No. 4

Aim: *To perform limit test of arsenic.*

Requirements:

Apparatus: Nessler cylinders, Glass rod, Stand.

Chemicals: Potassium iodide, Mercuric chloride paper, Zinc AsT.

Theory:

The limit for arsenic is indicated in the individual monographs in terms of ppm, i.e., the parts of arsenic. As per million parts (by weight) of the substance under examination, all reagents used for the test should have as low a content of arsenic as possible.

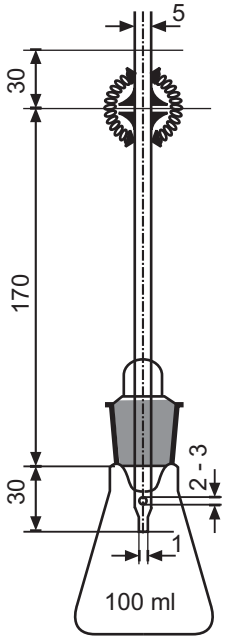

Fig. 1: Apparatus for arsenic limit test
[All dimensions in figure is in CGS unit]

Apparatus:

The apparatus (Fig. 1) consists of a 100 ml bottle or conical flask closed with a rubber or ground glass stopper through which passes a glass tube (about 20 cm × 5 cm). The lower part of the tube is drawn to an internal diameter of 1.0 cm, and 15 cm from its tip is a lateral orifice 2 to 3 cm indiameter. When the tube is in position in the stopper the lateral orifice should be at least 3 cm below the lower surface of the stopper. The upper end of the tube has a perfectly flat surface at right angles to the axis of the tube. A second glass tube of same internal diameter and 30 cm long, with a similar flat surface, is placed in contact with the first and is held in position by two spiral springs or clips. Into the lower tube insert 50 to

60 mg of lead acetate cotton, loosely packed, or a small plug of cotton and a rolled piece of lead acetate paper weighing 50 to 60 mg. Between the flat surfaces of the tubes place a disc or a small square of mercuric chloride paper large enough to cover the orifice of the tube (15 cm × 15 cm).

Procedure:

- Into the bottle or conical flask introduce the test solution prepared as directed in the individual monograph, add 5 ml of 1 M potassium iodide and 10 g of zinc AsT. Immediately assemble the apparatus and immerse the flask in a water bath at a temperature such that a uniform evolution of gas is maintained.
- After 40 minutes any stain produced on the mercuric chloride paper is not more intense than that obtained by treating in the same manner 1.0 ml of arsenic standard solution (10 ppm As) diluted to 50 ml with water.

Observation: When viewed transversely against a black background, any opalescence produced is **not more intense/more intense** than a 1.0 ml of arsenic standard solution (10 ppm As) diluted to 50 ml with water.

Result: The sample **passes/fails** the limit test as per Indian Pharmacopoeia.

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Chapter ... 2

VOLUMETRIC REAGENTS AND SOLUTIONS

Volumetric solutions, also known as standard solutions, are solutions of reagents of known concentrations intended primarily for use in quantitative determinations. Concentrations are usually expressed in terms of molarity (M).

Molar Solutions

A molar solution contains 1 g molecule of the reagent in 1000 ml of the solution. Thus, each litre of a molar solution of sodium nitrite contains 69.0 g of $NaNO_2$ and each litre of a molar solution of disodium edetate contains 372.2 g of $C_{10}H_{14}N_2Na_2O_8$, $2H_2O$. Solutions containing one-tenth of a gram-molecule of the reagent in 1000 ml are designated as 'tenth-molar' or 0.1 M; other molarities are similarly indicated.

Preparation and Standardization of Volumetric Solutions

It is not always possible nor is it essential, to prepare volumetric solutions of a desired theoretical molarity. A solution of approximately the desired molarity is prepared and standardized by titration against a solution of a primary standard. The molarity factor so obtained is used in all calculations, where such standardized solutions are employed. As the strength of a standard solution may change upon standing, the molarity factor should be redetermined frequently. Volumetric solutions should not differ from the prescribed strength by more than 10 per cent and the molarity should be determined with a precision of 0.2 per cent.

When solutions of a reagent are used in several molarities, the details of the preparation and standardization are usually given for the most commonly used strength. Stronger or weaker solutions are prepared and standardized using proportionate amounts of the reagent or by making an exact dilution of a stronger solution. Volumetric solutions prepared by dilution should be restandardised either as directed for the stronger solution or by comparison with another volumetric solution having a known ratio to the stronger solution.

The water used in preparing volumetric solutions complies with the requirements of the monograph on purified water, unless otherwise specified. When used for the preparation of unstable solutions such as potassium permanganate or sodium thiosulphate, it should be freshly boiled and cooled.

When a solutions to be used in an assay in which the endpoint is determined by an electrochemical process (e g. potentiometrically), the solution must be standardized in the same way.

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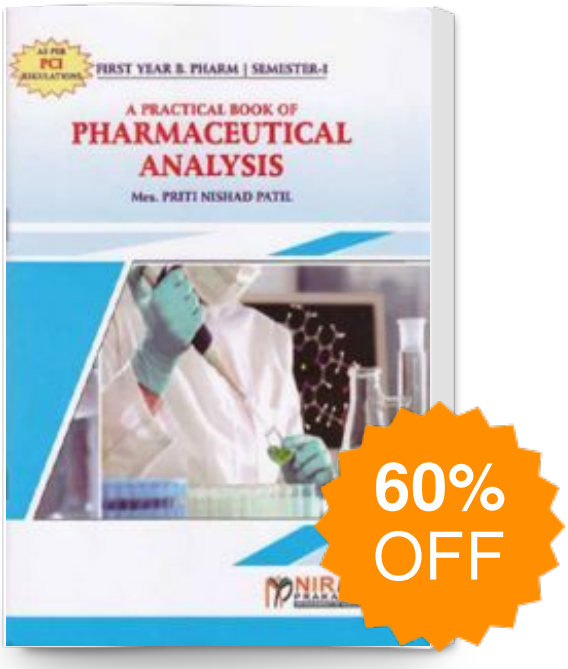
A Practical Book Of Pharmaceutical Analysis



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